USE OF FLORIDA PHOSPHOGYPSUM IN SYNTHETIC CONSTRUCTION AGGREGATE

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Abstract

In its role to provide technology to promote the efficient use of minerals and mineral process waste, the U.S. Bureau of Mines has conducted research to identify and develop high volume uses for phosphogypsum.

An asphalt-concrete containing 20 percent phosphogypsum met the Florida Department of Transportation specifications for their Type II, SAHM and ABC-1 asphalt concretes.

Mixtures containing up to 50 percent phosphogypsum, and the remainder clays, produced aggregate when fired at 1,000° C. Their compressive strengths exceeded 1,000 psi.

Mixtures containing a maximum of 50 percent phosphogypsum, 6 to 10 percent lime and the remainder fly ash had compressive strengths as high as 4,800 psi. The effects of the amounts of phosphogypsum, lime and fly ash were investigated. Also, the effects of fly ash to lime ratios were studied.

Mixtures of phosphogypsum with cement, cement-kiln-dust, silica powders and calcium chloride were also investigated. These were not considered useful for road construction.

Introduction

The phosphate industry in Florida is a vital segment of the Nation's economy and provides a critical mineral required for fertilizer production. In the 1980 Bureau of Mines' Mineral Yearbook, J. W. Pressler (22) reported that Florida supplied 83 percent of the domestic and 34 percent of the world's phosphate requirements. About 82 percent of the Florida phosphate is converted into phosphoric acid, which is used to make fertilizers.

Phosphogypsum and gypsum are both calcium sulfate dihydrate. The name phosphogypsum is used to designate the byproduct of wet-process phosphoric production, while gypsum refers to the natural mineral. Phosphate rock, which is composed of apatite minerals, (calcium phosphates containing varying amounts of carbonate and fluoride), is digested with sulfuric acid and water to produce phosphoric acid, phosphogypsum, and minor quantities of hydrofluosilicic acid.

A. May and J. W. Sweeney (20) reported that 335 million tons of phosphogypsum had accumulated in Florida in 17 phosphogypsum stockpiles or stacks. These piles occupied an average of 227 acres each and ranged from 30 to 140 ft in height. More phosphogypsum is being generated at a rate of about 33 million tons a year. By the year 2000, the projected accumulation will be over a billion tons of phosphogypsum.

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determined that phosphogypsum was not corrosive or toxic by the Environmental Protection Agency criteria, reported in the Federal Register (14), and that toxic elements and radium were not being leached from the stockpiles.

A. May and J. W. Sweeney (20) reported that the radium content of Florida phosphogypsum averages 21 pCi/g. On January 5, 1983, EPA issued its final standards to govern residual radioactivity at inactive uranium processing sites in the Federal Register (15). These restrict the radium activity to 5 pCi/g in the top 15 cm of surface layer and 15 pCi/g in layers below 15 cm. Similar standards may be extended to phosphogypsum. Thus, compositions containing up to 23.8 percent phosphogypsum may be used in surface layers and compositions containing up to 71.4 percent phosphogypsum may be used below 15 cm depth.

Gypsum used in the United States in 1980 for cement retarders, agricultural applications, fillers, plasters, and prefabricated products was 19.5 million tons; which includes only 0.66 million tons of phosphogypsum used in agriculture as reported by J. W. Pressler (22). Erlenstadt (13) discussed the use of phosphogypsum in cement retarders, fillers, plasters and prefabricated products. Florida phosphogypsum contains traces of phosphate and fluoride, which must be removed in order to use the phosphogypsum in these applications. Wheelock (26) reported on the manufacture of sulfuric acid made from phosphogypsum. Another use for phosphogypsum is in road construction, especially with lime and fly ash. Fly ash has been extensively used as a construction material. Hester (18) gave construction and performance data for approximately 200 miles of concrete pavement containing a small amount of fly ash constructed in south Alabama. The engineering properties, compositions, and performance of lime-fly ash mixtures were considered by Barenberg (11), and by the Transportation Research Board (24), in research sponsored by the American Association of State Highway and Transportation officials in cooperation with the Federal Highway Administration. Brink (12) gave details on a 200-acre parking lot paved with lime-fly ash-synthetic gypsum compositions, containing about 20 percent synthetic gypsum. The synthetic gypsum resulted from the manufacture of hydrofluoric acid, from acid mine drainage sludge, and from sulfur dioxide scrubber sludge. No phosphogypsum was used in these applications.

This investigation reports the use of phosphogypsum to make aggregate suitable for road construction. Its uses with asphalt, cement, cement-kiln-dust, silica, clays, oxycholorides, lime and fly ash are considered.

Acknowledgments

The authors wish to acknowledge the assistance of Dr. David P. Borris, Executive Director, Florida Institute of Phosphate Research for his advice and assistance and also to acknowledge the assistance of the Florida Department of Transportation for evaluating the synthetic phosphogypsum aggregate.

Materials Used

The phosphogypsum used was a sample obtained from a USS Agri-Chemicals' stockpile near Bartow, Florida. The analysis and characteristics of Florida phosphogypsum are fully described by A. May and J. W. Sweeney (20). The size distribution of phosphogypsum, dried at 45° C, is shown in figure 1, a plot of percent passing versus U.S. Standard sieve numbers. The scale used to locate the sieve numbers is \( \frac{4}{\log 2} [\log (\text{size, mm})] \) shown at the top of the graph. This scale was derived from the size openings of U.S. Standard sieves, which are the fourth root of 2 raised to an integer power specified in ASTM E-11.
Figure 1. - Particle size distribution of phosphogypsum dried at 45°C.
The scale is the integer. This plot has the same form as plotting against log (size, mm), but has the advantages that the scale is linear, the units are small integers, and the integers correspond exactly to the sieve numbers.

Two fly ash samples were used, one from Alabama and the other from Florida. The Alabama fly ash was from the Wilsonville, Alabama, steam plant and its production methods, quality control and characteristics were presented by Styron (23). The Florida fly ash was from Tampa Electric Company's Big Bend power plant. Analyses of these fly ashes are shown in Table 1. A commercial, high calcium, lime was also used.

