Unconfined Compressive Strength of Various Cement-Stabilized Phosphogypsum Mixes

S. Ong, J. B. Metcalf, R. K. Seals, and R. Taha

The unconfined compressive strength of various cement-stabilized phosphogypsum mixes is described. The mixes vary in compacted dry unit weight, moisture content, cement type and content, and curing procedure and time. It is shown that the mixes behave in similarly to cement-stabilized soil. The strength increases with increases in dry unit weight, cement content, and curing period. Strength decreases with increasing compaction moisture content and with a soaked curing regime. The strengths exceed a typical minimum specified unconfined compressive strength (1.7 MPa at 7 days) only at modified Proctor compacted densities (at 4 percent cement) and more than 14 percent at standard Proctor compaction level. A limited study of the effects of adding sand, hydrated lime, and calcium chloride showed that sand and small additions of calcium chloride increase the 7-day strength. Lime was ineffective. The use of constant volume molds was also shown to be effective in allowing direct control of unit weight and moisture content in compacted specimens.

Phosphogypsum is a non-hazardous by-product of the manufacture of phosphate fertilizers. It is being produced at the rate of 36 million Mg in the United States, where stockpiles are estimated to reach 1.8 billion Mg by 2000. A search for uses of this industrial by-product has been under way for some time (1), and there is potential for use in agriculture, as a soil conditioner; in building, for the production of plaster sheet; and in chemical engineering, as a source of sulfuric acid and other chemicals. All these uses are constrained to some degree by the presence of impurities, by a concern for potential environmental hazards and by the economics of the use of phosphogypsum in competition with alternative materials. A potential major use exists in civil engineering, where the material can be used as fill for road base and for cast block applications, such as artificial reef construction. The potentially largevolume uses of this nature are particularly attractive.

However, the road base and cast block applications require enhanced properties of the material, and stabilization of some form is necessary to achieve adequate properties for such uses. The stabilization process must be inexpensive and simple to be acceptable and to compete with other materials and processes.

The research described herein addresses the properties of cement-stabilized phosphogypsum (CSPG), principally unconfined compressive strength, and discusses the interaction of compacted density and moisture content, cement type and

S. Ong, J. B. Metcalf, R. K. Seals, Institute for Recyclable Materials, Louisiana State University, 1419 CEBA, Baton Rouge, La. 70803. R. Taha, Department of Civil Engineering, South Dakota State University, Box 2219, Brookings, S.D. 57007.

content, curing regimes, and the effects of the admixture of sand, hydrated lime and calcium chloride as secondary additives.

PREVIOUS WORK

Stabilization of phosphogypsum for road base construction is not new (2), and several earlier studies have been carried out, for example, in Florida (3), Louisiana (4), and Texas (5). Generally these studies showed that phosphogypsum, which is poorly crystallized calcium sulphate dihydrate, with small quantities of impurities, behaves like silty soil. Tables 1 and 2 give typical properties of Louisiana phosphogypsum (4). In the natural state the phosphogypsum is not suitable for road base. The addition of cement typically results in a change in the compacted unit weight and optimum moisture content and an increase in unconfined compressive strength. Phosphogypsum stabilized with cement is suitable for use as road base; both full-scale experiments and actual construction projects have demonstrated this in Texas (6) and Florida (7,8).

The properties of the CSPG depend on the source and nature of the phosphogypsum, of the cement type and content, and of the construction process. Because of the intrinsic variability of all three, any particular application requires a study of the specific mixtures to be used. An area of concern currently receiving increased attention is the low level of radioactivities exhibited by phosphogypsum originating from Florida phosphate rocks (9). Studies of the effects of stabilization on radon emanation will be part of future research at the Institute for Recyclable Materials, Louisiana State University. Radon emanation is a key issue in Environmental Protection Agency regulation currently under review.

PROPERTIES OF CSPG

This paper describes current studies at the Institute for Recyclable Materials that are directed to the design and conduct of full-scale field experiments with CSPG pavements. It is planned to prove the suitability of the product and to develop appropriate material and construction specifications for road base in Louisiana, where the lack of natural aggregate deposits has resulted in soil stabilization being widely used for road base. Because some 91 million Mg of phosphogypsum is stockpiled in the state, the potential for CSPG road base construction warrants serious and early consideration.

TABLE 1 Results of Chemical Analysis of Louisiana Phosphogypsum

Constituent	Content (%)		
Cao	29 - 31		
SO₄	50 - 53		
sio,	5 - 10		
F	0.3 - 1.0		
P_2O_5	0.7 - 1.3		
Fe ₂ O ₃	0.1 - 0.2		
Al_2O_3	0.1 - 0.3		
pH ^a	2.8 - 5.0		

^{*} pH: not measured as a percent.

