

PHOSPHATE FERTILIZER CHARACTERISTICS

Storage and Drilling Characteristics of High-Alumina Nitric Phosphate Prepared from Florida Leached-Zone Ore

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In current practice leached-zone phosphate material is discarded in Florida land-pebble mining operations. A possible use of this waste material is in the manufacture of high-alumina fertilizers with nitric acid treatment. To provide information on the storage and drilling characteristics of fertilizers produced in this manner, tests were made on two lots of experimental material produced by the Tennessee Valley Authority. Bag-storage properties were very good and drilling properties superior to nitric phosphates made from high grade land pebble previously tested. The results indicate that fertilizers of good physical quality can be prepared from the leached-zone ore.

THE HIGH GRADE BEDS of Florida land-pebble phosphate are overlain by beds of low grade, or leached-zone, phosphate material, which is discarded in mining operations. A possible use for this waste material is in the manufacture of fertilizer by nitric acid treatment. Nitric phosphate (also called nitrophosphate and nitrphosphate) prepared from leached-zone ore is characterized by a relatively high content of aluminum and iron in comparison with material similarly produced from commercial Florida land-pebble phosphate. The presence of substantial amounts of aluminum and iron phosphates could have a measurable influence on the physical character of the product. In order to provide information on this aspect of the technology of nitric phosphate, storage and drillability tests were made on two lots of the leached-zone product. The tests and results are described here. The nutrient value of nitric phosphates is given by Starostka, Norland, and MacBride (7).

Materials

Two lots of 11-22-11 leached-zone nitric phosphate, prepared by the Tennessee Valley Authority, were tested. Chemical and screen analyses of these materials are given in Table I, which includes, for comparison of chemical composition, data on a similar nitric phosphate made from land-pebble rock.

Procedure

Bag-Storage Tests. Storage periods of 1, 3, 6, and 11 months have been completed. For each of the first two storage periods (1 and 3 months), three

piles of each lot were stored with two 75-pound bags of fertilizer in the third and ninth positions from the top of a 10-bag pile. The other eight bags each contained 100 pounds of sand, thus providing storage weights of 200 and 775 pounds on the test bags in the third and ninth positions, respectively. For the 6- and 11-month storage periods, three piles of lot 51-2 and three piles of lot 51-1 were stored, respectively.

The materials were packaged in six-ply paper bags having two asphalt-laminated layers.

The bags removed from storage were weighed, dropped from waist level four times, once on each edge and each side,

Table II. Temperature and Humidity Conditions during Storage

| Storage Period, Months | Temperature, ° F. | | | | | | Relative Humidity, % | | |
|------------------------|-------------------|----------------|----------------|------|-----|----------------|----------------------|-----|--|
| | Av. for period | Av. daily max. | Av. daily min. | High | Low | Av. for period | High | Low | |
| | | | | | | | | | |
| 0-1 | 44 | 50 | 37 | 60 | 20 | 68 | 100 | 34 | |
| 2-3 | 53 | 60 | 46 | 77 | 31 | 68 | 98 | 27 | |
| 1-3 | 50 | 57 | 43 | 77 | 20 | 68 | 100 | 27 | |
| 3-6 | 72 | 80 | 64 | 92 | 48 | 71 | 96 | 39 | |
| 1-6 | 61 | .. | .. | 92 | 20 | 69 | 100 | 27 | |

