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SOURCES OF NITROGEN AND PHOSPHORUS

Crop Response to Nitrogen and Phosphorus in Metal Ammonium Phosphates

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$MgNH_4PO_4 \cdot 6H_2O$, $MgNH_4PO_4 \cdot H_2O$, and $FeNH_4PO_4 \cdot H_2O$ were compared in three granule sizes (−6 +9, −14 +20, and −35 +60 mesh) with $NH_4H_2PO_4$ as sources of nitrogen and phosphorus for two successive crops of corn grown in greenhouse pots. $MnNH_4PO_4$, $ZnNH_4PO_4$, and $CaHPO_4$ were also compared as sources of phosphorus. Response by crop 1 decreased markedly with increase in granule size of the fertilizers applied as nitrogen sources just prior to planting; after incubation in moist soil for 90 days prior to planting, granule size had only minor effects. Crop 2 showed residual effects only to −6 +9 mesh granules. Granulation of a nitrification inhibitor with $MgNH_4PO_4 \cdot H_2O$ significantly decreased recovery of the nitrogen by two crops of corn. Response by both crops to phosphorus in $CaHPO_4$ and the metal ammonium phosphates increased with decrease in granule size. $CaHPO_4$ showed the greatest granule size effects and the magnesium compounds the least. $FeNH_4PO_4 \cdot H_2O$ was the least effective source at all granule sizes.

Magnesium ammonium phosphate hexahydrate, $MgNH_4PO_4 \cdot 6H_2O$, dissolves incongruently in water with precipitation of a mixture of di- and trimagnesium phosphates, $MgHPO_4 \cdot 3H_2O$ and $Mg_3(PO_4)_2 \cdot 8H_2O$ (*5, 6*). In moist soil, a granule of magnesium ammonium phosphate dissolves to give a solution of constant composition corresponding to an invariant point in the system $MgO–NH_3–P_2O_5–H_2O$ at constant temperature and pressure. At 25° C. this solution is $2.6 \times 10^{-3}M$ in Mg, $6.6 \times 10^{-3}M$ in P, and $7.7 \times 10^{-3}M$ in NH_4, and has a pH of 7.12. The osmotic pressure of this solution will be no higher than that of the soil solution, and the rate of dissolution of the $MgNH_4PO_4 \cdot 6H_2O$ will be controlled by diffusion of the ions into the surrounding soil and by nitrification of NH_4 ions from the granule. Since both of these processes will be slowed as the area of contact between the granule and the soil is decreased, it is to be expected that crop response to the fertilizer will decrease with increase in granule size.

In contact with water, magnesium ammonium phosphate monohydrate, $MgNH_4PO_4 \cdot H_2O$, forms the hexahydrate, and the extent of hydration in the soil depends upon the temperature and moisture tension. Bridger, Salutsky, and Starostka (*1*) showed that the monohydrate is the stable form under dry summer conditions in a surface soil. Under the experimental conditions in this work complete conversion to the hexahydrate is to be expected. Little is known of the solubility of other metal ammonium phosphates.

The present experiments were undertaken to measure the response of corn (*Zea mays*) to the nitrogen and phosphorus in magnesium and other metal ammonium phosphates, as affected by granule size. Corn was grown under nonleaching conditions so that the results could be better related to the known chemical behavior of the compounds.

Materials and Methods

The compositions of the fertilizers are shown in Table I. All sources were prepared as fine powders and tableted under pressure; the tablets were crushed and screened to −6 +9, −14 +20, and −35 +60 mesh. The samples of metal ammonium phosphates other than $MgNH_4PO_4 \cdot 6H_2O$ (prepared by TVA) were supplied by W. R. Grace and Co.

The soil was Hartsells fine sandy loam (pH 5.2) limed to pH 6.3 with a mixture of 4 parts of $CaCO_3$ and 1 part of $MgCO_3$. Each pot, a polyethylene-lined No. 10 tin can, contained 3 kg. of soil (dry basis). All treatments were made in triplicate. Funk's G-76 hybrid corn was grown in all experiments.