Table 1. - Analyses of fly ash samples, percent

<table>
<thead>
<tr>
<th></th>
<th>Florida sample</th>
<th>Alabama sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon dioxide</td>
<td>47.1</td>
<td>48.9</td>
</tr>
<tr>
<td>Ferric oxide</td>
<td>24.2</td>
<td>14.9</td>
</tr>
<tr>
<td>Aluminum oxide</td>
<td>16.3</td>
<td>23.8</td>
</tr>
<tr>
<td>Calcium oxide</td>
<td>4.1</td>
<td>3.1</td>
</tr>
<tr>
<td>Magnesium oxide</td>
<td>0.7</td>
<td>1.1</td>
</tr>
<tr>
<td>Sulfur trioxide</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Water soluble</td>
<td>2.2</td>
<td>1.4</td>
</tr>
<tr>
<td>Moisture, weight loss at 110° C</td>
<td>.3</td>
<td>.3</td>
</tr>
<tr>
<td>Loss on ignition, weight loss at 750° C</td>
<td>1.6</td>
<td>1.9</td>
</tr>
<tr>
<td>Available alkali</td>
<td>.2</td>
<td>.3</td>
</tr>
<tr>
<td>Sieve analysis: plus 30 mesh U.S. sieve</td>
<td>.1</td>
<td>.0</td>
</tr>
<tr>
<td>plus 200 mesh U.S. sieve</td>
<td>7.5</td>
<td>5.9</td>
</tr>
<tr>
<td>minus 200 mesh U.S. sieve</td>
<td>92.4</td>
<td>94.1</td>
</tr>
</tbody>
</table>

Some tests were made using commercial ball clays, clay solids from phosphate rock beneficiation, cement-kiln-dust, commercial calcium chloride, and reagent grade calcium oxide, calcium hydroxide, and calcium chloride.

Criteria for Evaluating Samples

The criteria for evaluating samples of asphalt-concrete were free from ambiguity. The requirements, listed in the Florida Department of Transportation Specifications (16), were applied to phosphogypsum, sand, aggregate, asphalt mixtures. Requirements for aggregates are also listed in the Florida specifications.

Tests, such as the Los Angeles abrasion test, are needed for compliance with specifications, but are not suitable for screening large numbers of compositions, because of the quantities of materials needed. Unconfined compression strength was used to evaluate the samples because small samples sufficed and quantitative results were obtained which related to useful applications of aggregates. However, a specific value of compressive strength was needed as a criterion to indicate potentially useful products.

A study of 16 lime-fly ash pavements by the Transportation Research Board (24) gave the initial compressive strengths of the pozzolanic base materials; and the strengths and performance evaluations of the pavements after 1 to 8 years of service. If the pavements were poorly constructed, severely overloaded at an early age, or if the subbase was unstable; the pavements cracked and deteriorated under repeated loading. The over-all data indicated that an initial compressive strength of 1,000 psi gave a high quality pavement. Thus, a minimum strength of 1,000 psi was adopted in this investigation as the criterion to judge the phosphogypsum compositions. For comparison, the strength requirements of various mortars cured at 23° C, 95 percent relative humidity are shown in Table 2.
In addition to the compressive strength criterion, practical considerations were also employed. Thus, uses of expensive reagents, high energy requirements, or factors which would cause the final product to be non-competitive were also used to evaluate the potential usefulness of the products.

**Asphalt-Concrete**

These tests were performed to determine if phosphogypsum could be used to replace sand or fine aggregate in asphalt-concrete. Twenty mixes of various combinations of asphalt, phosphogypsum, sand, and coarse aggregate were tested.

The tests run were: stability, resistance to plastic deformation, and flow of asphalt-concrete by the Marshall apparatus, American Society for Testing and Materials (ASTM), standard method D1559 (9). Also bulk density, ASTM D1188 (6), maximum specific gravity, ASTM D2041 (10), and the percent air voids, ASTM D3203 (8) were determined. The optimum asphalt content and the voids in the mineral filler were derived from the test data.

The supplement to the Florida Department of Transportation Specifications (17) list requirements for 12 types of asphalt-concrete. There are four surface course mixes, S-I, S-II, Type II, and Type III; one patching mix, sand-asphalt hot mix, SAHM; three asphalt base course mixes, ABC-1, ABC-2, and ABC-3; and four wearing surface or friction course mixes, FC-1, FC-2, FC-3, and FC-4.

A mix containing 7.0 percent grade AC-20 asphalt and 93 percent mineral filler; composed of 20 percent phosphogypsum, dried at 150° C, 30 percent sand, and 50 percent limerock aggregate; met the requirements for asphalt-concrete for Type III, SAHM, and ABC-1 mixes. Thus, phosphogypsum could be used as a mineral filler in asphalt-concrete mixes for surface or base courses or for patching. However, none of the mixes met the requirements for wearing or friction courses. Table 3 shows the test results for the 20 percent phosphogypsum mix, and the requirements for Type III, SAHM, and ABC-1 mixes.

The maximum amount of phosphogypsum that proved satisfactory was only 20 percent. When higher percentages were tested, coating of all the aggregate particles with asphalt was incomplete, and the asphalt-concrete test results and the aggregate particle size distribution did not meet specifications. The particle sizes of the phosphogypsum are much smaller than the size requirements for aggregate in asphalt-concrete. This can be seen by comparing figure 1 and table 3. For example, 20 to 45 percent of the aggregate is required to pass the number 20.

The 20 percent refers to phosphogypsum dried at 150° C, the temperature normally used to dry aggregate in asphalt plants. Under these conditions phosphogypsum may dehydrate to the hemihydrate; and 20 percent of hemihydrate is equivalent to 23.7 percent of phosphogypsum.

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**Table 2. - Typical specifications for compressive strengths**

<table>
<thead>
<tr>
<th>Mortars</th>
<th>Compressive strength, 28 day, psi</th>
<th>ASTM¹ spec.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pozzolans...</td>
<td>600</td>
<td>C593 (2)</td>
</tr>
<tr>
<td>Natural cement</td>
<td></td>
<td>C10 (3)</td>
</tr>
<tr>
<td>Blended</td>
<td></td>
<td>C595 (1)</td>
</tr>
<tr>
<td>hydraulic cement...</td>
<td>1,200-3,500</td>
<td>C150 (4)</td>
</tr>
<tr>
<td>Portland cement...</td>
<td>3,200-4,000</td>
<td></td>
</tr>
<tr>
<td>Criterion adopted for this study...</td>
<td>1,000</td>
<td>NAP</td>
</tr>
</tbody>
</table>

NAP Not applicable.