Table I. Chemical and Screen Analyses of Nitric Phosphates

| TVA No. | Chemical Analysis, % by Weight | | | | | | | | | | | | | | Screen Analysis, % Passing U. S. Screen No. | | | | | |
|--------------------------------|--------------------------------|-----------------|-------------------------------|-------------------|------------------------------|------------------|------|--------------------------------|--------------------------------|-----|-----------------|-----|------------------|-----|---|-----|-----|-----|----|----|
| | Nitrogen | | P ₂ O ₅ | | | | | AOAC | | | | | | | 6 | 8 | 12 | 20 | 40 | 50 |
| | Total | NH ₃ | Total | C.I. ^a | WS ^b , % of total | K ₂ O | CaO | Al ₂ O ₃ | Fe ₂ O ₃ | F | SO ₄ | Cl | H ₂ O | | | | | | | |
| Leached-Zone Nitric Phosphates | | | | | | | | | | | | | | | | | | | | |
| 51-1 | 11.4 | 6.9 | 23.6 | 0.5 | 20 | 11.8 | 4.7 | 9.6 | 1.3 | 0.8 | 1.0 | 8.8 | 1.2 | 100 | 88 | 60 | 9 | 0 | .. | |
| 51-2 | 11.6 | 7.0 | 22.4 | 0.4 | 22 | 11.0 | 4.7 | 9.0 | 1.2 | 1.1 | 1.0 | 8.4 | 3.4 | 100 | 87 | 60 | 8 | 0 | .. | |
| Land-Pebble Nitric Phosphate | | | | | | | | | | | | | | | | | | | | |
| 45 | 12.1 | ... | 23.5 | 0.9 | 27.7 | 11.7 | 15.0 | 0.6 | 0.1 | ... | 0.7 | 9.2 | ... | ... | ... | 100 | ... | ... | 0 | |

^a Citrate-insoluble.

^b Water-soluble.

opened, and screened through a three-mesh screen.

The temperature and relative humidity as recorded by a hygrothermograph in the storage area appear in Table II.

Drillability Tests. The equipment used for the tests consisted of a drill-hopper mounted so as to deliver fertilizer to a variable-speed, endless belt 37 feet long. The hopper and belt speeds were adjusted to conform to an application rate equivalent to 300 pounds per acre, based on a distributor speed of 3 miles per hour, and a distance of 40 inches between rows. Rubber strips were vulcanized on the belt to form 34 6-inch sections in which the fertilizer was collected. Comparative weights of fertilizer from the different sections of the belt indicated the uniformity of drilling rate.

Drillability tests were made on material from freshly opened bags of both lots in a 16 × 24 foot air-conditioned room. The drilling rates of these materials were measured. Each lot was further tested by exposing a 1/4-inch layer of the fertilizer on trays to a relative humidity of 84 to 88% and a temperature of 74° to 76° F. for an hour, and then determining the new drilling rate. Racks were used to hold the trays, one above the other with a 5-inch space between each tray. Air was circulated over the trays by a fan. This procedure of exposing the fertilizer for an hour and then measuring the drilling rate was continued until the total exposure was 5 hours. After each hour's exposure, the angle of repose was measured, and the coefficient of variation was calculated from the weights of fertilizer collected on 25 consecutive 6-inch sections of the belt.

Performance of Test Materials

Storage. Both lots of fertilizer removed from storage after 1, 3, 6, and 11 months were in very good condition. No lumps were found in any bag. The moisture in the six bags of lot 51-1 averaged 2.63% after 1 month, 2.72% after 3 months, and 4.32% after 11 months. The moisture in the six bags of lot 51-2 averaged 3.62% after 1 month, 3.71% after 3 months, and 4.34% after 6 months. These moisture values are on the wet basis as determined by the air-flow method.

None of the bags showed any appreciable change in weight after 1 month of storage. At the end of 3 months, three fourths of the bags showed a slight increase in weight of about 0.5 pound and the remainder of the bags did not change in weight. At the end of 6 months, each bag had gained from 0.5 to 1.5 pounds, the average being 1 pound. After 11 months of storage each bag had gained 1.5 to 2 pounds, averaging 1.75 pounds.

Drillability. The drilling rate, referred to a moisture-free basis, was deter-

mined for both fertilizers after each hour of exposure. The percentage of fertilizer drilling after exposure compared to that drilling originally is called the relative drilling rate, and is the basis for rating the fertilizers as to drillability. Table III shows that lot 51-1 drilled at a rate close to 100% after each hour of exposure up to 5 hours. Lot 51-2 drilled at a rate averaging about 111% of original after each hour up to 4 hours of exposure. After the fifth hour, the rate decreased to slightly below 100%. Both materials still seemed dry after 5 hours' exposure when the moisture content was about 14%, wet basis, although some individual particles did appear wet.