Nitrogen Source Test (Experiment 1). One series of nitrogen treatments (150, 300, and 600 mg. of nitrogen per pot) was mixed with the soil and incubated at 18% moisture for 90 days before planting to determine nitrogen release from the fertilizers during this period. The second series was prepared just before planting. In each series, phos-

Table I. Partial Composition of Fertilizers

Source	Total N, %	Total P, %	Total Metal,[a] %
$MgNH_4PO_4 \cdot 6H_2O$	5.6	12.8	9.9
$MgNH_4PO_4 \cdot H_2O$	8.9	20.0	15.8
$MnNH_3PO_4$	7.3	16.6	28.7
$FeNH_4PO_4 \cdot H_2O$	7.2	16.4	29.4
$ZnNH_4PO_4$	7.8	17.4	35.7
$CaHPO_4$	..	22.7	29.4
$NH_4H_2PO_4$	12.2	26.9	..

[a] Mg, Mn, Fe, Zn, or Ca.

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phorus was equalized with $CaHPO_4$ to a total of 2 grams of phosphorus per pot.

Corn was planted (seven seeds, thinned to five plants) on September 19, 1962. K_2SO_4 was added in solution to supply 200 mg. of potassium per pot. The crop was harvested on November 2. Corn was planted similarly as a second crop on November 7 and harvested on January 4, 1963. For this crop, each pot received 200 mg. of potassium as K_2SO_4, and a micronutrient solution. Weights of dry forage and nitrogen uptake by both crops of corn forage were determined.

Effect of Nitrification Inhibitor (Experiment 2). $MgNH_4PO_4.H_2O$, (−6 +9 mesh) granulated with 2% of N-Serve (Dow Chemical Co.), and $NH_4H_2PO_4$ were mixed with Hartsells soil in amounts to supply 60, 150, 300, and 600 mg. of nitrogen per pot. Fine $CaHPO_4$ in amounts to equalize all pots to a total of 2 grams of phosphorus was also mixed with the soil. Corn was planted at once on August 23, 1963. K_2SO_4 was added in solution to supply 100 mg. of potassium per pot. This crop was harvested on October 7. A second crop was planted on October 18. Only K_2SO_4 to supply 100 mg. of potassium per pot was added for this crop. The crop was extremely nitrogen-deficient when harvested on December 11. Yields of dry forage and nitrogen uptake were determined.

Phosphorus Source Test (Experiment 3). The sources were mixed with the soil on April 18, 1963, in amounts to supply 80 and 160 mg. of phosphorus per pot. Nitrogen was equalized to 200 mg. per pot with NH_4NO_3 in solution on April 19, after planting corn. K_2SO_4 was added to supply 200 mg. of potassium per pot on April 25, and NH_4NO_3 was added to supply 100 mg. of nitrogen each on May 7, 16, and 28. The crop was harvested on June 4. A second crop of corn was planted on June 14 without further application of phosphorus. NH_4NO_3 was added to supply 200 mg. of nitrogen per pot each on June 14 and July 7, and 100 mg. each on July 17 and 25. K_2SO_4 was added to supply 100 mg. of potassium per pot each on June 21 and July 2. This crop was harvested on July 31. Dry forage yield and phosphorus uptake were determined for both crops.

Table II. Yields of Dry Forage by First Crop of Corn with Metal Ammoniurr Phosphates as Sources of Nitrogen (Experiment 1)

N Source	N Applied for Crop 1, Mg./Pot	Incubated 90 Days, Grams/Pot			Not Incubated, Grams/Pot		
		−6 +9 mesh	−14 +20 mesh	−35 +60 mesh	−6 +9 mesh	−14 +20 mesh	−35 +6(mesh
$MgNH_4PO_4 \cdot 6H_2O$	150	27.0	28.6	...	23.2	27.3	...
	300	37.7	38.4	42.8	31.7	39.3	40.5
	600	52.9	52.8	...	44.1	58.0	...
$MgNH_4PO_4 \cdot H_2O$	150	28.4	28.5	28.6	20.1	25.0	27.5
	300	37.3	38.4	42.1	26.5	34.9	38.6
	600	54.6	51.8	55.1	40.5	50.8	56.7
$FeNH_4PO_4 \cdot H_2O$	150	26.6	26.9	...	20.0	21.8	...
	300	34.0	35.3	36.7	26.5	32.8	35.7
	600	47.6	52.3	...	37.3	43.8	...
$NH_4H_2PO_4$	150	26.6	...	...	26.9	...	...
	300	38.4	...	...	38.8	...	...
	600	55.8	...	...	50.6	...	...
No N	0	15.6	...	...	12.3	...	...
L.S.D., 5% level		3.3	3.3	3.3	3.0	3.0	3.0

