

# The Use of By-Product Phosphogypsum for Road Bases and Subbases

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## ABSTRACT

Phosphogypsum is a chemical by-product of phosphoric acid processing plants and consists mainly of calcium sulfate dihydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ). Because of trace impurities, this material has not been used to replace natural gypsum in the housing industry causing domestic stockpiles to accumulate at rates that could produce an inventory of one billion tons by the year 2000. Environmental pressures as well as increased land costs associated with stockpiling phosphogypsum are causing researchers to look for better utilization of this material. One concept involves the use of fly ash or portland cement stabilized by-product phosphogypsum mixtures for construction of road bases and subbases. The results of a study conducted by the Texas Transportation Institute for the Mobil Chemical Company are presented. Phosphogypsum from the Mobil Chemical Company's stockpile in Pasadena, Texas, is used in the study. Use of this locally available material would result in significant energy savings by reducing expensive transportation charges usually incorporated in the cost of conventional construction materials that are presently used in the Houston area. Factors involving strength development such as degree of compaction, stabilizer content, optimum moisture content, and chemical make-up of the phosphogypsum are presented relative to acceptability for use in base courses and subbases. In particular, excellent compressive strengths were obtained so long as compacted dry densities were at least 97 percent of optimum modified Proctor. It was also found that the pH of the phosphogypsum may have a significant effect on the compressive strength. Strengths of mixtures containing highly acidic (pH < 3) phosphogypsum were significantly less than those containing a less acidic (pH around 5) material.

The resulting filter cake containing the hydrated calcium sulfate is called phosphogypsum. Typically, the filter cake slurry and wash solution are piped to large stockpiles where they are allowed to disperse.

Approximately 4.5 to 5.5 tons of phosphogypsum are generated for every ton of phosphoric acid produced (2). Worldwide demand for phosphoric acid further magnifies the problem of efficiently and economically dealing with growing phosphogypsum inventories. In 1980 phosphogypsum was generated at an annual rate of 840,000 tons in Australia alone (3). In 1979 Japan was producing phosphogypsum at a rate of 2.748 million metric tons per year (4). In Florida, more than 334 million tons of phosphogypsum had been stockpiled as of 1980. At that time, Florida's phosphogypsum production was 33 million tons per year. Long-term projections predict that more than 1 billion tons will be stockpiled in Florida by the year 2000 (5). Obviously, these large quantities of material must be dealt with before they become unmanageable. Similar problems with utilization and disposal of this material also exist in Europe and Canada (6).

Crystals of calcium sulfate can exist in at least three forms: anhydrate ( $\text{CaSO}_4$ ), hemihydrate ( $\text{CaSO}_4 \cdot 1/2\text{H}_2\text{O}$ ) and dihydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ). The percentages of each crystal type in a given sample determine, both directly and indirectly, the properties of the phosphogypsum. Goers (7) states that crystal type and size affect the amount of phosphoric acid remaining in the filter cake after filtration, which, in turn, may affect the engineering properties of the phosphogypsum (8).

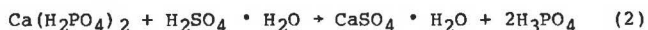
Chemical impurities in phosphogypsum hinder cost-effective utilization. A study presented by Sweeney and May (5) summarizes an analysis of nine different phosphogypsum storage sites in Florida. Thirty-nine different elements were detected. The same study also examined radiation emissions from the stockpiles. Results showed that radium concentration averaged 21 picocuries per gram and was greatest in the finer size fractions. The complexity and variety of components within a stockpile coupled with possible radiation hazards further deters the widespread acceptance and utilization of phosphogypsum in building materials and cements.

Present attempts to develop beneficial uses of phosphogypsum can be summarized as follows:

1. As a cement retarder (9),
2. As a sulfur source through desulfurization (10),
3. As building plaster (Plaster of Paris) (11), and
4. In reclamation of sodic soils (12).

Each of these applications have met with varying degrees of success. However, the common denominator, especially concerning uses in building materials and cement, appears to be the degree of purity of the phosphogypsum.