Twenty-seven pounds of initial material from each of lots 51-1 and 51-2 were exposed on trays overnight for 17.5 hours. The conditions of exposure differed from those in the 5-hour exposure tests in that the trays were 1 inch apart instead of 5 inches, the relative humidity varied from 94 to 96%, and the temperature was 75° F. No fan was blowing directly on the trays as in the first test, and the trays of fertilizer were not stirred each half hour to aid in uniform moisture absorption throughout the mass.

After the 17.5-hour period of exposure, the fertilizers from lots 51-1 and 51-2 were found to have absorbed moisture rather unevenly. Approximately half the surface area of fertilizer on each tray was wet and soft, while another area adjacent to it appeared "dry" and hard, though caked to a slight extent. The fertilizer on the trays varied in the extent of area wetted, because of uneven exposure, but both lots were similar in this respect. When the material was removed from the trays and mixed, it appeared "damp," and caked when squeezed in the hand.

However, the fertilizer from both lots had remained free-flowing. Table III shows that after 17.5-hour exposure, lot 51-1 drilled at 74.5%, and lot 51-2 at 76.5% of the original drilling rate.

The rate of moisture absorption by both fertilizers was nearly equal. During the 17.5-hour exposure, the moisture content of lot 51-1 increased from 2.11 to 17.37%, a gain of 15.26%. The moisture content of lot 51-2 increased from 3.49 to 17.70%, a gain of 14.21%. During this time the relative drilling rates of both lots decreased to about 75%. At the same time the angles of repose of both lots of material increased appreciably. As the angle increases, the drilling rate usually decreases.

The uniformity of drilling rate of both materials was good as indicated by low coefficients of variation. These high-aluminum nitric phosphate fertilizers have drilling characteristics superior to those of any land-pebble nitric phosphates previously tested. The best land-pebble nitric phosphate fertilizer tested, an 11-11-11 grade, drilled at 65% of the original rate after 2 hours of exposure and failed to drill after 3 hours when the moisture was 9.92%. An 8-12-12 superphosphate type of commercial mixed fertilizer drilled at 28% of the original rate after 3 hours of exposure, and failed to drill after 4 hours when the percentage of moisture was 9.33. Ammonium nitrate failed to drill after an hour's exposure, and it was estimated that it probably would not have drilled after only 15-minute exposure.

Conclusion

The storage properties of the high-aluminum nitric phosphate fertilizer

Table III. Effect of Exposure on Moisture Absorption, Relative Drilling Rate, Coefficient of Variation, and Angle of Repose

| Hours of Exposure | Moisture ^a , % | Relative Drilling Rate, % | Coefficient of Variation | Angle of Repose, ° |
|-------------------|---------------------------|---------------------------|--------------------------|--------------------|
| Lot 51-1 | | | | |
| 0 | 2.00 | 100 | 9.7 | 39 |
| 1 | 5.27 | 98.9 | 8.3 | 39 |
| 2 | 7.87 | 98.2 | 10.7 | 39 |
| 3 | 10.26 | 98.7 | 11.2 | 39 |
| 4 | 12.38 | 102.1 | 10.8 | 38 |
| 5 | 13.92 | 100.6 | 8.4 | 38 ^{1/2} |
| 0 ^b | 2.11 | 100 | 9.7 | 39 |
| 17.5 | 17.37 ^c | 74.5 | 5.6 | 42 ^{1/2} |
| Lot 51-2 | | | | |
| 0 | 3.13 | 100 | 6.2 | 37 ^{1/2} |
| 1 | 5.74 | 112.4 | 6.1 | 37 ^{1/2} |
| 2 | 8.41 | 108.9 | 5.4 | 38 |
| 3 | 10.56 | 114.6 | 6.0 | 39 |
| 4 | 12.60 | 108.8 | 6.2 | 40 |
| 5 | 14.63 | 98.5 | 5.7 | 42 |
| 0 ^b | 3.49 | 100 | 6.2 | 37 ^{1/2} |
| 17.5 | 17.70 ^c | 76.5 | 7.9 | 43 |

^a Moisture determined by air-flow method except as noted.

