

Crop Response to High-Alumina Nitric Phosphate Fertilizers

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Fertilizers produced by the Tennessee Valley Authority from low grade high-alumina leached-zone ores from the Florida phosphate deposits were evaluated in greenhouse and field trials during three cropping seasons. The high-alumina nitric phosphates proved generally as satisfactory a source of phosphorus as concentrated superphosphate when applied to long-season crops in the Southeast, but were less satisfactory in promoting early growth or as a starter fertilizer for corn. The degree of water solubility of the high-alumina nitric phosphates was of minor importance in final yields on long-season crops of the Southeast. The high-alumina fertilizers having a larger portion of the phosphorus (20 to 30%) in the water-soluble form were more effective in increasing yields of early forage crops, such as wheat or oats, and as a starter fertilizer for corn. In an average of one year's data, the high-alumina nitric phosphates were as effective as nitric phosphates or commercial-type mixtures having approximately the same degree of phosphorus water solubility.

USE OF FLORIDA LEACHED-ZONE PHOSPHATE ORES for the production of fertilizers has been investigated by the Tennessee Valley Authority. These materials represent a considerable reserve of phosphate material, in addition to serving as a potential source of uranium. The use of such ores for the production of fertilizers presents three main problems. (1) They are low in phosphorus content and do not lend themselves to beneficiation; (2) they are high in alumina content; and (3) they are high in silica. The ores used in the Tennessee Valley Authority's experiments ranged from 10 to 15% phosphorus pentoxide, 8 to 16% alumina, and 52 to 66% silica. The calcium oxide content varied from 2 to 12%.

The phosphorus in the ores is present primarily in the form of the minerals wavellite and pseudowavellite, although some of the ores contain varying amounts of apatite. Phosphorus in the fertilizers produced from the ore is present largely as aluminum phosphate, with small quantities of ammonium and calcium phosphates. Petrographic examinations indicate that the aluminum phosphate in the product is amorphous or cryptocrystalline.

Patterson (4), Ellett and Hill (2), Truog (5), and Marias (3) showed that freshly precipitated aluminum phosphates were almost as effective as superphosphate. Some natural aluminum phosphates, such as lazullite, wavellite, and saldanha, were much less effective than super-

phosphate in studies conducted by Marias (3). Bartholomew and Jacob (7) reported that ignited and unignited precipitated aluminum phosphates from New Guinea were satisfactory sources of phosphorus in greenhouse trials. Preliminary data from TVA greenhouse experiments indicate that calcining of natural aluminum phosphate ores at 500° C. increases the availability of the phosphorus.

In the preparation of the fertilizer materials the leached-zone ores were calcined at 1100° C. and extracted with a mixture of nitric and sulfuric acids, after which the slurry was filtered. Following filtration, the filtrate was ammoniated; in some cases potassium chloride was added; and the resulting slurry was dried and ground. The degree of water solubility of the phosphorus content was dependent on the composition of the ore, the proportion of sulfuric and nitric acids used in the extraction, and the degree of ammoniation. The chemical composition of the various materials tested is shown in Table I.

Table I. Chemical Composition of Phosphate Fertilizers Used in Field and Greenhouse Experiments

Nominal Grade ^a	P ₂ O ₅			Total N	K ₂ O	Al ₂ O ₃	CaO
	Total	Citrate-soluble	Water-soluble, % of total				
15-15-15 Al NP	15.9	15.8	27.7	15.2	14.4	8.0	1.7
15-15-15 Al NP	15.1	14.8	4.0	15.1	15.0	5.2	0.5
11-22-11 Al NP	22.2	21.9	19.8	11.7	10.7	9.8	4.9
14-16-14 Al NP	16.9	16.6	30.8	14.6	14.2	7.3	2.4
12-15-15 Al NP	15.2	14.9	4.6	11.8	16.6	6.9	8.6
14-14-14 Al NP	13.5	13.4	10.4	13.1	13.6	10.2	2.2
11-14-16 Al NP	15.2	14.9	4.6	11.8	16.6	8.6	0.5
15-14-15 Al NP	15.1	14.8	4.6	15.1	15.2	5.6	5.2
15-14-14 Al NP	14.0	14.0	18.6	15.0	14.9	5.2	4.4
15-29-0 Al NP	29.1	29.0	27.1	15.3	0.0	11.4	5.7
12-12-12 NP III	12.2	11.8	15.6	12.3	12.6	...	17.3
12-12-12 NP IV	12.6	12.5	10.4	12.0	12.4	...	17.8
0-49-0 CSP	50.1	49.7	92.0	20.9
10-10-10 Com.	10.1	10.0	34.0	10.0	11.2	...	14.7

^a Al NP. High-alumina nitric phosphate. CSP. Concentrated superphosphate.