Phosphogypsum is a by-product of phosphoric acid production. Of the several different phosphoric acid production processes, the wet process is the most frequently used (1). In the wet process, finely ground phosphate rock,  $\text{Ca}_{10}\text{F}_2(\text{PO}_4)_6$ , is dissolved in phosphoric acid to form a monocalcium phosphate solution (see Equation 1 below). Sulfuric acid, which is added to the slurry, reacts with the monocalcium phosphate to produce a hydrated calcium sulfate that can then be separated from the phosphoric acid by filtration (see Equation 2):



## ENERGY CONSIDERATIONS

In 1980 Texas was the nation's leading producer of crushed stone, with 177 quarriers producing 76 million tons (13). Texas is the nation's second leading state in the production of construction sand and gravel, producing about 47 million tons in 1980 (13). The uses of these materials include:

1. Aggregate for asphaltic concrete and portland cement concrete,
2. Material for the construction of highways and streets,
3. Material for pipe bedding, and
4. Material for load-bearing surfaces.

While phosphogypsum does not present a viable alternative to use Number 1, it should be considered for the remaining categories.

A significant portion of this construction material is needed to satisfy the needs of Houston, Texas. Although Houston accounts for only slightly more than 20 percent of the state's population, it accounts for some 26 percent of anticipated new highway construction (14). Sizable amounts of aggregate are currently used as pipe bedding in sewer and wastewater project construction in Houston. Although the quantities of aggregate used in load-bearing surfaces such as parking lots, container storage yards, and so forth, is presently not definable, it is possible to say that this application is a potentially viable segment for phosphogypsum application (14).

Although an aggregate demand exists, an adequate supply in the Houston area does not. "There is virtually no crushed stone production near the Houston market. The production occurs in the San Antonio, Austin, and Georgetown, Texas, area--approximately 200 miles from the Houston market" (14). Some large shipments have been made to Houston area markets from as far away as Kentucky. Although little or no crushed stone is produced in Houston, there is a significant volume of sand and gravel quarried in the same area. One major supplier is located approximately 60 miles from Houston city limits.

It comes as no surprise, then, that transportation charges represent a significant share of the total costs of aggregate materials (including sand and gravel) in the Houston market (14). As stated by one observer, "the major factor, which gives a common and nearly ubiquitous mineral resource a measure of intrinsic value, is the cost of transporting the mineral to market" (15).

By using a supply of aggregate material located nearer the market, there would be a significant reduction in energy consumption. It is estimated that approximately 410 BTUs are required to move a ton of aggregate one mile by rail (BTU/TM) (16). Aggregate shipments by truck require 2,280 BTUs/TM (16). It is obvious that by using an aggregate such as phosphogypsum located in the Houston area, considerable amounts of energy, both in plant operations and hauling, could be saved.

## POSSIBLE SOLUTIONS

As stockpiles continue to grow and environmental constraints become more stringent, widespread uses of phosphogypsum must be developed. One such possible use, especially in aggregate-poor areas such as Houston, could be as a base material in highway construction. If feasible, large tonnages of stockpiled material could be used. In an effort to enhance the engineering properties of most phosphogypsum, some cementitious or pozzolanic binder could be added to

the system. Two such materials readily available are high lime fly ash and portland cement. Each have been used successfully as soil stabilizers for many years. High lime fly ash (ASTM C 618 Class C) (17) is a pozzolan that contains appreciable amounts of calcium oxide (CaO) and exhibits a cementitious reaction with water in addition to its pozzolanic reactivity. This reactivity could be used to create a mixture comparable to fly ash stabilized soils.

In general, portland cement is more readily available than is high lime fly ash and does not have the potential problem of variability associated with a by-product such as fly ash. Addition of sufficient percentages of portland cement may produce a material comparable to soil cement.

This report contains the results of a study in which high lime fly ash and portland cement were used as stabilizing agents for phosphogypsum. The phosphogypsum was collected from two different stockpiles at the same storage site. Design, construction, and evaluation of seven experimental test sections resulted from the laboratory study.