^b Repeated test with longer exposure period.

^c Moisture determined by vacuum-desiccation method.

are very good, and their drilling characteristics are considered excellent.

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SOIL CONDITIONERS

Dextran and Dextran Products as Soil-Conditioning Materials

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The recent prominence given to dextran and its use as a plasma extender for humans, as well as the development of techniques for making dextran products, has suggested other commercial possibilities. The presence of dextrans in many soils and their stability to influence the binding of soil particles have been noted. Additional work is reported dealing with the effect of molecular weight ranges of dextran polymers on soil conditioning, including data on the influence of the structural branching of dextran polymers. Marked stabilization of soils, up to 60% increased plant seedling emergence, and up to 70% better crop yield, have been demonstrated with soils containing specific dextran products.

SINCE ANCIENT TIMES the plow has symbolized man's agricultural efforts in tilling soils and improving their structure. Within recent times, however, it has become evident that natural agents in many soils help the farmer by imparting increased stability or permanence to soil aggregates. If this were not true, such soil aggregates would be broken down to an undesirable degree mechanically, by tillage instruments, by the action of raindrops, or by excess moisture during rainy seasons. Without such natural stabilizing agents it would be necessary to expend much more than the currently estimated one-third available draft power on the farm (9) for plowing.

Organic and inorganic soil colloids are believed to be largely responsible for the natural structural stability of many good soils. Unfortunately, much of the organic matter has been lost from many of our tillable soils. This continued depletion has focused attention on the development of synthetic materials, which stabilize and condition soil aggregates. Such synthetic materials, however, have not been considered complete substitutes for natural organic materials such as stable manure, nor are they economically feasible from the farmer's viewpoint.

Long before man recognized the need for tillage and the addition of plant nutrients to the soil and before the term "soil conditioner" was coined, Nature supported and sustained plant and vege-

table life, by establishing a relationship between plant nutrients taken from the soil and those returned to it. Soil nutrients taken in water-soluble forms by plants are returned to the soil as organic material—dead plant and animal matter. This organic material is attacked by the multitudinous soil microorganisms and converted to forms again available to plants, and to a product of a mucilaginous nature called humus. It is this material, composed in part of bacterial polysaccharides (3-5, 8, 9, 14), which is mainly responsible for the desirable, well drained, granular condition of typical virgin soils. Dextrans, synthesized by a number of soil bacteria (8), comprise one of these bacterial polysaccharid groups.

Studies on soil-water extracts, particularly rich in organic matter, demonstrate that water-soluble, alcohol-precipitable polysaccharid substances may be present. Certain investigators have reported the influence of bacterial polysaccharides on soils (3-6), but the use of bacterial dextran polysaccharides as soil conditioners has not been sufficiently investigated.

Some properties of dextran were clarified in 1869 by Scheibler (7) in Germany. Dextran is now known to be a polysaccharide-type polymer made up of many molecules of glucose in long chains, having definite repeating linkages and branchings, and chemically related to glycogen.

Dextrans are produced from bacteria

in typical fermentation media, which may contain such materials as corn steep liquor, peptone, and inorganic salts, or may be made in cell-free media from an exocellular enzyme termed dextran sucrose. Such enzymes can be concentrated so that, when added to strong sucrose solutions, they will rapidly form enough dextran to prevent perceptible flow in the system when the container is inverted.

Leuconostoc mesenteroides NRRL-B512, produces the native dextran used for the manufacture of blood plasma volume expanders. Dextrans, as they are biosynthesized, may have molecular weights in the millions and yet many can be dissolved in water to form colloidal solutions of high viscosity. Few, if any, related materials possess such high molecular weights with concomitant water solubility.

The solubility of native dextrans is related to their molecular structures. Those having a high ratio of alpha-1,6 to non-1,6 repeating anhydroglucose unit linkages may be more water-soluble than those with a low ratio; although some dextrans are very insoluble in water, they are apparently all swollen when added to water.

Physical and chemical properties such as water solubility, viscosity, molecular weight, and structure vary widely among different types of dextrans. The number of dextran-producing organisms is large and only a few of the products made by these organisms have been in-