Table III. Percentage Recoveries of Applied N (Experiment 1)

N Source	First Crop			Both Crops		
	−6 +9 mesh	−14 +20 mesh	−35 +60 mesh	−6 +9 mesh	−14 +20 mesh	−35 +6(mesh
Incubated 90 Days before Planting						
$MgNH_4PO_4 \cdot 6H_2O$	63	70	..	63	70	..
$MgNH_4PO_4 \cdot H_2O$	66	60	73	66	61	74
$FeNH_4PO_4 \cdot H_2O$	51	57	..	55	58	..
$NH_4H_2PO_4$	74	..	..	76	..	..
Applied Just before Planting						
$MgNH_4PO_4 \cdot 6H_2O$	63	76	..	77	76	..
$MgNH_4PO_4 \cdot H_2O$	44	73	81	53	73	81
$FeNH_4PO_4 \cdot H_2O$	41	53	..	57	61	..
$NH_4H_2PO_4$	71	..	..	71	..	..

Results

Nitrogen-Source Test (Experiment 1). There was a marked response by the first crop of corn to applied nitrogen, as shown in Table II. With the magnesium fertilizers incubated in soil 90 days prior to planting, response was rather similar for the three granule sizes. However, with these fertilizers applied just prior to planting, response increased markedly with decrease in granule size. Response to −6 +9 mesh $FeNH_4PO_4 \cdot H_2O$ was poorer than to the same size of the magnesium fertilizers.

There was no response by crop 2 (data not shown) to any of the fertilizers incubated for 90 days prior to planting. However, there was a significant residual response to −6 +9 mesh magnesium and iron compounds applied at the rate of 600 mg. of nitrogen just prior to planting.

Nitrogen uptake by both crops was related linearly to amount of nitrogen applied, within experimental error, so that the effectiveness of the fertilizers can be compared in terms of the slopes of the uptake curves calculated by the least squares method. These data expressed as percentage recoveries of applied nitrogen (Table III) show that the −35 mesh $MgNH_4PO_4 \cdot H_2O$ applied just before planting was superior to $NH_4H_2PO_4$ as a source of nitrogen, but that the −6 +9 mesh granules were less effective. When the fertilizers were incubated in the soil for 90 days before planting, the recovery of nitrogen from the −6 +9 and − 14+ 20 mesh magnesium ammonium phosphates was somewhat less than that from $NH_4H_2PO_4$.

Data in Table III show that only the coarse granules of metal ammonium phosphates supplied nitrogen to crop 2. On the basis of total dry matter and nitrogen recovered in both crops, response of corn to the coarse granules of the metal ammonium phosphates was less than to $NH_4H_2PO_4$ over the 15-week cropping period.

Although some nitrogen was obtainec from the $FeNH_4PO_4 \cdot H_2O$ by both crops this compound was a rather inefficien source of nitrogen for corn. $ZnNH_4PO$ was also included as a nitrogen source ir the experiment but was toxic to corr at high rates of the −35 +60 and −1 +20 mesh material, particularly for crop 2. In order to supply 150, 300, and 60(mg. of nitrogen, very high amounts o 686, 1373, and 2746 mg. of zinc were applied per pot.

Effect of Nitrification Inhibitor (Experiment 2). As shown in Table IV granulation of N-Serve with $MgNH_4PO_4 \cdot H_2O$ markedly reduced the release of nitrogen to two crops of corn. Percentage recoveries of applied nitrogen corresponding to the calculated slopes of the uptake curves were in the order $NH_4H_2PO_4 > MgNH_4PO_4 \cdot H_2O > MgNH_4PO_4 \cdot H_2O$ + N-Serve. Total yields o dry forage by both crops, however, were higher for $MgNH_4PO_4 \cdot H_2O$ than fo $NH_4H_2PO_4$. This apparently reflects the greater residual effects of the magnesium compound. Petrographic examination after crop 2 of the $MgNH_4PO_4 \cdot H_2O$ granules showed the presence of both $MgNH_4PO_4 \cdot 6H_2O$ and $MgHPO_4 \cdot 3H_2O$.