## MATERIALS

Phosphogypsum (PG)

All phosphogypsum used in this study was furnished by Mobil Chemical Company (MCC) of Pasadena, Texas. Presently, their inventory contains about 25 million tons of PG that are stored in three separate stockpiles referred to as Pile Nos. 1, 2, and 3, respectively. The numbering system reflects the relative age of the pile with Pile No. 1 being the oldest. Pile Nos. 2 and 3 are considered active piles, but only the latter is currently being used for PG discharge and therefore has been exposed to the elements for the shortest time. Initial research involving the use of Pile No. 3 material produced mixtures with low compressive strengths. Mixtures prepared with PG from Pile No. 2 possessed more acceptable strength characteristics. Therefore, subsequent laboratory and field experimentation involved material from Pile No. 2. Chemical and physical properties of phosphogypsum from Piles 2 and 3 are given in Table 1.

Fly Ash (FA)

The fly ashes used in this study were furnished from the W.A. Parrish power plant located in Thompsons, Texas. Laboratory analyses were conducted in which the ash was established to be acceptable for classification as a Class C fly ash in accordance with ASTM C 618 (17). The results of the chemical analysis are given in Table 2. Total CaO contents analyses were also performed by use of a heat evolution test developed at Texas Transportation Institute (18). A total CaO content of 25.4 percent was determined.

In addition, pH and specific gravity measurements produced values of 11.6 and 2.55, respectively. Particle size analysis showed the fly ash fineness, which was evaluated on the basis of the amount retained on the Nos. 200 and 325 sieves to be 10 percent and 15 percent, respectively.

Portland Cement (PC)

The cement used in this study was Type I. Common percentages of cement used for stabilization range from 3 to 8 for sands and gravels to 10 to 16 for plastic clays (19).

**TABLE 1 Chemical Breakdown and Physical Properties for MCC Phosphogypsum**

Constituent <sup>a</sup>	Pile No. 3 (Weight Percent)
Ca	20.8
SO <sub>4</sub>	53.1
SiO <sub>2</sub>	2.5
PO <sub>4</sub>	2.1
Mg	0.1
H <sub>2</sub> O	20.0
Other	1.4
<hr/>	
pH	
Pile No. 2	5.0
Pile No. 3 (below surface)	2.6
Specific Gravity	2.3
Moisture Content, % (Average)	20.0

<sup>a</sup>Samples were known to contain varying quantities of Li, Al, Fe and F. These were not qualified due to incomplete dilute mineral acids.

**TABLE 2 Laboratory Analysis of Fly Ash**

Test	Results on Dry Basis
Loss on Ignition	< 0.01
Silicon Dioxide, SiO <sub>2</sub>	38.01
Aluminum Oxide, Al <sub>2</sub> O <sub>3</sub>	21.44
Calcium Oxide, CaO	17.66
Ferric Oxide, Fe <sub>2</sub> O <sub>3</sub>	6.13
Magnesium Oxide, MgO	4.25
Sulfur Trioxide, SO <sub>3</sub>	2.33
Titanium Dioxide, TiO <sub>2</sub>	1.65
Sodium Oxide, Na <sub>2</sub> O	3.79
Potassium Oxide, K <sub>2</sub> O	0.79
Manganese Oxide, MnO <sub>2</sub>	0.04
Phosphorus Pentoxide, P <sub>2</sub> O <sub>5</sub>	0.15
Total	96.24

Chemical Requirements for ASTM C 618 Class

Sample	Actual, %	Class C Requirements, %
SiO <sub>2</sub> + Al <sub>2</sub> O <sub>3</sub> + Fe <sub>2</sub> O <sub>3</sub>	65.58	50.0 Minimum
SO <sub>3</sub>	2.33	5.0 Maximum
% Moisture	1.31	3.0 Maximum
Loss on Ignition (Dry)	<0.01	6.0 Maximum
MgO	4.25	5.0 Maximum

<sup>a</sup>This sample meets C 618 requirements for Type C Mineral Admixture.