Procedure and Results

Greenhouse Experiments In preliminary tests in the TVA greenhouse at the University of Tennessee, three nitric phosphate fertilizers high in alumina were compared to concentrated superphosphate as a source of phosphorus for rye grass. Yield data are shown in Table II. On the unlimed Clarksville soil (pH 5.4) the yields of rye grass from treatments with the various materials were not different

from those where concentrated superphosphate was applied, nor did they differ significantly among themselves. When the Clarksville soil was limed (pH 6.1), the high-alumina nitric phosphate compounds were not so effective as concentrated superphosphate. The nitric phosphate very low in water-soluble alumina was also less effective than the two having a higher degree of phosphorus water solubility. The experimental materials were all as effective as concentrated superphosphate on the lightly limed Hartsells soil (pH 5.0), with no significant differences among themselves. When the Hartsells soil was heavily limed (pH 6.1), the yield from the pots where the nitric phosphate very low in water-soluble alumina was used was significantly less than either concentrated superphosphate or the other alumina nitric phosphate compounds. High-alumina nitric phosphate compounds having 18 to 27% of the total phosphorus pentoxide in the water-soluble form were as effective as concentrated superphosphate on this heavily limed soil. In general, liming of these two soils resulted

Table II. Response of Rye Grass to High-Alumina Phosphates and Concentrated Superphosphate in Greenhouse Trials

Material	Water-Soluble P ₂ O ₅ , % of Total	Yield, Grams Dry Matter per Pot ^a			
		Clarksville Soil ^b		Hartsells fsl ^c	
		No lime, pH 5.4	Limed, pH 6.1	0.5 ton lime, pH 5.0	3 tons lime, pH 6.1
15-14-15 Al NP	4.6	13.1	7.4	9.2	4.8
15-14-14 Al NP	18.6	14.2	10.7	10.0	8.2
15-29-0 Al NP	27.1	13.8	10.1	9.3	8.4
Av.		13.7	9.4	9.5	7.1
CSP ^d	90.0	14.1	14.5	8.8	8.7
No P	...	4.9	1.8	0.9	0.6
LSD (sources) ^b	...	1.5	1.4	0.6	1.5

^a Average yields from 40 and 80 pounds of P₂O₅ per 2,000,000 pounds of soil.

^b Silt loam.

^c Fine sandy loam.

^d Least significant difference, calculated.

In this study the alumina nitric phosphate materials were least effective on the calcareous (high pH) Ida soil. The high water-soluble material produced only approximately 65% of the yield obtained from concentrated superphosphate on this soil. The effect of water solubility of the alumina nitric phosphate was also most pronounced on

using various crops over a 3-year period. Several experimental high-alumina nitric phosphates were used in the study. For convenience, they have been grouped into two classifications: (1) those containing approximately 20 to 30% of the total phosphorus pentoxide in a water-soluble form, designated high water-soluble; and (2) those containing less than 5% water-soluble phosphorus pentoxide, designated low water-soluble. In all tests the phosphorus-containing fertilizer was supplemented with sufficient ammonium nitrate and potassium chloride to supply ample amounts of nitrogen and potassium. Rates of phosphate varied from 15 to 18 pounds of phosphorus pentoxide in the several tests. As no appreciable interaction between rates of application and sources of phosphorus was apparent, comparisons in all cases were made between equal rates of concentrated superphosphate and the experimental fertilizers.

Data are presented only from those experiments in which a significant yield response to phosphorus was obtained.

Corn High-alumina nitric phosphate fertilizers were applied to corn in six states during the 1953, 1954, and 1955 growing seasons. As shown in Table IV, they were generally slightly less effective than concentrated superphosphate as a source of phosphorus. With a few exceptions, the low water-soluble high-alumina nitric phosphates were as effective as those having a higher percentage of water-soluble phosphorus.