Phosphorus Source Test (Experiment 3). Yields of dry forage in Table

Table IV. Response of Corn to $MgNH_4PO_4 \cdot H_2O$ as Affected by Nitrification Inhibitor (Experiment 2)

N Source	N Applied for Crop 1, Mg./Pot	Yield of Dry Matter, Grams/Pot: Crop 1	Yield of Dry Matter, Grams/Pot: Both crops	Uptake of N, Mg./Pot: Crop 1	Uptake of N, Mg./Pot: Both crops	% Recovery of Applied N: Crop 1	% Recovery of Applied N: Both crops
No N	0	17.5	21.4	115	140	...	...
$MgNH_4PO_4 \cdot H_2O$	60	21.4	27.1	155	188	42	59
	150	26.4	33.7	194	237		
	300	32.8	44.0	256	318		
	600	38.6	57.2	376	501		
$MgNH_4PO_4 \cdot H_2O$ + N-Serve	60	20.4	25.1	122	149	33	47
	150	24.1	31.8	162	211		
	300	27.8	38.7	230	300		
	600	33.6	47.7	303	409		
$NH_4H_2PO_4$	60	24.7	27.9	166	186	87	89
	150	31.4	36.3	242	273		
	300	38.1	43.8	360	391		
	600	41.0	51.6	613	673		
L.S.D., 5% level		2.6	...	20	...	...	...

Table V. Yields of Dry Forage (Grams/Pot) by Two Successive Crops of Corn (Experiment 3)

P Source	P Applied, Mg. per Pot	−6 +9 Mesh: Crop 1	−6 +9 Mesh: Crop 2	−6 +9 Mesh: Both crops	−14 +20 Mesh: Crop 1	−14 +20 Mesh: Crop 2	−14 +20 Mesh: Both crops	−35 +60 Mesh: Crop 1	−35 +60 Mesh: Crop 2	−35 +60 Mesh: Both crops
$MgNH_4PO_4 \cdot 6H_2O$	80	16.9	27.6	44.5	24.9	29.6	54.5	24.9	28.1	53.0
	160	27.4	30.2	57.6	38.7	32.1	70.8	38.0	31.8	69.8
$MgNH_4PO_4 \cdot H_2O$	80	11.1	22.2	33.3	19.2	31.6	50.8	23.3	25.2	48.5
	160	14.7	30.6	45.3	27.5	35.2	62.7	34.6	31.5	66.1
$MnNH_4PO_4$	80	8.1	15.4	23.5	14.3	27.0	41.3	25.4	27.1	52.5
	160	9.4	18.5	27.9	22.8	34.6	57.4	34.9	30.7	65.6
$FeNH_4PO_4 \cdot H_2O$	80	7.2	13.4	20.6	8.4	15.5	23.9	12.9	17.4	30.3
	160	8.5	17.0	25.5	10.3	20.4	30.7	19.0	23.2	42.2
$ZnNH_4PO_4$	80	12.1	14.8	26.9	10.9	16.8	27.7	18.9	25.9	44.8
	160	15.7	19.1	34.8	15.6	21.2	36.8	28.3	29.2	57.5
$CaHPO_4$	80	6.6	15.1	21.7	9.9	25.6	35.5	19.4	31.6	51.0
	160	8.4	19.2	27.6	13.0	32.1	45.1	28.0	35.8	63.8
$NH_4H_2PO_4$	80	28.7	25.6	54.3	26.4	26.8	53.2	25.7	27.3	53.0
	160	41.3	29.9	71.2	38.4	29.9	68.3	36.0	34.6	70.6
No P	0	...	...	...	...	...	...	5.9	11.2	17.1
L.S.D., 5% level		2.5	2.7	...	2.5	2.7	...	2.5	2.7	...