¹American Society for Testing and Materials.
40 sieve for Type III, shown in table 3. Figure 1 shows that almost 80 percent of phosphogypsum passes the number 40 sieve. Therefore, only part of the sand can be replaced by phosphogypsum and retain the proper aggregate size distribution to produce a satisfactory asphalt-concrete.

Table 3. - Asphalt-concrete containing phosphogypsum

<table>
<thead>
<tr>
<th>Aggregate sizes</th>
<th>Percent passing</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sieve, U.S. std.</td>
<td>Observed</td>
<td>Type III</td>
</tr>
<tr>
<td>1/2 inch</td>
<td>100.0</td>
<td>100</td>
</tr>
<tr>
<td>3/8 inch</td>
<td>99.0</td>
<td>80-100</td>
</tr>
<tr>
<td>No. 4</td>
<td>72.5</td>
<td>65-100</td>
</tr>
<tr>
<td>No. 10</td>
<td>52.1</td>
<td>40-75</td>
</tr>
<tr>
<td>No. 40</td>
<td>39.1</td>
<td>20-45</td>
</tr>
<tr>
<td>No. 80</td>
<td>18.1</td>
<td>10-30</td>
</tr>
<tr>
<td>No. 200</td>
<td>7.0</td>
<td>2-10</td>
</tr>
</tbody>
</table>

Asphalt-concrete

<table>
<thead>
<tr>
<th>Tests</th>
<th>Observed</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stability, lb....</td>
<td>2,024</td>
<td>&gt;1,000</td>
</tr>
<tr>
<td>Flow, 0.01 inch..</td>
<td>14</td>
<td>8-16</td>
</tr>
<tr>
<td>Voids in mineral aggregate, percent........</td>
<td>20</td>
<td>&gt;15</td>
</tr>
<tr>
<td>Air voids, percent........</td>
<td>5</td>
<td>5-12</td>
</tr>
<tr>
<td>Asphalt content, percent........</td>
<td>7</td>
<td>4.5-9</td>
</tr>
<tr>
<td>Unit weight, pcf.</td>
<td>142</td>
<td>NAP</td>
</tr>
</tbody>
</table>

NAP Not applicable. No requirements listed.

Asphalt-concrete: 7 percent grade AC-20 asphalt, 93 percent aggregate. Aggregate: 20 percent phosphogypsum, 30 percent sand, 50 percent limerock stone.

Cement, Cement-Kiln-Dust and Silica Powder

These tests were performed to determine if the phosphogypsum could be used to replace sand in cement mortars, of if phosphogypsum would produce strong, hard materials with cement-kiln-dust or with silica powder. Kiln dust is essentially waste cement, so its use with byproduct phosphogypsum would be advantageous. The mixtures with silica powder were used in attempts to reproduce claims in a patent by R. C. Vickery (25). He claimed that a building material, having compressive strengths of 3,000 to 5,000 psi, could be made from compositions of 1 to 25 percent siliceous material and 2 to 10 percent lime with the remainder consisting of phosphogypsum; and variations and modifications of the formulation.

Various mixtures of phosphogypsum, cement, cement-kiln-dust, hemihydrate made from phosphogypsum, silica powder, lime and water were made into standard 2-inch cubes and tested by the ASTM method C109 (7) for compression strength. Typical results are shown in table 4. The compositions of the cement and the cement-kiln-dust mixtures were the same as that of Portland cement mortar used in the standard compression test; but with phosphogypsum replacing sand and kiln dust replacing cement. The composition using silica powder was one listed in the patent cited. These compositions are shown in table 4.

Table 4. - Cement, cement-kiln-dust, and silica powder mixtures containing phosphogypsum

<table>
<thead>
<tr>
<th>Age of test specimens, days</th>
<th>Compressive strength, psi</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Phosphogypsum plus</td>
</tr>
<tr>
<td></td>
<td>Kiln dust</td>
</tr>
<tr>
<td>---</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>60</td>
</tr>
<tr>
<td>7</td>
<td>230</td>
</tr>
<tr>
<td>28</td>
<td>420</td>
</tr>
</tbody>
</table>

1 65 percent phosphogypsum, 24 percent kiln dust, 11 percent water.
2 65 percent phosphogypsum, 24 percent type 2 cement, 11 percent water.
3 70 percent hemihydrate, 8 percent silica powder, 325 mesh, 3 percent lime, 19 percent water.
To determine if an accelerated reaction would occur between phosphogypsum and cement or cement-kiln-dust, 2-inch cubes, of the same compositions mentioned, were autoclaved at 200° C and 225 psi for 16 hours. The compressive strength of the cement mortar dropped to 290 psi, and that of the cement-kiln-dust to 200 psi. The autoclave contained more water after the samples were treated than the amount of water initially placed in the autoclave. This indicated that the phosphogypsum had dehydrated to hemihydrate with the release of water of hydration. Other mixes on autoclaving gave the same results - lowering of their strengths and release of water.

The strengths of the cement and silica powder compositions met the criterion, 1,000 psi, adopted for a useful aggregate. Cement-kiln-dust compositions failed this test. However, the strength of the cement-phosphogypsum mortar was less than half that required for Portland cement-sand mortars. Considering the relatively low strengths obtained, and the cost of cement and silica powder, further investigations using cement, cement-kiln-dust, or silica powders were curtailed.

Clays - Fired Samples

Briquettes, balls, and cylinders were prepared containing 25 percent, 50 percent, and 75 percent phosphogypsum and the remainder either commercial ball clay or clay from phosphate rock beneficiation. A minimum amount of water was added to form a plastic mass. The briquettes were hand formed in a mold 1.5- by 0.3- by 1-inch dimensions. The balls were hand formed about 0.5 to 0.75 inch diameter. The cylinders, 1 inch diameter by 1.3 inch length, were formed in a press under 8,000 psi and then extruded. All samples were dried at least 24 hours before firing.