Table III. Effect of Phosphorus Carriers on Yield of Oat Forage in Greenhouse Trials

Material ^a	Water-Soluble P ₂ O ₅ , % of Total	Yield, Grams Dry Matter per Pot ^b		
		Ida ^c si l ^d	Carrington si l ^d	Edina si l ^d
11-14-16 Al NP	4.6	9.7	12.8	16.7
15-15-15 Al NP	27.7	10.7	12.6	17.4
14-14-14 NP	4.8	11.6	12.7	19.1
12-32-0 NP	40.3	13.6	13.9	20.3
CSP	89.5	16.3	14.1	21.5
No P	...	5.4	6.2	10.1
LSD (sources)	...	1.3	0.7	0.9

^a Al NP. High-alumina nitric phosphate. NP. Nitric phosphate. CSP. Concentrated superphosphate.

^b Yield from 40 pounds of P₂O₅ per 2,000,000 pounds of soil.

^c Average of 2 years' results.

^d Silt loam.

in a lowered effectiveness of the alumina nitric phosphate compounds as a source of phosphorus, with the most pronounced effect on the one having lower water solubility. All of the experimental fertilizers were equally effective on unlimed or lightly limed soils used in this study.

Further greenhouse trials were conducted by Iowa State College, using a number of experimental fertilizers on oats (Table III). Three soil preparations were used: Ida silt loam, pH 8.0; Carrington silt loam, pH 5.5; and Edina silt loam, pH 5.6. All the soils were mixed half and half with sand. The use of alumina nitric phosphate materials resulted in every case in significantly lower yields of oat forage than was obtained from concentrated superphosphate. Although the yields from nitric phosphate low in water-soluble alumina were lower than those obtained from the high water-soluble compound, this difference was not statistically significant.

this soil series. Yield obtained from the low water-soluble material was 9% less than that from the high water-soluble material.

Field Results

The high-alumina nitric phosphates were compared with concentrated superphosphates in field trials in several states,

Table IV. Response of Corn to High-Alumina Nitric Phosphates

State	Av. Yield CSP, Bu./Acre	Hi H ₂ O Sol Al NP ^a		Lo H ₂ O Sol Al NP ^b	
		No. of comparisons	Relative yield, % CSP	No. of comparisons	Relative yield, % CSP
Georgia	69.1	3	92	5	95
Kentucky	38.6	4	88	4	92
New York	86.1	6	94	0	..
Tennessee	27.0	5	99	5	94
Virginia	55.6	2	96	2	88
Mississippi	62.8	3	100	3	97
All states	60.8	23	95	19	94

^a Al NP containing 20.0 to 30.8% of total phosphorus in water-soluble form.

^b Al NP containing 4.0 to 4.6% of total phosphorus in water-soluble form.

Table V. Yield of Corn from High-Alumina Nitric Phosphates and Concentrated Superphosphate Used as Starter Fertilizers in Iowa

Phosphorus Source	Soil Series			
	Floyd ^a	Primghar	Edina ^a	Webster
	Yield, Bushels per Acre			
CSP ^b	65.3	83.0	67.6	53.1
Al NP Lo H ₂ O sol. ^c	57.6	79.4	59.2	46.0
Al NP Hi H ₂ O sol. ^d	62.1	81.5	64.4	47.6
Av.	61.7	80.4	63.5	46.7
LSD (sources)	4.9	3.4	4.2	6.6

^a Average of 1953 and 1955.

^b Concentrated superphosphate.

^c High-alumina nitric phosphate containing 27.7% of total phosphorus in water-soluble form.

^d High-alumina nitric phosphate containing 4.6% of total phosphorus in water-soluble form.

Most of these studies were conducted in the Southeast, but in six comparisons in New York where the high water-soluble materials were used, the yield from the experimental fertilizer was 94% of that obtained from concentrated superphosphate. It was assumed that in many of the trials summarized in Table IV, early season growth was better on the plots which received concentrated superphosphates. Early growth also was better with high than with low water-soluble high-alumina nitric phosphates. However, this effect disappeared as the season advanced and in many cases was not reflected in final yields. Similar results were obtained from 12 tests with cotton in Alabama and Mississippi. This is in general agreement with other results obtained on long-season crops in the Southeast, where degree of water solubility of phosphorus has shown little effect on final yields.

The high-alumina nitric phosphates were used as a starter fertilizer for corn in Iowa. The proximity of the fertilizer to the hill and, possibly, different climatic conditions, resulted in a somewhat different response to the experimental fertilizers than was observed in the other states. The response of high water-soluble high-alumina nitric phosphate was similar to that found in the Southeast; however, the low water-soluble material was less effective on three of the four soils listed. Relative yields, com-

pared to concentrated superphosphate, of the low water-soluble high-alumina phosphates were 87% on Floyd silt loam, 88% on Edina silt loam, and 87% on Webster silt loam. The data from the Floyd and Edina soils represent averages of 2 years' data; the yields from the other soils were collected in 1953. The results of this series of experiments are shown in Table V. The relatively low yields from low water-soluble phosphorus compounds under Iowa conditions are not limited to the high-alumina nitric phosphates. Webb (6) has recently shown that corn yield response from starter fertilizers in Iowa is closely related to water solubility of the phosphorus, regardless of the source.