Table VI. Per Cent Recovery of P by Two Successive Crops of Corn (Experiment 3)

P Source	Crop No.	−6 +9 Mesh	−14 +20 Mesh	−35 +60 Mesh
$MgNH_4PO_4 \cdot 6H_2O$	1	10.1	18.0	15.4
	2	14.3	15.8	17.6
	Both	24.4	33.8	33.0
$MgNH_4PO_4 \cdot H_2O$	1	4.8	13.0	14.5
	2	14.3	18.7	17.3
	Both	19.1	31.7	31.8
$MnNH_4PO_4$	1	2.1	9.2	13.8
	2	5.7	15.1	16.0
	Both	7.8	24.3	29.8
$FeNH_4PO_4 \cdot H_2O$	1	1.7	3.2	7.8
	2	3.4	5.7	7.6
	Both	5.1	8.9	15.4
$ZnNH_4PO_4$	1	4.8	5.6	11.2
	2	4.5	5.6	13.6
	Both	9.3	11.2	24.8
$CaHPO_4$	1	1.8	5.3	13.1
	2	4.6	15.8	20.5
	Both	6.4	21.1	33.6
$NH_4H_2PO_4$	1	18.2	14.7	16.6
	2	14.9	15.6	16.0
	Both	33.1	30.3	32.6

V show marked response to applied phosphorus, which increased with decrease in granule size of the magnesium, manganese, iron, and zinc ammonium phosphates, and of $CaHPO_4$. Response with $NH_4H_2PO_4$ decreased slightly with decrease in granule size.

Uptake of phosphorus by the crops was essentially linear with amount applied. Consequently, slopes of the linear responses were calculated by the least squares method and are shown in Table VI as percentage recoveries of applied phosphorus.

The data for $CaHPO_4$ and $NH_4H_2PO_4$ are characteristic of these compounds. The recovery of phosphorus from $CaHPO_4$ decreased markedly with increase in granule size, and was higher in the second crop than in the first. The recovery of phosphorus from $NH_4H_2PO_4$ was virtually independent of granule size and was about the same in both crops. The behavior of the magnesium compounds was intermediate. In the first crop there was a marked decrease in phosphorus uptake with increase in granule size, although the effect was much less marked than with $CaHPO_4$. More phosphorus was recovered by the second crop than by the first, and the granule-size effect was much less than in the first crop. The phosphorus recovered from the magnesium compounds by the two crops was between 58 and 100% that from $NH_4H_2PO_4$ and was much greater than that from $CaHPO_4$, except in the smallest granule size.

$FeNH_4PO_4 \cdot H_2O$ was inferior to $CaHPO_4$ throughout the experiment. Examination of the residues of $FeNH_4PO_4 \cdot H_2O$ after the second crop showed that the salt had hydrolyzed to amorphous ferric phosphate in protective layers that covered some unchanged material. Results with $MnNH_4PO_4$ and $ZnNH_4PO_4$ were intermediate between $CaHPO_4$ and the magnesium ammonium phosphates.

There was no indication that $ZnNH_4PO_4$ was toxic to corn in the amounts necessary to supply 80 and 160 mg. of phosphorus per pot. These amounts supplied 164 and 328 mg. of zinc per pot. $CoNH_4PO_4$ and $CuNH_4PO_4$, also compared as sources of phosphorus, were highly toxic and the data are not reported.

Discussion

Under the nonleaching conditions of the experiments, the metal ammonium phosphates all showed a decrease in nitrogen availability with increase in granule size, but the effect was temporary and largely disappeared after 90 days' incubation in the soil prior to cropping. Sources that supplied least nitrogen to the first crop yielded some nitrogen to the second crop, so that there were larger residual effects from large granules than from small granules. However, the increase in recovery by the second crop was less than the decrease in the first, and the total dry matter yield and nitrogen uptake were usually less than those from $NH_4H_2PO_4$. Different results would be expected under leaching conditions. Lunt, Kofranek, and Clark (*2*) also found slower release of nitrogen from surface-applied than from mixed application of magnesium ammonium phosphates. Granules larger than −6 +9 mesh might release nitrogen more slowly, but there is no evidence that such retardation would result in increased efficiency of utilization of the nitrogen. Granulation of a nitrification inhibitor (N-Serve) with $MgNH_4PO_4 \cdot H_2O$ significantly reduced the release of nitrogen. This indicates that N-Serve retarded dissolution of the granules, probably by retarding the rate of nitrification of the dissolving ammonium nitrogen.