The study initially examined the effect of moisture, dry unit weight, cement type and content, and curing procedure and time on the unconfined compressive strength of the CSPG. Unconfined compressive strength is the common criterion for the suitability of stabilized soils for road base with a 7-day strength of 1.7 MPa frequently adopted as a minimum (10).

A second exploratory series of tests examined the effect of secondary additives on the unconfined compressive strength.

Moisture-Unit Weight Relations

The strength and bearing capacity of earthen materials depend critically on the compacted dry unit weight and moisture content. However, the addition of cement to soil usually changes the compacted dry unit weight and optimum moisture content for both standard and modified Proctor compaction; the first task was to establish these parameters for samples of phosphogypsum produced in Louisiana. Figure 1 shows a typical result, and Table 3 summarizes the tests conducted as part of this study.

The results are typical of the behavior of a cement-stabilized material and show standard Proctor compaction (ASTM D698) maximum dry densities of the order of 1.4 T/m³ at moisture contents of the order of 20 percent. The modified Proctor (ASTM D1557) maximum dry densities were 10 to 15 percent higher at 15 to 30 percent lower moisture contents. (Note that this moisture content is derived by drying at 55°C; a higher drying temperature than 60°C will decompose the phosphogypsum into a hemihydrate form.)

TABLE 2 Results of Physical Analysis of Louisiana Phosphogypsum

Property	Value
Free Moisture (Top)	8 - 18% (Varies with depth)
Free Moisture (phreatic water)	25 -30%
Specific Gravity (Average)	2.35
Fineness (< # 200 si (Average)	eve) 75%

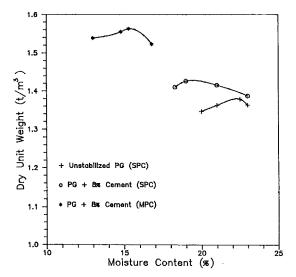


FIGURE 1 Effect of cement content on compacted dry unit weight and optimum moisture content.

Cement Type and Content

The effect of cement type and content on unconfined compressive strength (ASTM D1633) was investigated by preparing samples by both standard and modified compaction. Typical results are shown in Figures 2 and 3. The results again showed typical behavior, with unconfined compressive strength increasing with cement content but varying with cement type. Type I (ordinary portland cement), Type II (moderate sulphate resistance), Type IIA (a low C3A cement), and Type V (high sulphate resistance) cements were used initially. The Type IIA cement was included as earlier studies by Freeport-McMoRan and others (11) had shown that a high C3A content could lead to expansion of the CSPG. The formation of ettringite is a possible cause (12), and studies of the fundamental mechanisms are continuing. However, the ASTM D1633 procedure, representing the constant compactive effort process used in construction, does not separate the effects of cement content from those of dry unit weight and moisture content changes the standard and modified Proctor dry densities at only the moisture content actual. Later tests, described in the following, were addressed to this issue and also examined the effect of a Type III (high early strength) cement.

Curing

Most samples were cured in double-sealed plastic bags at a constant temperature (70°F) for 7, 28, 56, 90, and 180 days. Selected mixes were also cured for 7 days by this procedure and then soaked for 21 days in water at room temperature. The soaked curing regimes generally reduce strength gain and may reduce the strength below that achieved at 7 days (Figure 4).

ADEQUACY FOR ROAD BASE

The results of the first series of tests demonstrated that CSPG behaved as a typical cement-stabilized road base material.

TABLE 3 Range of Dry Unit Weight and Moisture Content

Cement Type/Content (%)	Compaction Procedure				
	Standard Proctor		Modified	Modified Proctor	
	Dry Unit Weight (t/m³)	Moisture Content (%)	Dry Unit Weight (t/m³)	Moisture Content (%)	
No Cement	1.38	22.0			
II/4	1.36	21.4	1.57	14.6	
V/4	1.37	22.7	1.55	13.8	
IP/4	1.38	19.3	1.54	16.7	
IIA/4	1.40	21.5	1.55	14.2	
II/8	1.41	20.8	1.57	14.4	
V/8	1.43	19.2	1.56	15.3	
IP/8	1.40	20.7	1.56	14.2	
IIA/8	1.40	19.3	1.58	13.5	
II/10	1.35	23.4			
V/10	1.38	21.6			
IIA/10	1.36	23.5			
II/14	1.38	23.5			
V/14	1.37	23.8			
IIA/14	1.38	23.5			

^{*} The Cement percentages are based on dry weight of phosphogypsum

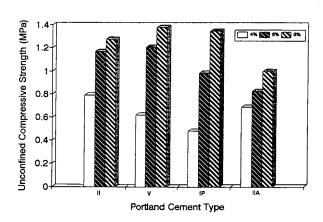


FIGURE 2 Effect of cement type and content on 7-day unconfined compressive strength, standard compaction.