The results of 2 years' data in Kentucky, where high-alumina nitric phosphates were compared to concentrated superphosphate applied in the row as a starter fertilizer, are somewhat different. In these experiments the yield from the high-alumina nitric phosphates was 92% of that obtained from concentrated superphosphate, but there was no difference in the relative yield from plots where high water-soluble alumina nitric phosphate was used as compared to those receiving low water-soluble alumina nitric phosphates.

Small Grain The high-alumina nitric phosphates were generally somewhat less effective as a source of phosphorus for small grain than for corn

or cotton. The results from seven states are shown in Table VI. Although it was impossible to establish significant differences between sources at any given location, combined comparisons of all tests using the high-alumina nitric phosphates with equal amounts of concentrated superphosphate resulted in significantly lower yields from the use of high-alumina nitric phosphates. There were no apparent differences in the yield of grain between high and low water-soluble alumina nitric phosphates, but in many of the locations only one type of high-alumina nitric phosphate was included in the study. In some cases it was obvious that early growth of forage was delayed in plots receiving high-alumina nitric phosphates. In most instances this effect lessened or disappeared before the grain reached maturity.

Wheat Forage The yield differential between concentrated superphosphate and high-alumina nitric phosphate was more pronounced on early growth of wheat forage than for any other crop. Eleven such comparisons were made at a number of locations in Mississippi. The high-alumina nitric phosphate was 4.0% phosphorus water-soluble. The fertilizer was applied prior to seeding in the fall. Where concentrated superphosphate was applied, the average yield for all locations was 2587 pounds of dry matter per acre as compared with 2171 pounds where alumina nitric phosphate was applied. This represents a relative yield of 84% from the high-alumina nitric phosphate material. An identical relative response was obtained from one test on winter wheat in Kentucky. In neither case were the high and low water-soluble alumina nitric phosphates compared in the same study. Six comparisons made in Alabama, using both high and low water-soluble alumina nitric phosphates, showed the high water-soluble material was 94% as effective as concentrated superphosphate, while the low water-soluble material was 90% as effective.

Effect of Soil pH

Soils from all tests conducted in 1953 and 1954 were grouped into six arbitrary classifications according to soil pH. The yield in each classification was compared with the yield obtained from concentrated superphosphate. The average relative yields are shown in Table VII. There were not equal numbers of tests in each classification and yields on soils of a given pH may represent only one or two locations.

The data in Table VII do not indicate any consistent effect of soil pH on phosphorus availability from the various

Table VI. Response of Wheat to High-Alumina Nitric Phosphates

State	Av. Yield CSP, Bu./Acre	Hi H ₂ O Sol Al NP ^a		Lo H ₂ O Sol Al NP ^b	
		No. of comparisons	Relative yield, % CSP	No. of comparisons	Relative yield, % CSP
Georgia	25.2	2	88	6	90
Kentucky	24.3	3	95	3	95
New York	55.5	3	97	0	..
Tennessee	30.3	2	89	0	..
Virginia	31.3	4	95	4	90
Washington	28.1	4	91	2	94
Alabama	15.6	4	97	2	93
All states	22.1	22	92	17	92

^a Al NP containing 20.0 to 30.8% of total phosphorus in water-soluble form.

^b Al NP containing 4.0 to 4.6% of total phosphorus in water-soluble form.

fertilizers. There is an indication that the high-alumina nitric phosphates were somewhat less effective at high than at low pH, particularly where the low water-soluble material was applied. There were insufficient trials on limed and unlimed soils at the same location to make valid comparisons.

Evaluation of Phosphorus Sources

The yields of plots to which high-alumina nitric phosphates were applied have been compared to those obtained from concentrated superphosphate because this material was used as the standard phosphate source and thus appeared in all tests in comparable amounts. More valid evaluation of the relative merits of the experimental fertilizers may be gained by comparing them to other materials more readily comparable in their ratios of plant food elements and degree of phosphorus water solubility.