The contrast between the magnesium phosphates and dicalcium phosphate as sources of phosphorus is of interest. After the harvest of the second crop, the residues of the −6 +9 mesh magnesium compounds were found petrographically to consist entirely of dimagnesium phosphate, $MgHPO_4 \cdot 3H_2O$.

As long as the granules contained $MgNH_4PO_4 \cdot 6H_2O$, they dissolved to give a solution $6.6 \times 10^{-3}M$ in phosphate. Moreno, Brown, and Osborn (*4*) showed that when dicalcium phosphate dissolves incongruently with precipitation of octacalcium phosphate, the solution is $2.2 \times 10^{-3}M$ in phosphate and has a pH of 6.4, which is close to that of the soil used in the phosphorus-source test. The smaller granule-size effect shown by the magnesium compounds in the first crop, when some of the original material remained in the granules, may reflect this difference in phosphate concentration.

The results of chemical and petrographic studies of the magnesium ammonium phosphates indicate that the granules would consist largely of $MgHPO_4 \cdot 3H_2O$ after the first crop. The pH of the solution within the granules would then fall to that of the surrounding soil, and the phosphate concentration in the solution released would be controlled by the solubility of the dimagnesium phosphate. At pH 6.3 and below, $Mg_3(PO_4)_2 \cdot 8H_2O$ would not persist. The uptake of phosphorus by the second crop from the magnesium and calcium compounds would therefore reflect the differences in the solubility of dicalcium and dimagnesium phosphates, the solubility products of which at 25° C. are 2.8×10^{-7} and 1.5×10^{-6}, respectively (*3*, *6*). Since the activity of magnesium in the soil solution is lower than that of calcium, the difference in phosphate ion concentration may favor dissolution of the magnesium compound even more than these values indicate.

The decrease from the first to the second crop of the granule-size effect shown by the magnesium compounds, in contrast to the behavior of dicalcium phosphate, probably reflects this change in phosphate solubility as ammonia was lost from the granules. A lower rate of hydrolysis of $MgNH_4PO_4 \cdot H_2O$ than that of the hexahydrate may also account for the greater granule-size effect of the monohydrate in the first crop. Little difference between the two hydrates was evident in the second crop.

The results provide a striking example of solublity and granule-size effects of phosphorus sources. With a soluble source, such as $NH_4H_2PO_4$, there is considerable movement of the phosphorus, and the amount of soil saturated with phosphorus does not vary greatly with different granule sizes of the source, the chemical character of the form in which the phosphorus is precipitated being the most important factor. With the less soluble materials, in which the phosphorus is virtually confined to the volume of the original granule, the phosphate concentration established within the granule becomes the dominant factor.

The changes in granule-size effects between the first and second crops may, however, be due only partly to changes in solubility with alterations within the granule. Translocation of phosphorus from the granules by the plant roots causes some redistribution of the phosphorus, and the second crop may recover some phosphorus from the decaying roots of the first. The amount of phosphorus taken up from decaying roots, however, probably is small in comparison with that taken up from the granule sites, where the concentration will be much higher than in the rest of the soil.

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PHOSPHORUS COMPONENTS

Crop Response to Water-Soluble and Water-Insoluble Phosphorus Components of Granular Fertilizers

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THE WATER-INSOLUBLE PHOSPHORUS COMPONENT makes up a considerable portion of the AOAC-available P_2O_5 content of many fertilizers, and it is important that its effectiveness for crop growth be determined, as well as that of the water-soluble fraction.

During the ammoniation of ordinary superphosphate, water solubility of the phosphorus decreases with increasing degree of ammoniation. At the same time, increasing amounts of apatite-type compounds of low solubility are being formed. Thus, the quality of the AOAC (Association of Official Agricultural Chemists) water-insoluble fraction of the phosphorus is changed along with the amount of the water-soluble fraction. Similar changes may occur during ammoniation of concentrated superphosphate and nitric phosphate. The