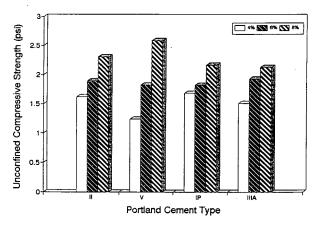


FIGURE 3 Effect of cement type and content on 7-day unconfined compressive strength, modified compaction.

However, the results also showed that the strengths attained at standard Proctor compaction levels and economic cement contents (about 8 percent) were barely adequate in terms of the Louisiana Department of Transportation and Development criterion of 1.7 MPa at 7 days. This strength could be achieved by increasing cement content to more than 14 percent or by increasing the compacted dry unit weight to modified Proctor compaction levels at 4 to 6 percent cement. The practicality of the first is in doubt because of the increased

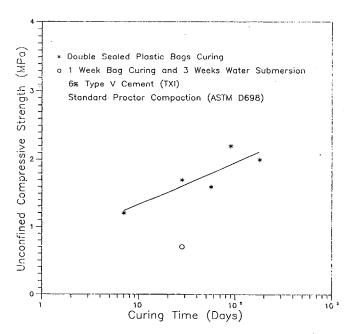


FIGURE 4 Effect of curing time and procedure on unconfined compressive strength.

cost, and of the second because of the difficulty in attaining high compaction levels on the typically soft subgrades of the region.

These results were sufficiently different from earlier studies (12) to suggest that particle-size variability of the phosphogypsum may have an effect. Further studies of this are necessary. However, as small changes in compacted dry unit weight can have a major effect on the unconfined compressive strength (10), this also needs clarification.

It therefore was decided to seek ways of increasing the unconfined compressive strength by the use of secondary additives and to seek to better understand the effects of moisture content and unit weight changes. This required the capability to make samples of controlled unit weight or moisture content. The simple way to achieve this is to move to a constantvolume mold technique, where a known mass of materials is compacted into a known-volume mold. At the same time a means of conserving material and reducing experimental effort was sought. The solution was to adopt the British Standard (BS1924) procedure, using a right cylinder specimen 2 in. in diameter and 4 in. long. The fine grain size of phosphogypsum (75 percent passing #200; Table 2) means that there is no effect of mold size and a saving of 80 percent in material (and effort) between this size specimen and the Proctor mold size is achieved.

ENHANCEMENT OF CSPG MIXES

The second series of tests, therefore, was designed to enhance the program to give a better understanding of the effects of dry unit weight and moisture content, by using the more convenient specimen size with the ability to control unit weight and moisture content separately, and to seek enhanced unconfined compressive strength of the stabilized mixes, by using secondary additives. Mixes that have evident potential for application will then be examined in more detail. In particular, the resilient modulus of the mixes will be determined by repeated loading test for use in mechanistic pavement design procedures.

Density, Moisture Content, and Curing

A limited series of tests was conducted to separate the effects of dry unit weight and moisture content on strength and to look again at the effects of curing. The results are shown in Figures 5 and 6. At constant moisture content it is demonstrated that the strength increases as the dry unit weight increases, and, at constant dry unit weight, it is shown that the strength varies little with moisture content at compaction. The effect of soaking is to reduce strength, but the reduction decreases with increasing dry unit weight or cement content.

High Early Strength Cement

As the strength criterion commonly adopted is applied at 7 days, and because it is known that calcium sulphate retards the set of cement, it was considered that the use of a high early strength (Type III) cement might result in higher 7-day

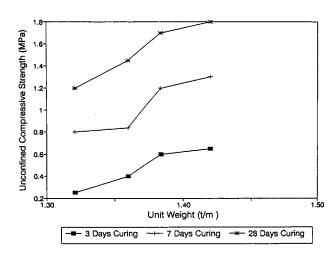


FIGURE 5 Effect of increasing dry unit weight on unconfined compressive strength.

strengths than an ordinary portland cement. The results showed that although 3-day strengths were higher the effect at 7 days was too small to be of practical value (Figure 7).

Secondary Additives

The low early strength suggested that three potential secondary additives should be examined: the use of another mineral component (sand) to improve the grading of the mix, the use of a cement set accelerator (calcium chloride), and the use of a neutralizing agent (hydrated lime) to increase the pH of the mix. The results of the tests are given in Figure 7.

It is evident that lime had little effect, that the sand mixes have evident potential for application, and that calcium chloride was effective at small proportions.