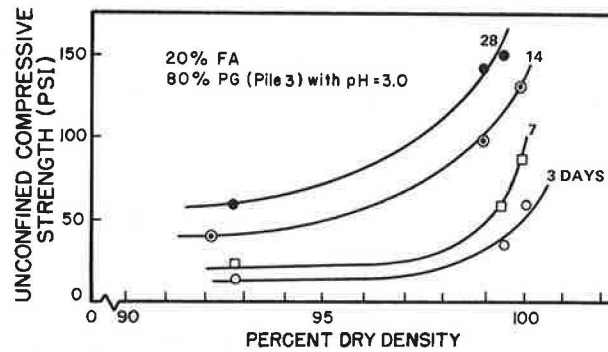
**LABORATORY STABILIZATION PROGRAM**

General

Unconfined compressive strength was chosen as the governing parameter for evaluation. Typically, relations have been developed relating unconfined compressive strength to other material characteristics (flexural strength, modulus, etc.) for stabilized soils and concrete. Although no such correlation currently exists for stabilized phosphogypsum mixtures, unconfined compressive strength was selected as an appropriate starting point.

Experimental Outline

Results from work performed with Pile No. 3 material were instrumental in the evaluation of pile No. 2 material (20). In particular, the effects of pH and dry density on compressive strength helped to limit further experimental efforts to phosphogypsum from the surface of the pile compacted at optimum moisture contents. The Pile No. 3 material was acidic with a pH of about 3.0. Dry density levels of at least 97 percent optimum modified Proctor resulted in a dramatic upturn in strength (see Figure 1).



**FIGURE 1 Relationship between optimum dry density and unconfined compressive strength for fly-ash stabilized phosphogypsum from Pile No. 3.**

To analyze other factors affecting mixture strength, a detailed experimental design was developed for Pile No. 2 (pH around 5) material. Several percentages for each stabilizer were investigated. Strength was measured at four ages with three repetitions for each sample type. The 4-in. x 4.5-in. specimens, subjected to modified Proctor compaction (21), were cured under sealed conditions at 25°C and 25 percent RH. Table 3 gives the stabilizer contents evaluated in this phase of the study.

Figure 2 shows strength development of mixtures of fly ash and phosphogypsum from Pile No. 2. ASTM C-593 (22) specifies an unconfined compressive strength of 400 psi, after curing at elevated temperatures and subjection to vacuum saturation. Although these specimens were not subjected to this curing condition, Smith's data (23) have shown that addition of 20 percent fly ash meets this level. As can be observed in Figure 2, the ASTM recommended strength level was attained with the addition of only 15 percent fly ash. Twenty-eight day strengths range from 200 psi at the 10 percent fly ash level to 1,800 psi at the 30 percent fly ash level.

Strength development with the use of portland cement is shown in Figure 3. The Portland Cement Association (PCA) (24) recommends minimum 7-day unconfined compressive strengths for fine-grained

**TABLE 3 Summary of Mix Designs Evaluated with Pile No. 2 Phosphogypsum**

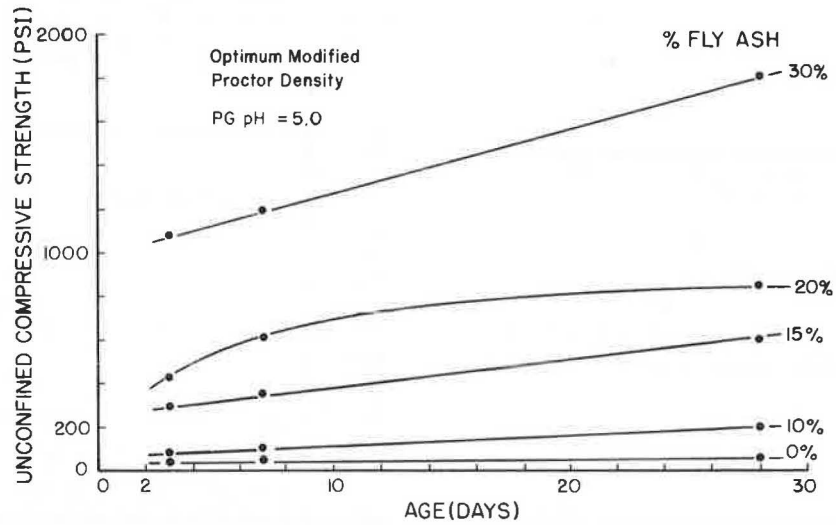
Stabilizer	Percent Stabilizer	Optimum <sup>a</sup> Moisture Content
Fly Ash	0	14
	10	13
	15	13.5
	20	14
	30	13
Portland Cement	3	14
	6	14
	10	14

<sup>a</sup>Based on total dry weight of mix.