The briquettes and cylinders were placed in a cold furnace, brought up to 600° C, 800° C, or 1,000° C and kept at these temperatures 1 hour, after which the furnace was turned off and the samples allowed to cool in the furnace. The balls were placed in a hot furnace at 600° C, 800° C, or 1,000° C for 15 minutes and then removed. Some briquettes were heated to 1,300° C.

The briquettes were used for visual determination of structural soundness and measurement of dimensional stability. Samples were sound at 600° C, 800° C, and 1,000° C and disintegrated at 1,300° C, probably because gypsum decomposes at the higher temperature. The higher temperatures produced lighter colors and gradual increase in shrinkage.

The balls were used to estimate the tolerances of the mixes to thermal shock. All samples remained intact when suddenly placed in the hot furnace as described. These specimens were hard and could be dropped from 6 to 10 feet on a hard surface with no damage.

The cylinders were used to measure the compressive strengths of the mixtures. These strengths are shown in table 5. The commercial ball clays with 25 percent phosphogypsum, fired at 600° C, 800° C, or 1,000° C, or with 50 percent phosphogypsum fired at 1,000° C may be suitable as an aggregate. Clay from phosphate rock beneficiation with 25 percent phosphogypsum fired at 800° C or 1,000° C or with 50 percent phosphogypsum fired at 1,000° C may also be suitable as an aggregate. The clays from the phosphate rock beneficiation and the phosphogypsum are both byproducts of the phosphate industry. Their use, in proportions up to 50 percent of each would utilize both materials. Energy considerations, to heat the materials, may be a detriment to their use to make an aggregate.

Calcium Oxychlorides

Solutions of soluble alkaline earth chlorides, sulfates, or nitrates react with calcium or magnesium oxide or
hydroxide to form oxy-compounds, such as calcium oxychloride, $3\text{CaO} \cdot \text{CaCl}_2 \cdot n\text{H}_2\text{O}$.

R. S. Kalyoncu, (19) reported on the applications of these types of compounds for refractories. The initial reaction occurs in minutes, followed by a slower reaction to form hard cementitious materials.

Table 5. - Clay mixtures containing phosphogypsum - fired samples

<table>
<thead>
<tr>
<th>Phosphogypsum, percent</th>
<th>Firing temperature, °C</th>
<th>Commercial Ball Clay</th>
<th>Clay from Phosphate Rock Beneficiation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>600</td>
<td>800</td>
<td>1,000</td>
</tr>
<tr>
<td>25</td>
<td>1,340</td>
<td>2,070</td>
<td>3,450</td>
</tr>
<tr>
<td>50</td>
<td>770</td>
<td>970</td>
<td>1,030</td>
</tr>
<tr>
<td>75</td>
<td>210</td>
<td>220</td>
<td>240</td>
</tr>
<tr>
<td>25</td>
<td>610</td>
<td>1,260</td>
<td>2,790</td>
</tr>
<tr>
<td>50</td>
<td>280</td>
<td>410</td>
<td>1,050</td>
</tr>
<tr>
<td>75</td>
<td>90</td>
<td>90</td>
<td>210</td>
</tr>
</tbody>
</table>

Numerous mixtures of reagent grade calcium hydroxide, calcium chloride, and water were made to select a uniform procedure to produce the oxychlorides. Of these mixtures, four were machined into cylinders 1-inch diameter and 1-inch height for compression tests. The strengths of these samples averaged 2,200 psi. An oxychloride, containing 20 percent phosphogypsum, had a compressive strength of 2,100 psi.

Three different compositions, containing 10 percent, 30 percent, and 50 percent phosphogypsum, and commercial lime and commercial calcium chloride were investigated. All compositions had a 3 to 1 molar ratio of lime to calcium chloride. Four batches of each composition were made, and the samples were cast into molds 1.5 inch diameter by 1.5 inch height. The samples were stored at approximately 25° C and 75 percent relative humidity until tested for their compressive strengths, which are shown in table 6. Each value in table 6 is the average of four batches.

The results showed that the strengths were very low, and decreased with age and with increased phosphogypsum content. The samples prepared from both reagent grade and commercial chemicals were also tested for their stability in water. The samples cracked and deteriorated extensively within several months.

<table>
<thead>
<tr>
<th>Phosphogypsum, percent</th>
<th>Compressive strength, psi</th>
<th>Curing time, days</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>7</td>
<td>28</td>
</tr>
<tr>
<td>10</td>
<td>450</td>
<td>416</td>
</tr>
<tr>
<td>30</td>
<td>270</td>
<td>256</td>
</tr>
<tr>
<td>50</td>
<td>188</td>
<td>239</td>
</tr>
</tbody>
</table>

NA Not available. Samples cracked on curing.

Moles Ca(OH)$_2$/moles CaCl$_2=3$.

Averages of 4 batches of 3 samples per batch.

At 23° C and 95 percent Rh.

The objective of this investigation was the practical development of an aggregate from phosphogypsum. Thus, we did not investigate why commercial chemicals gave products whose strengths were so much lower than the products obtained with reagent grade chemicals. Because of the low strengths and the instability of the products in water, further work on oxychlorides was discontinued.

Fly Ash

As mentioned in the introduction; a lime-fly ash-synthetic gypsum mixture was used to pave a 200 acre parking lot. This lot was used for only 1 week during an exposition, and insufficient time was available to adequately prepare the surface, design the mixture, or to evaluate the results. Although the pavement served its purpose much of it was damaged by traffic. The project did not clearly establish the factors involved in using synthetic gypsum with lime and fly ash.

A systematic investigation of the effects of lime, fly ash and
phosphogypsum on the strengths of aggregate mixtures was pursued in this research effort. Standard 2-inch cubes of various compositions were tested by the ASTM method C109 (7) for compressive strengths.

As reported in the materials section, two fly ash samples were available. These were compared by measuring the strengths of the mixtures, containing 40 percent phosphogypsum, 10 percent lime and 50 percent of each fly ash, on a dry basis; plus a minimum amount of water to form mortars. The average results are shown in table 7.

Table 7. - Compressive strengths of Alabama and Florida fly ash compositions

<table>
<thead>
<tr>
<th>Fly ash</th>
<th>Compressive strength, psi</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Curing time, days¹</td>
</tr>
<tr>
<td></td>
<td>7</td>
</tr>
<tr>
<td>Alabama...</td>
<td>180</td>
</tr>
<tr>
<td>Florida...</td>
<td>310</td>
</tr>
</tbody>
</table>

¹At 23°C and 95 percent relative humidity.