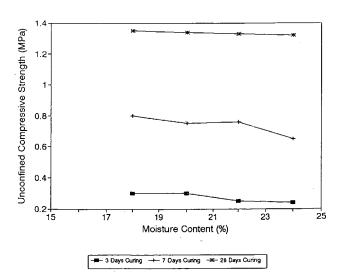


FIGURE 6 Effect of increasing moisture content on unconfined compressive strength.

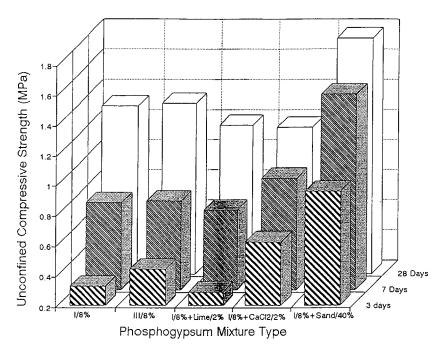


FIGURE 7 Effect of rapid-setting cement (Type III) and secondary additives on unconfined compressive strength.

CONCLUSIONS

Phosphogypsum can be stabilized with cement to produce an adequate material for road base construction to the requirements of the Louisiana Department of Transportation and Development. The final choice of a mix will depend on economics and construction practicability, but the use of more than 14 percent of ordinary portland (Type I) cement will produce adequate strength, as will the use of modified Proctor compaction standards for the construction. The strength can also be increased by the use of an admixture of sand with the phosphogypsum or by the use of a cement set accelerator, calcium chloride. The economics of the various options must now be examined and the experimental program extended for a better knowledge of the effects of the proportions of the various secondary additives and combinations of these additives. Attention must also be turned to the effects of the stabilization process on the permeability and leachability (13) of the compacted stabilized road base materials to determine the possible effects on the environment.

REFERENCES

- Taha, R., and R. K. Seals. Applications Identification of Phosphogypsum. Report I-91-3D. Institute for Recyclable Materials, College of Engineering, Louisiana State University, Baton Rouge, May 1991.
- Taha, R., and R. K. Seals. Phosphogypsum Literature Review. Report I-90-3. Institute for Recyclable Materials, College of Engineering, Louisiana State University, Baton Rouge, March 1991.
- 3. Ho, R. K. H., and W. H. Zimpfer. Comments on the Investigations of Phosphogypsum for Embankment Construction. *Proc.*,

2nd Workshop on By-Products of the Phosphate Industries, Florida Institute of Phosphate Research, May 1985.

- Taha, R., and R. K. Seals. Utilization of FMI's Phosphogypsum in Road Construction—A Review of FMI's Research Results. Report to Freeport-McMoRan, Inc.; Institute for Recyclable Materials, Louisiana State University, Baton Rouge, April 1990.
- Gregory, C. A., B. Saylak, and W. B. Ledbettes. The Use of By-Product Phosphogypsum for Road Bases and Subbase. In Transportation Research Record 998, TRB, National Research Council, Washington, D.C., 1984.
- Wong, C., and M. K. Ho. The Performance of Cement Stabilized Phosphogypsum as Base—State Highway 146, LaPorte, Texas. Report DHT-11. Texas Department of Highways and Public Transportation, Austin, Oct. 1988.
- 7. Kenley, W. C., and W. F. Chang. Polk County Experimental Road. *Proc.*, 2nd International Symposium on Phosphogypsum, Florida Institute of Phosphate Research, Vol. 2, 1988.
- 8. Mo, R. K. M., R. W. Williams, L. L. Cogdill, and W. F. Chang. Columbia County Experimental Road. *Proc.*, 2nd International Symposium on Phosphogypsum, Florida Institute of Phosphate Research, Vol. 2, 1988.
- May, A., and J. W. Sweeny. Evaluation of Radium and Toxic Element Leaching Characteristic of Florida Phosphogypsum Stockpiles. In ASTM STP 801, Atlanta, Ga., April 1985.
- 10. Metcalf, J. B., and O. G. Ingles. Soil Stabilization—Principles and Practice. Butterworths, Sydney, Australia, 1972.
- Taha, R., R. K. Seals, M. E. Tittlebaum, W. Thornsberry, and J. T. Houston. The Use of By-product Phosphogypsum in Road Construction. Presented at 71st Annual Meeting of the Transportation Research Board, Washington, D.C., 1992.
- 12. Taha, R., and R. K. Seals. Expansion Test Specimens—Results and Recommendations. Report 1-91-4D. Institute for Recyclable Materials, College of Engineering, Louisiana State University, Baton Rouge, July 1991.
- 13. Taha, R., and R. K. Seals. Engineering Properties and Potential Uses of By-Product Phosphogypsum. ASCE (in press).

Publication of this paper sponsored by Committee on Cementitious Stabilization.