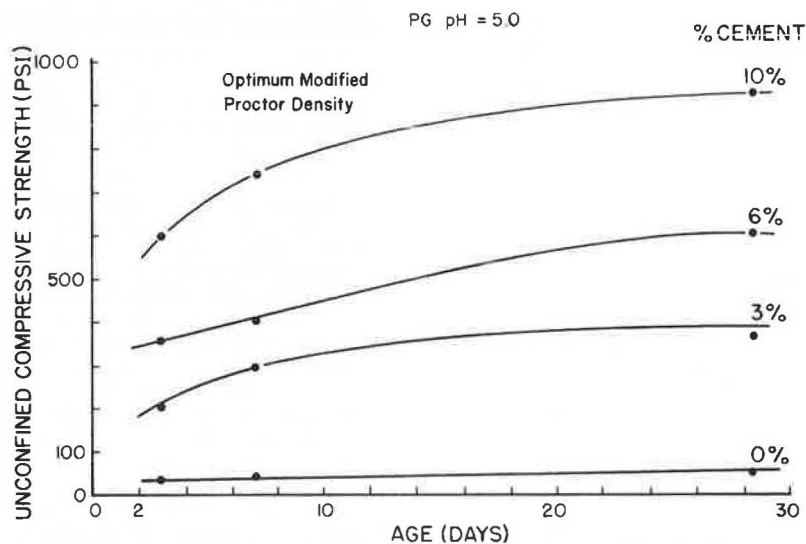
soils of 200 to 300 psi. All 7-day strengths of the stabilized mixtures evaluated exceeded 200 psi whereas the 6 percent and 10 percent specimens each exceeded the 300 psi level.

The Texas State Department of Highways and Public Transportation (SDHPT) specifies a minimum strength of 650 psi after 7 days for cement-stabilized bases (CSB) (25). Although mixtures of Pile No. 3 material did not meet this specification, Pile No. 2 material stabilized with 30 percent fly ash and 10 percent portland cement materials met these specifications (see Figures 2 and 3). Furthermore, 28-day strengths of the 20 percent fly ash specimens exceeded this strength level. With this in mind, stage construction of a pavement system may be a viable possibility when using such materials. This would mean keeping the base free from heavy traffic and well-drained until sufficient strength had been developed.

Stabilized Pile No. 2 strengths were much greater than those from stabilized Pile No. 3 phosphogypsum. These differences appear to be linked with pH. Strengths from Pile No. 3 material (pH around 3)



**FIGURE 2** Strength development of Pile 2 phosphogypsum (stabilized with fly ash) with time.



**FIGURE 3** Strength development of Pile 2 phosphogypsum (stabilized with portland cement) with time.

stabilized with 30 percent fly ash were 300 psi after 28 days. Pile No. 2 material (pH around 5), compacted and stabilized similarly, achieved 1,800 psi at the same age. Mixtures of Pile No. 2 material reached strengths in excess of 900 psi with the addition of 10 percent portland cement.

In any event, data from two different sources point out that pH, or something associated with pH, can have a significant effect on compressive strength of mixtures containing phosphogypsum. Whether pH itself is the key indicator of phosphogypsum quality or just a flag for some other factor has yet to be determined. The determination of this fact, perhaps through neutralization of the material, should be the subject of further research. Such a determination may result in a process whereby existing fresh phosphogypsum could, if necessary, be modified for use.

#### CONCLUSIONS

The strength of stabilized phosphogypsum was evaluated in this study from two different stockpiles located within the same storage site. The effects of stabilizer type and content, age, and pH on unconfined compressive strengths were investigated.

Based on the results generated to date, the following conclusions are drawn.