The strengths of the mixtures containing either fly ash would be adequate for aggregate. However, the remaining tests were made using the Florida fly ash because of its proximity to Florida sources.

A series of tests were performed on mixtures with fly ash:lime ratios of 3:1, 5:1 and 10:1; each containing 20 to 80 percent phosphogypsum. Another series of tests were run on mixtures containing 35 percent phosphogypsum, 0-25 percent lime, and the remainder fly ash. The fly ash:lime ratios necessarily differed for each of these mixtures. Compressive strengths were determined on each specimen after curing for 7, 28 and 84 days at 23°C and 95 percent relative humidity.

Compressive strengths of the mixtures with the 3:1 fly ash:lime ratio are shown in table 8 and shown in figure 2. Figure 2 shows that the compressive strength increases with time and with a decrease in the percentage of phosphogypsum.

Table 8. - 3:1 fly ash:lime mixtures containing phosphogypsum

<table>
<thead>
<tr>
<th>Test No.</th>
<th>G</th>
<th>L</th>
<th>F</th>
<th>Compressive strength, psi</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>G</td>
<td>L</td>
<td>F</td>
<td>Curing time, days¹</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>28</td>
<td>84</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>20</td>
<td>20.00</td>
<td>60.00</td>
<td>420 2,360 4,010</td>
</tr>
<tr>
<td>2</td>
<td>35</td>
<td>16.25</td>
<td>48.75</td>
<td>230  2,100  3,540</td>
</tr>
<tr>
<td>3</td>
<td>50</td>
<td>12.50</td>
<td>37.50</td>
<td>160  1,370  3,110</td>
</tr>
<tr>
<td>4</td>
<td>65</td>
<td>8.75</td>
<td>26.25</td>
<td>100   670  1,650</td>
</tr>
<tr>
<td>5</td>
<td>80</td>
<td>5.00</td>
<td>15.00</td>
<td>60   310   540</td>
</tr>
</tbody>
</table>

Compressive strengths of the 5:1 fly ash:lime mixtures are shown in table 9 and shown in figure 3; and the results for the 10:1 ratio are shown in table 10 and figure 4. Results for both of these ratios, show the same trends as in figure 2--an increase in strength with time and with a decrease in the phosphogypsum content.

Table 9. - 5:1 fly ash:lime mixtures containing phosphogypsum

<table>
<thead>
<tr>
<th>Test No.</th>
<th>G</th>
<th>L</th>
<th>F</th>
<th>Compressive strength, psi</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>G</td>
<td>L</td>
<td>F</td>
<td>Curing time, days¹</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>28</td>
<td>84</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>0</td>
<td>16.67</td>
<td>83.33</td>
<td>320  1,918  2,690</td>
</tr>
<tr>
<td>7</td>
<td>20</td>
<td>13.33</td>
<td>66.67</td>
<td>280   2,980  4,840</td>
</tr>
<tr>
<td>8</td>
<td>35</td>
<td>10.83</td>
<td>54.17</td>
<td>260   1,950  3,670</td>
</tr>
<tr>
<td>9</td>
<td>50</td>
<td>8.33</td>
<td>41.67</td>
<td>210   1,730  2,330</td>
</tr>
<tr>
<td>10</td>
<td>65</td>
<td>5.83</td>
<td>29.16</td>
<td>140   730    790</td>
</tr>
<tr>
<td>11</td>
<td>80</td>
<td>3.33</td>
<td>16.67</td>
<td>80    290    310</td>
</tr>
</tbody>
</table>

Compressive strengths of the 10:1 fly ash:lime mixtures are shown in table 10 and figure 4; and the results for the 3:1 ratio are shown in table 8 and figure 2. Results for both of these ratios, show the same trends as in figure 1--an increase in strength with time and with a decrease in the phosphogypsum content.
Figure 2. - Compressive strength versus time 3:1 fly ash:lime ratio.
Figure 3. - Compressive strength versus time 5:1 fly ash:lime ratio.
Figure 4. - Compressive strength versus time 10:1 fly ash:lime ratio.
Table 10. - 10:1 fly ash:lime mixtures containing phosphogypsum

<table>
<thead>
<tr>
<th>Test No.</th>
<th>G (percent phosphogypsum)</th>
<th>L (percent lime)</th>
<th>F (percent fly ash)</th>
<th>Compressive strength, psi</th>
<th>Curing time, days'</th>
</tr>
</thead>
<tbody>
<tr>
<td>12</td>
<td>20</td>
<td>7.27</td>
<td>72.73</td>
<td>480</td>
<td>2,520</td>
</tr>
<tr>
<td>13</td>
<td>35</td>
<td>5.91</td>
<td>59.09</td>
<td>410</td>
<td>1,390</td>
</tr>
<tr>
<td>14</td>
<td>50</td>
<td>4.55</td>
<td>45.45</td>
<td>110</td>
<td>760</td>
</tr>
<tr>
<td>15</td>
<td>65</td>
<td>3.18</td>
<td>31.82</td>
<td>80</td>
<td>260</td>
</tr>
<tr>
<td>16</td>
<td>80</td>
<td>1.82</td>
<td>18.18</td>
<td>40</td>
<td>50</td>
</tr>
</tbody>
</table>

At 23° C and 95 percent relative humidity.

G = percent phosphogypsum, dry basis.
L = percent lime, dry basis.
F = percent fly ash, dry basis.

The change in strength with time is indicated by the slopes of the lines in figures 2, 3, and 4. In figure 2, at 80 percent phosphogypsum; in figure 3, at 65 and 80 percent phosphogypsum; and in figure 5, at 35, 50, 65, and 80 percent phosphogypsum, the slopes are small between 28 and 84 days.

This data indicated small changes in strength that occurred after 28 days. Each of these "flat" lines represent mixtures having less than 6 percent lime.

The time for the mixtures to reach 1,000 psi is about the same for each fly ash:lime ratio for the same percent phosphogypsum. From the intersections of the lines with 1,000 psi on figures 2, 3, 4, we obtain times shown in table 11.

