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The Influence of Cation Exchange Capacity and Depth of Incorporation on Ammonia Volatilization from Ammonium Compounds Applied to Calcareous Soils¹

L. B. FENN AND D. E. KISSEL²

ABSTRACT

The objective of this study was to determine the influence of soil cation exchange capacity (CEC) and depth of incorporation on NH3-N volatilization from NH4+-N compounds applied to calcareous soil. This study was conducted in the laboratory on soils with a wide range of CEC. An increasing CEC resulted in decreasing NH₃ losses. Ammomium sulfate produced higher soil pH values and NH, losses than did NH4NO3. The pH of the soil decreased with increasing NH4NO3 application rates. With NH4NO3, percent NH3-N losses decreased with increasing application rate; however, with (NH4)2SO4, percent NH3-N losses increased as the application rates increased.

Incorporation of the NH4+-compounds into the soil reduced NH3 losses. Increasing depths of NH4+-incorporation resulted in reduced NH3 loss. Losses decreased as the CEC of soil increased. The effectiveness of soil depth in reducing NH₃ loss was associated with soil water content. Decreasing the soil water increased the effectiveness of soil incorporation for reducing NH₃ losses.

Two regression equations were developed to describe NH₃ losses with respect to CEC, soil pH, time, NH4+-N application rate and temperature. Correlation coefficients were 0.86 and 0.81 for (NH4)2SO4 and NH₄NO₃ systems, respectively.

Additional Index Words: (NH4)2CO3 formation, N fertilization, soil CaCO₃, NH₃-N loss model.

OST RESEARCH concerning the effect of cation ex-Mchange capacity (CEC) on NH₃ retention has been associated with the use of anhydrous ammonia (7, 8, 10, 11, 13, 14, 15). Working with anhydrous ammonia, Stanley and Smith (13) found that NH3-N losses to a depth of 23 cm

were greatest in sandy soil, intermediate in silt loam and least in clay soil. McDowell and Smith (11) reported similar results, and concluded that NH₃-N loss was inversely related to CEC. Wahhab et. al. (15) applying (NH₄)₂SO₄ to a sand and a sandy loam, found that the sand lost greater percentages of NH₃.

Soil water content has also been noted to affect ammonia retention. Stanley and Smith (13) noted that with air-dry soil, near maximum NH3-N losses occurred following anhydrous ammonia applications. They also found that NH₃-N losses decreased as the soil water content increased to an intermediate level, with a rapid increase in NH₃-N loss occurring as field capacity was reached. In several cases greater losses of applied NH₃-N were recorded for soil at field capacity than for air dry soils. They stated that evaporating water probably helped transport this NH₃ to the surface and hence increased NH₃-N loss. McDowell and Smith (11) and Jackson and Chang (8), however, noted the opposite effect. They found that greater soil water content reduced NH₃-N losses. Jenny et al. (9), and Brown and Bartholomew (1), noted that dilute NH₃ solutions were competitive with water. Increasing the water contents tended to replace adsorbed NH₃. They stated that even in

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Table 1—Selected physical and chemical properties of the experimental soils.

Soil mixture	Soil CaCO ₃ content	Added CaCO ₃	Soil	Field water capacity	Cation exchange capacity	Working water content
	%	%	pН	%	meq/100 g	%
Houston Black clay						
(HBc)	29	0	7.6	55	58	30
50% HBc + 50% Sand		5		87	29	23
25% HBc + 75% Sand		10		26	18	19
15% HBc + 85% Sand		10		23	9	17
10% HBc + 90% Sand		10		20	6	14
100% Sand		10		17	ŏ	10
Harkey silty clay loam		-•		- '	•	
(Ha sid)	6	10	7.6		22	22

clay suspensions, NH_3 was not strongly adsorbed. Many of the apparently different conclusions might be explained by the physical design used in the research.

The objective of this research was to examine the effect of cation exchange capacity on NH_3 -N volatilization from NH_4^+ -N compounds applied to the surface of calcareous soils and to determine the effect of shallow soil incorporation in reducing these losses.

MATERIALS AND METHODS

The experimental apparatus used has been described in an earlier publication (3). All research was conducted in the laboratory with Houston Black clay (HBc), Harkey silty clay loam (Ha sicl), and sand + CaCO₃. Various values of CEC were produced by mixing HBc and sand. Calcium carbonate was added, at the rate of 5% by weight of the 50-50% HBc-sand mixture and 10% by weight to all other HBc-sand mixtures, sand and to Ha sicl. The mixtures with their characteristic CEC, CaCO₃ contents, gravimetric water content at field capacity and experimental soil water contents are given in Table 1. The 75% sand + 25% HBc. 85% sand-15% HBc, 90% sand-10% HBc, and 100% sand were wetted and then dried at 105°C for 24 hours. The soil mixtures were then pulverized and rewetted. The HBc-sand mixtures were placed 5 to 10 cm deep on saturated sand (23% water by weight). Unsieved Ha sicl tended to form large spheres and puddle in wetting, therefore, it could not be used. The Ha sicl therefore, was sieved and only 4.8- to 19.0-mm sized aggregates were used due to better physical condition in the wetting process.