In 1955 a series of experiments was conducted, in which two high-alumina nitric phosphates of 1-1-1 ratio were compared to nitric phosphates from Florida pebble ore and to a commercial-type 1-1-1 mixture having approximately 30% of the total phosphorus in the water-soluble form. Concentrated superphosphate was included in all trials as a standard phosphorus source. The fertilizers were applied on cotton, corn, wheat, and wheat forage. The nitric phosphate materials differed from the high-alumina nitric phosphates in that they were made from Florida pebble ore and did not contain appreciable quantities of aluminum phosphates. The nitric phosphate process III was made by treating rock phosphate with nitric acid, ammoniating the resulting slurry, and adding potassium sulfate. Nitric phosphate process IV was manufactured by extracting rock phosphate with nitric acid, followed by ammoniation with the addition of carbon dioxide. Potassium chloride was added to supply potassium.

Relative yields of the experimental fertilizers compared to concentrated superphosphate are shown in Table VIII. All comparisons were made in the Southeast, except where the materials were applied as starter fertilizers on corn in Iowa. The nitric phosphates produced from Florida pebble ore were about equal to the high water-soluble alumina nitric phosphates in their effectiveness and were generally somewhat more effective than the low water-soluble alumina nitric phosphates. The 1-1-1 grade material using high-alumina phosphates induced about the same response as commercial-type fertilizers having approximately the same degree of phosphorus water solubility. In some cases the commercial-type mixture was more effective than the low water-soluble alumina nitric phosphate, especially on wheat forage and wheat.

Table VII. Effect of Soil pH on Response of Crops to High-Alumina Nitric Phosphates

Soil pH	Al NP Hi H ₂ O Sol ^a		Al NP Lo H ₂ O Sol ^b		All Al NP
	No. of comparisons	Relative ^c yield	No. of comparisons	Relative ^c yield	Av. relative ^c yield
4.5-5.0	6	102	6	97	100
5.1-5.5	2	101	4	88	92
5.6-6.0	16	91	18	95	93
6.1-6.5	5	94	5	91	92
6.5-7.0	5	91	0	..	91
7.0	7	97	7	81	89

^a High-alumina nitric phosphates containing 20.0 to 30.8% of the total phosphorus in water-soluble form.

^b High-alumina nitric phosphates containing 4.0 to 4.6% of the total phosphorus in water-soluble form.

^c Relative yield CSP = 100.

Summary

High-alumina nitric phosphate fertilizers prepared from leached-zone ores were satisfactory sources of phosphorus for cotton, small grain, or corn, when the fertilizer was applied in the row prior to planting, or was broadcast and worked into the soil, but were somewhat less effective than concentrated superphosphate.

In some cases these materials were less satisfactory as a source of starter fertilizer for corn than concentrated superphosphate or nitric phosphates. This was especially true of high-alumina nitric phosphates having 5% or less of the total phosphorus pentoxide in the water-soluble form.

The high-alumina nitric phosphates were less effective as a source of phosphorus during the early growth of plants. Yields of wheat grown for forage from plots fertilized with the low water-soluble high-alumina nitric phosphates were 87% of those obtained from concentrated superphosphate. The high water-soluble alumina nitric phosphates were more effective.

One year's comparison of the high-alumina nitric phosphates with nitric phosphates made from Florida pebble ore and with a commercial-type mixture showed little difference in the three types of materials. This was especially obvious when the phosphorus water solu-

bility of the three types of materials was approximately the same. None of the three was as effective as concentrated superphosphate.

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Table VIII. High-Alumina Nitric Phosphates Compared with Other Materials as a Source of Phosphorus

	Av. Yield CSP, Bu. or Lb./Acre	Average Relative Yield, % of CSP				Com. ^c type 10-10-10
		Lo H ₂ O sol ^a Al NP	Hi H ₂ O sol ^b Al NP	Nitric phos. process III	Nitric phos. process IV	
Corn	51.9	91	95	87	87	88
Corn ^d	74.5	88	95	90	90	92
Wheat	20.6	93	99	101	94	97
Cotton	1567	96	98	98	103	98
Wheat forage	2666	87	91	98	92	109

^a 15-15-15 Al NP containing 4.0 to 4.6% of total phosphorus in water-soluble form.

^b 15-15-15 Al NP containing 20.0 to 30.8% of total phosphorus in water-soluble form.

^c 10-10-10 commercial-type fertilizer containing approximately 30% of total phosphorus in water-soluble form.

^d Materials applied as starter fertilizer on corn in Iowa.