The curing time ranges from 2 to 7 weeks for compositions achieving greater than 1,000 psi in 28 days.

The change in compressive strength as a function of phosphogypsum content is illustrated in figure 5; as a function of fly ash content in figure 6 and as a function of lime content in figure 7. These figures also indicate effects of fly ash:lime ratios.

Table 11. - Times for lime, fly ash phosphogypsum compositions to reach 1,000 psi

<table>
<thead>
<tr>
<th>Phosphogypsum,</th>
<th>Average time to reach 1,000 psi, day</th>
</tr>
</thead>
<tbody>
<tr>
<td>percent</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>13</td>
</tr>
<tr>
<td>35</td>
<td>17</td>
</tr>
<tr>
<td>50</td>
<td>20</td>
</tr>
<tr>
<td>65</td>
<td>47</td>
</tr>
<tr>
<td>80</td>
<td>NAp</td>
</tr>
</tbody>
</table>

NAp Not applicable, does not reach 1,000 psi.

'Averages for compositions whose strengths reached 1,000 psi.

The trends in compressive strengths appear to be well defined from figures 5, 6, and 7. For example, figure 5 shows that the 5:1 ratio is almost a straight line from 20 to 65 percent phosphogypsum. The correlation coefficient for this line is -0.998. However, the percent phosphogypsum, plus lime, plus fly ash, equals 100. These components are not independent variables. Higher phosphogypsum content necessarily requires lower lime and fly ash content. Thus, it is not certain if the lower strengths with increasing phosphogypsum content, shown in figure 5, were due to phosphogypsum, lime, or fly ash.

The 3:1, 5:1, and 10:1 fly ash:lime ratios gave different compressive strengths. To show how these ratios affected the strengths, they are compared in figure 8. The strengths are greatest for 20 percent phosphogypsum and least for the 80 percent phosphogypsum for all ratios.

Figure 8 shows a distinctive feature of the data. The 20 and 35 percent phosphogypsum mixtures increased in strength from the 3:1 to the 5:1 ratios while the 50, 65, and 80 percent phosphogypsum mixtures decreased in strength. Overall consideration of the data indicated that this drastic change was due to the lime content.
Figure 5. - Compressive strength versus percent phosphogypsum.
Figure 6. - Compressive strength versus percent fly ash.
Figure 7. - Compressive strength versus percent lime.
Figure 8. - Compressive strength versus fly ash:lime ratio.
Compressive strengths, as a function of lime content, with a constant phosphogypsum content, are shown in table 12. The changes in the strengths of these mixtures with time are shown in figure 9, which indicated that the lime requirements are between 5 and 10 percent.

Table 12. - Thirty-five percent phosphogypsum mixtures containing 0 to 25 percent lime

<table>
<thead>
<tr>
<th>Test No.</th>
<th>L</th>
<th>F</th>
<th>F:L</th>
<th>Curing time, days</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>7</td>
</tr>
<tr>
<td>17</td>
<td>0</td>
<td>65</td>
<td>N/A</td>
<td>90</td>
</tr>
<tr>
<td>18</td>
<td>5</td>
<td>60</td>
<td>12.00:1</td>
<td>190</td>
</tr>
<tr>
<td>19</td>
<td>10</td>
<td>55</td>
<td>5.50:1</td>
<td>260</td>
</tr>
<tr>
<td>20</td>
<td>15</td>
<td>50</td>
<td>3.33:1</td>
<td>250</td>
</tr>
<tr>
<td>21</td>
<td>20</td>
<td>45</td>
<td>2.25:1</td>
<td>230</td>
</tr>
<tr>
<td>22</td>
<td>25</td>
<td>40</td>
<td>1.60:1</td>
<td>400</td>
</tr>
</tbody>
</table>

N/A: Not applicable.
1At 23°C and 95 percent relative humidity.

L - percent lime, dry basis.
F - percent fly ash, dry basis.
F:L - wt ratio of fly ash to lime.

Strengths as a function of percent lime is shown in figure 10 for mixtures containing 35 percent phosphogypsum. Figure 10 contains data from tables 8, 9, 10, and 12; and also indicates fly ash:lime ratios. Maximum strength was obtained with a 5:1 fly ash:lime ratio and 10.8 percent lime. The fly ash:lime ratio and percent lime for maximum strength differs from these values when the percent phosphogypsum is different from 35 percent.

Figure 10 indicates that to develop adequate strength, the fly ash:lime ratio should be between 3 and 10 and the percent lime should be between 6 and 11 percent. Mixtures containing more than 65 percent phosphogypsum would be excluded, since all samples with 6 percent or more of phosphogypsum had low strengths. The range of compositions for adequate strength are shown in table 13.

Table 13. - Range of compositions of lime, fly ash, phosphogypsum to produce aggregate strengths of 1,000 psi

<table>
<thead>
<tr>
<th>Percent</th>
<th>Min.</th>
<th>Max.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phosphogypsum</td>
<td>34</td>
<td>56</td>
</tr>
<tr>
<td>Lime</td>
<td>6</td>
<td>11</td>
</tr>
<tr>
<td>Fly ash</td>
<td>33</td>
<td>60</td>
</tr>
<tr>
<td>Fly ash:lime ratio</td>
<td>3</td>
<td>10</td>
</tr>
</tbody>
</table>

A mixture containing 10 percent lime, 40 percent phosphogypsum and 50 percent fly ash was tested for abrasion by the Los Angeles abrasion test. The loss was less than 50 percent, meeting the requirement of the Florida Department of Transportation Specifications. Unit weight of the aggregate was 106 lb/cu ft, also meeting the specifications.

Tests were conducted to evaluate the swelling properties of lime, fly ash, phosphogypsum compositions when immersed in water. The expansion of specimens, immersed in water 28 days, ranged from 0.31 to 4.18 percent, with greater expansion corresponding to higher lime or phosphogypsum contents. Preliminary results indicate that the lime content must be 10 percent or less, and the phosphogypsum content must be less than 20 percent to limit swelling to less than 1 percent.

Discussion

This investigation showed that the use of phosphogypsum with cement, cement-kiln-dust, silica powder, or calcium oxychlorides was not practical to make aggregates. The products obtained had low strengths, or stability, or would be expensive to produce.