The NH₃ volatilization studies were conducted at 12, 22, 30,

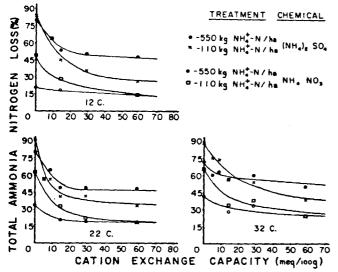


Fig. 1—Influence of cation exchange capacity, temperature, NH₄⁺-salt and NH₄⁺-N application rate on total NH₃-N losses from HBc and sand mixtures.

and 32°C for 100 hours with surface-applied NH₄⁺-compounds. When the NH₄⁺-compounds were incorporated into the soil, NH₃ collection times approached 300 to 400 hours. Ammonia collection with soil-incorporated NH₄⁺-compounds was terminated when <1% of the applied NH₄⁺-N was collected as NH₃N in 24 hours. Ammonium-nitrogen as $(NH_4)_2SO_4$ or NH₄NO₃ was applied to the soil at rates of 110 and 550 kg/ha. The NH₄⁺-N was applied as a dry salt in the following manner; (i) to the soil surface at 110 and 550 kg/ha, (ii) placed 2.5, 5.0 or 7.5 cm deep at rates of 550 kg/ha, (iii) or uniformly incorporated into the top 2.5, 5.0, or 7.5 cm of soil simulating field incorporation by disking.

A study was conducted with different aggregate sizes of HBc as a soil cover for reduction of NH₃ losses. Ammonium sulfate was used as the N source. Sieved air-dry soil (8% H₂O by weight) of either 2.5- to 4.5-mm or 0- to 4.5-mm aggregates was placed 1.3 or 2.5 cm deep on HBc at 25% H₂O. A similar study was conducted with air-dry Ha sicl (3% H₂O by weight), with 0- to 4.8mm or 4.8- to 19.0-mm soil aggregates at 2.5- and 5.0-cm depths. The underlying sand was added at either 6% or 23% moisture. Treatments used are given in Table 3.

Statistical analysis of the NH_3 losses from surface-applied NH_4^+ - salts was run using a multiple linear regression program. Additional detail can be found in an earlier publication (4).

RESULTS AND DISCUSSION

Influences of Cation Exchange Capacity on NH₃ Losses

The effect of CEC on NH₃ volatilization from surfaceapplied $(NH_4)_2SO_4$ and NH_4NO_3 is shown in Fig. 1. Losses of NH₃ varied from 14% to 91% of the NH₄⁺-N applied. In general NH₃-N loss decreased as the CEC was increased. The reduction in percentage of NH₃ loss as the CEC increased was greatest at 110 and least at 550 kg NH₄⁺-N/ha.

The water content of the soil-sand mixtures also increased as the CEC's increased. Because of this problem, there was some question if the decrease in NH_4^+ -N loss was due entirely to the increase in CEC. In other unpublished work, we have found NH_4^+ -N volatilization from HBc to be nearly constant over a range of water contents from 0.15 to 0.30 g/g. Because of this, it was felt that CEC exerted the major influence (as opposed to changes in water content) on the results shown in Fig. 1.

Volatilization losses of NH₃ were greater from (NH₄)₂SO₄ than from NH₄NO₃, which agreed with previously reported data (3,4). The difference in NH₃-N losses between these two chemicals was greatest at 12°C (Fig. 1). The percentage of NH₃-N losses from NH₄NO₃ were consistently higher at 110 than at 550 kg NH₄⁺-N/ha except for pure HBc where losses were identical. Losses of NH₃ from $(NH_4)_2SO_4$ were generally highest at the highest NH_4^+-N application rates and lowest at the lowest application rates as reported earlier for HBc (3,4). At 110 kg N/ha and a CEC of 0, (NH₄)₂SO₄ lost 35, 28, and 23% more of applied NH₄⁺-N than NH₄NO₃ at 12, 22, and 32°C, respectively. Differences in NH₃ volatilization at 550 kg NH₄⁺-N/ha and a CEC of 0 were 60, 47, and 30% of applied NH₄⁺-N at 12, 22, and 32°C, respectively. At the same CEC and temperature, two possible differences existed between (NH₄)₂SO₄ and NH₄NO₃: (i) the type of chemical reaction each undergoes with CaCO₃, and (ii) differences in soil pH because of the particular soil chemical reaction. Sauchelli (12) states that an equivalent quantity of $(NH_4)_2SO_4$ is more acidic than NH₄NO₃. However, when placed in a calcareous soil, lower pH values were observed with NH₄NO₃ in this experiment