1. The potential exists for use of fly ash-phosphogypsum and portland cement-phosphogypsum mixtures as a base material.
2. Stabilizer content, mixture dry density, and phosphogypsum pH have a major influence on unconfined compressive strength of the stabilized mixtures.
3. The ASTM specification for lime-fly ash stabilized material (400 psi) was met with the addition of 20 percent fly ash.
4. Portland Cement Association recommendations for unconfined compressive strength for soil cement (200 to 300 psi) were met by using at least 3 percent portland cement and modified Proctor compaction procedures.
5. Compressive strength of stabilized samples prepared with Pile No. 3 material (with a phosphogypsum pH around 3) did not meet SDHPT specifications for cement-stabilized base (650 psi in 7 days). Compressive strength of stabilized samples prepared with Pile No. 2 material (with a phosphogypsum pH around 5) did meet this specification when mixed with 30 percent fly ash or 10 percent portland cement.

#### FUTURE WORK

Although much has been learned about the properties of phosphogypsum, many areas are left unexplored. The actual role played by phosphogypsum as reflected by its pH must be determined. Successful research may lead to phosphogypsum modification for more optimum utilization. Other engineering properties for stabilized phosphogypsum should also be evaluated. These include determination of wet-dry susceptibility, fatigue properties, shrinkage characteristics and a modulus, preferably by a repeated load tri-axial test.

#### ACKNOWLEDGMENT

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## Construction and Performance of Experimental Base Course Test Sections Built with Waste Calcium Sulfate, Lime, and Fly Ash

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### ABSTRACT

Although a modest amount of information is available on the properties of lime-fly ash-calcium sulfate mixtures, little is known about their field performance. Consequently, an investigation was undertaken to determine the practical feasibility of using such mixtures as roadway base course materials. This study involved the construction and performance evaluation of four experimental pavement base course sections 100 ft long and 10 ft wide incorporating predetermined formulations of waste calcium sulfate (HF residue), lime and fly ash. Mix I was composed of 30 percent fine calcium sulfate, 5 percent lime, and 65 percent fly ash; Mix II consisted of 50 percent fine calcium sulfate, 5 percent lime, and 45 percent fly ash; Mix III contained 65 percent fine calcium sulfate, 5 percent lime, and 30 percent fly ash; and Mix IV was composed of 75 percent coarse calcium sulfate, 5 percent lime, and 20 percent fly ash. The results of a full range of laboratory studies and field evaluation of the performance of the experimental base course sections over an 18-month period indicated that all of the mixtures tested generally had adequate strength and durability and performed well in service. It was thus concluded that such mixtures show good promise for use as pozzolanic base or sub-base courses, and relatively large percentages of waste calcium sulfate can be successfully used in such mixtures. Mixtures II and III exhibited slightly higher strength and durability in laboratory tests and somewhat better performance in the field compared to Mixtures I and IV.

In recent years, considerable attention has been given to the utilization of waste materials in highway construction to cope with local aggregate shortages, waste disposal costs, and environmental constraints. Fly ash, a by-product of the electric power industry, has been extensively researched over the past few decades, and the technology for its use in pozzolanic lime-fly ash-aggregate (LFA) base courses is fairly well-advanced (1,2). The actual use of such products, however, has not been very extensive, probably because of incomplete familiarity with field performance and relatively slow strength gain. Although fly ash utilization in general construction (concrete, lightweight aggregate, fills, etc.) increased significantly in the 1970s, vast quantities are still unused and undisposed of. Of the total 1978 power plant ash production of 68 million tons in the United States, only 24.3 percent was utilized, and it was projected that nearly one-half of the total production by 1990 would still have to be disposed of (3).

Waste sulfate is another material generated in large amounts and holding promise as a highway base course component. Included in the category of waste sulfate materials are mainly the products resulting from industrial effluent discharges (hydrofluoric acid and phosphoric acid production, fluidized bed combustion), flue gas desulfurization in power plants by scrubbers, and neutralization of acid mine drainage with lime. Annual production of waste sulfates in the United States have been estimated to be between 20 and 30 million tons (4,5), and a drastic increase is expected with a full implementation of flue gas desulfurization processes, and advent of fluidized bed plants. With the exception of hydrofluoric acid (HF) residue, such as that produced by the Allied Chemical Company's Nitro Plant, which is in solid form, all of these sulfate wastes are produced as sludges with varying percentages of solids that are composed of calcium sulfate, calcium sul-