Phosphogypsum can be used as a mineral filler in asphalt-concrete. However, only about 20 percent phosphogypsum can be used because its particle size is too small to replace all the fine aggregate in these mixtures.
Figure 9. - Compressive strength versus time 35 percent phosphogypsum.
Figure 10. - Compressive strength versus percent lime, with 35 percent phosphogypsum.
Mixtures of clays and phosphogypsum produce aggregate when fired at 800-1,000° C. The cost of firing the mixes may be a detriment to this use. However, clays from phosphate beneficiation and phosphogypsum are both waste products of the phosphate industry, and both could be utilized in this application.

Mixtures of fly ash, lime and phosphogypsum produced aggregates having compressive strengths near 5,000 psi, requiring no energy other than mixing. These mixtures may contain as much as 50 percent phosphogypsum. However, they are slow curing, requiring from 2 to 7 weeks to reach a strength of 1,000 psi and about 3 months to develop considerable strength.

The strength decreases with an increase in phosphogypsum content and increases with an increase in fly ash content. For adequate strength the lime content should be about 6 to 11 percent. Below 6 percent lime, strengths are low, and above 11 percent lime, strengths remain about the same. The fly ash:lime ratios may vary from 3:1 to 10:1. The strengths decrease when this ratio is greater than 5:1. However, the higher ratios require less lime and may be more economical than the lower ratios.

The swelling characteristics of the lime, fly ash, phosphogypsum compositions may limit the amount of phosphogypsum that can be used in these mixtures.

Conclusions

Asphalt-concrete containing 7 percent grade AC-20 asphalt and 93 percent aggregate; consisting of 20 percent phosphogypsum, 30 percent sand and 50 percent limerock stone; met the Florida Department of Transportation Specifications (17) for Type III, SAHM, and ABC-1 asphalt-concrete.

Mixtures containing 25 and 50 percent phosphogypsum and the remainder clay from phosphate rock beneficiation produced aggregate when fired at 1,000° C for 1 hour. The compressive strengths exceeded 1,000 psi.

Coarse aggregate made from mixtures of lime, fly ash and phosphogypsum, obtained maximum compressive strengths of 4,800 psi. Mixtures containing a maximum of 50 percent phosphogypsum 6 to 10 percent lime, 3:1 to 10:1 fly ash:lime ratio and a minimum of 38 percent fly ash produced aggregate with 28-day compressive strengths exceeding 1,000 psi.

References


APPENDIX

Reviewers' Comments and USBM Rebuttal
Comments on the Final Report on

“Use of Florida Phosphogypsum in Synthetic Construction Aggregate”

1. This report is a good preliminary study on the use of Florida phosphogypsum in synthetic construction aggregates. The findings of the report indicated that phosphogypsum mixtures can be successfully used as synthetic construction aggregates.

2. A minimum compression strength of 1000 psi, which was adopted in this study as the criterion to judge the phosphogypsum compositions, tends to limit the scope of the study and the use of phosphogypsum in highway construction. It may be desirable for the highway base course to have the compressive strength of 1000 psi. The requirement for compressive strength for subbase course materials is certainly much lower than 1000 psi.

3. A follow-up study of the mixture of phosphogypsum with fly ash and lime is necessary to implement the use of the mixture in highway construction. Optimum water content and fatigue testing should be studied for the phosphogypsum mixture.

4. One of the findings as stated in in the report, "...For adequate strength the lime content should be about 6 to 11 percent. Below 6 percent lime, strengths are low...." is contradictory to Dr. Don Saylak’s findings as reported in the ASTM meeting on Gypsum held in Atlanta, Georgia, on April 14-15, 1983, and the results obtained at the University of Miami. Judging from the scope of this study, the reviewer believes that it is premature to make such a statement.
Dr. David Borris, Executive Director
Florida Institute of Phosphate Research
1855 West Main Street
Bartow, Florida 33830

Subject: FIPR #81-01-008

Dear Dr. Borris:

We have reviewed the Research Project FIPR #81-01-008 on "Use of Florida Phosphogypsum in Synthetic Construction Aggregate."

The statements made in the report regarding the use of this synthetic aggregate in asphalt concrete are misleading. Specifically, on Page 1, the second paragraph of the Abstract, although the mix may have met the Marshall design requirements, there is no indication the aggregate itself met the requirements of the Florida Department of Transportation Standard Specifications. Similar synthetic aggregates had been evaluated by this Bureau and were found to degrade in water. Attached is a copy of the letter to Alexander May indicating the findings.

Strengths sufficient for base structures can be attained using phosphogypsum in combination with lime and fly ash. However, expansion seems to be a problem. Durability was not considered, thus considerably more development work in the lab, as well as in the field, is required before the usefulness of this material as a base structure can be properly evaluated.

We appreciate the opportunity to comment, since road construction materials are under constant review by this Bureau for the Department of Transportation, and we trust that this review will be of some interest to your organization.

Very truly yours,

L. L. Smith, P.E.
Deputy State Materials and Research Engineer

LLS: ay
cc: Dr. K. H. Ho
Attachment
Mr. Alexander May
Bureau of Mines
Tuscaloosa Research Center
P. O. Box L
University, Alabama 35486-9777

Subject: Phospho-gypsum Synthetic Aggregate

Dear Mr. May;

We have reviewed the test results sent to us and based on additional tests in our laboratory, we find that the aggregate sent to us for evaluation is not suitable for use in asphalt concrete due to its moisture susceptibility.

Attached is a summary of the tests performed. It should be noted, as discussed with you over the phone, that the water immersion of the Marshall specimens is not a standard test. Additionally, samples of the crushed synthetic aggregate were immersed in water and degradation of the aggregate was observed.

It is possible that your tests using cubes of the synthetic are not representative of the surface of the crushed material. The cubes could have a "glazed" surface preventing the intrusion of water where the crushed surface is open and porous appearing.

We hope that this information will assist you in your development process.

Very truly yours,

G. C. Page
Bituminous Materials and Pavement Evaluation Engineer

GCP: cjg

Attachment

CC: Mr. C. F. Potts
    Mr. L. L. Smith
    Mr. K. H. Ho
    Mr. K. H. Murphy
<table>
<thead>
<tr>
<th>Co.</th>
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<th>Job No.</th>
<th>Road</th>
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<td></td>
<td>0824</td>
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**Material:** Phosphogypsum (Synthetic aggregate)  
**Identification marks:**  
**Quantity represented:** 60 pounds  
**Producer:** Mineral Aggregate, Inc.  
**Source of supply:** Producer  
**Submitted by:** Alexander May, Bureau of Mines, Ala.  
**Intended use:** Evaluation for use in Asphalt Concrete Mixtures  
**Sampled by:** Producer  
**Sampled from:**  
**Date sampled:**  
**Date received:** 8-26-82  
**Date tested:** 10-11-82  
**Date reported:** 10-28-82

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**TEST RESULTS**

**Chemical Analysis:**

Total Sulfur  
Phosphate as $P_2O_5$

5.4%  
0.6%

**L.A. Abrasion:**

$C = 39.1\%$ (max. allowable = 35%)  
$D = 34.9\%$ (max. allowable = 35%)

**Effects of Water on Cohesion of Compacted Bituminous Mix:**

Marshall specimens were compacted using 50% phosphogypsum aggregate and 6% AC-20. The specimens were then immersed in water for 72+ hours at ambient temperatures. After approximately 24 hours, expansion of the specimens was determined to be approximately 5% and a light colored substance was draining from the specimens, which appeared to be some form of oxide.

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**c.c:** Mr. G. C. Page  
Mr. K. H. Murphy  
Cen Bit Lab (2)

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Tested by...
REPLIES TO REVIEW OF MANUSCRIPT

Manuscript: "Use of Florida Phosphogypsum in Synthetic Construction Aggregate"

Comment: 1. This report is a good preliminary study on the use of Florida phosphogypsum in synthetic construction aggregates. The findings of the report indicated that phosphogypsum mixtures can be successfully used as synthetic construction aggregates.

Reply: The report does indicate that phosphogypsum mixtures have a potential for use as a construction aggregate. However, additional research, development and field testing would be required.

Comment: 2. A minimum compression strength of 1000 psi, which was adopted in this study as the criterion to judge the phosphogypsum compositions, tends to limit the scope of the study and the use of phosphogypsum in highway construction. It may be desirable for the highway base course to have the compressive strength of 1000 psi. The requirement for compressive strength for subbase course materials is certainly much lower than 1000 psi.

Reply: The comment is valid, the criterion of a minimum 1,000 psi tends to limit the scope of the study for evaluating subbase or base materials. The criterion was adopted as a working guide based on the strengths reported to give a high quality pavement as reported on page 4 of the manuscript. We were principally interested in finding a substitute for hard-rock aggregate.

Comment: 3. A follow-up study of the mixture of phosphogypsum with fly ash and lime is necessary to implement the use of the mixture in highway construction. Optimum water content and fatigue testing should be studied for the phosphogypsum mixture.

Reply: We agree that more testing would be required. The Bureau of Mines did investigate the swelling properties of these mixes, but priorities within the Bureau prevented further research in this area.

Comment: 4. One of the findings as stated in the report, For adequate strength the lime content should be about 6 to 11 percent. Below 6 percent lime, strengths are low...." is contradictory [sic] to Dr. Don Saylak's findings as reported in the ASTM meeting on Gypsum held in Atlanta, Georgia, on April 14-15, 1983, and the results obtained at the University of Miami. Judging from the scope of this study, the reviewer believes that it is premature to make such a statement.
Reply: The statement cited was based on the results obtained and is valid. The authors discussed the seeming contradiction with Dr. Saylak, at the meeting cited, and during his visit to the Bureau in November, 1983. The fly ash used by Dr. Saylak contained 24.0 to 25.4 percent total calcium oxide (lime) while the Bureau of Mines used fly ash containing only 4.1 percent calcium oxide. Thus, Dr. Saylak did not have to add lime since it was already present in his fly ash.

Dr. Saylak was also seeking strengths to meet Texas highway specifications of 100 psi for stabilized subbases or 650 psi for cement stabilized bases, while the Bureau was seeking strengths in excess of 1000 psi. Actually, there is no contradiction involved when reviewing the data.

Comment: The statements made in the report regarding the use of this synthetic aggregate in asphalt concrete are misleading. Specifically, on Page 1, the second paragraph of the Abstract, although the mix may have met the Marshall design requirements, there is no indication the aggregate itself met the requirements of the Florida Department of Transportation Standard Specifications. Similar synthetic aggregates had been evaluated by this Bureau and were found to degrade in water. Attached is a copy of the letter to Alexander May indicating the findings.

Reply: The authors appreciate the cooperation of the Florida Department of Transportation (FDT) and the letter cited in the comment. The Bureau of Mines agreed with the FDT conclusions that the Bureau's synthetic aggregate "is not suitable for use in asphalt concrete due to its moisture susceptibility." This preliminary evaluation by the FDT assisted us in redirecting our research efforts.

The reviewers' comment states "The statements made in the report regarding the use of this synthetic aggregate in asphalt concrete are misleading." However, there are NO statements in the report on using the Bureau of Mines' synthetic aggregate in asphalt concrete!

The statement referred to in the abstract and also in the conclusions, referred to the asphalt concrete containing phosphogypsum not synthetic aggregate. This asphalt concrete was made using Florida limerock and sand, commercially used in Florida road construction plus phosphogypsum. This mixture met FDT specifications for Type II, SAHM, and ARC-1 asphalt concrete. The phosphogypsum was substituted for part of the fine aggregate in the mix. The Bureau of Mines also immersed this Marshall test specimen in water and it showed no swelling, bleeding or detrimental effects. The Bureau of Mines will not print misleading or inaccurate statements in its reports, only statements of fact, based on research evidence presented.
Comment: Strengths sufficient for base structures can be attained using phosphogypsum in combination with lime and fly ash. However, expansion seems to be a problem. Durability was not considered, thus considerably more development work in the lab, as well as in the field, is required before the usefulness of this material as a base structure can be properly evaluated.

Reply: We agree, as previously stated (3rd reply). We did investigate swelling of these mixes but were unable to continue further research because of the project being terminated, due to a change in research priorities made by our Headquarters Office.

These replies are respectfully submitted to help clarify any misunderstandings or statements in the manuscript.

Alexander May

Alexander May