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Soil mixture,† % sand+ % HBe	CEC	Ammonium chemical	NH4 ⁺ -N application rate	Initial‡ pH	Final § pH
	meq/100 g		kg/ha		
100-0	0	(NH4)2SO4	110 550	7.6 7.4	8.1 8.0
90-38	5.8 5.8	(NH4)2SO4	110 550	7.4 7.4	7.8 7.9
85-15	8.7 8.7	(NH4)2SO4	110 550	7.5 7.4	7.8 7.8
75-25	14.5 14.5	(NH4)2SO4	110 550	7.4 7.4	7.6 7.6
0-199	58.0 58.0	(NH4)2SO4	110 550	7.4 7.4	7.6
100-8	0 0	NH4NO3	110 550	7.4 7.1	7.6 7.1
75- 25	14.5 14.5	NH4NO3	110 550	7.1 6.8	7.5 7.2
50 -58	29.0 29.0	NH4NO3	110 550	7.0 6.8	7.5 7.1
0-190	58.0 58.0	NH4NO3	110 550	7.1 6.8	7.0

Table 2—Soil pH as influenced by cation exchange capacity, ammonium chemical, ammonium-N application rate, and amount of ammonia volatilization.

Tabl	le 3 — Ammonia-nitrogen losses from applied (NH4)2SO4 placement
	depth, surface and subsurface moisture content, and soil
	aggregate size. Nitrogen applied at 550 kg/ha.

Surface

moisture

30 30

8

8

20

20 20

20

3

3

3

Subsurface

soil

moisture

23

23

6

8

Soil

120

101,000

0-4.5

25-45

0-4.5

4.8-19.0

0-4.8

gregati

Applied

loss

%

50

18

88

1 66 52

52

32

0

rogen

† Uniformly mixed.

Experimental

soil

Houston Black

clay

Harkey silty

ciay loam

Covering

soii

depth

CTT

0

2.5

2.5

2.5

0

2.5 2.51

5.0 5.0†

7.5 7.5† 0 2.5

2.5

5.0

Tempera

ture

°C

32 32

32

32

30

30

30 30

† All soil mixtures had at least 10% CaCO by weight.

‡ pH measured immediately after NH4⁺-compound addition.

§ pH measured after approximately 120 hours of NH₃-N losses.

(Table 2). The higher rate of NH_4NO_3 application resulted in the lowest soil pH values and consequently the lowest percentage NH_3 -N losses. Ammonium nitrate, from a practical standpoint, has one-half of its N in the NO_3^- form; therefore, NH_3 losses are much less than from $(NH_4)_2SO_4$ on an equivalent N basis.

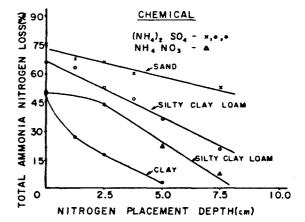
The percentage of NH_3 -N losses dcreased more rapidly at 10 kg NH_4^+ -N/has than at 550 kg/ha as the CEC increased. The more rapid decrease in NH_3 losses at the lower NH_4^+ -N application rates was probably due to a greater percentage of NH_4^+ being absorbed on the cation exchange sites. Precipitation of CaSO₄ would probably occur more quickly at CEC=0 than at higher CEC's. At the higher CEC's the rate of $(NH_4)_2CO_3$ production would likely be reduced because a greater percentage of the NH_4^+ would be adsorbed, resulting in a lower soil solution pH. This explanation is consistent with the final pH values for $(NH_4)_2SO_4$, where the highest pH occurs at the lowest CEC (Table 2).

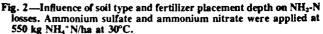
The percent losses of applied N at 110 kg NH4+-N/ha with $(NH_4)_2SO_4$ exceeded the N losses from the 550 kg NH4+-Nha treatment at CEC values of 0 to 4, 6, and 17 meg/100 g soil at 12, 22, and 32°C, respectively. This resulted in the 110 kg NH+-N/ha treatment losing 4, 10, and 18% more NH₃-N than the 550 kg NH⁺-N/ha treatment at 12, 22, and 32°C, respectively, at zero CEC. It seems reasonable that the lowest NH4+-N application rate would have the least loss of N at the highest CEC and losses close to, or equal to losses with the 550 kg/ha treatment at the lowest CEC. The fact that the percentage NH₃-N losses were less at 550 kg NH⁺-N/ha with sand, suggested a slower reaction of (NH₄)₂SO₄ with CaCO₃ within the 100 hour NH₃-N collection time. The percent loss of applied NH4+-N (110 kg/ha) from a sand as NH₃-N was 91, 90, and 89% at 12, 22, and 32°C, respectively. Percent NH₃-N losses at 550 kg N/ha were 87, 80, 71% of applied NH+-N, respectively, at 12,

22, and 32°C. Previous work (6) indicated that 10% CaCO₃ by weight was necessary for maximum NH₃ loss. The prior research, however, was done on a soil with a CEC of 26 meq/100g. Previous losses of NH₃ were approximately 50% of applied NH₃⁺-N as compared to the present maximum 91% loss recorded on sand. Since NH₃-N losses with low CEC soils were so high, greater CaCO₃ contents would have decreased the necessary reaction time at high (NH₄)₂SO₄ application rates. Collection of the evolved NH₃ for 200 hours at the high N application rate, however, resulted in NH₃ loss values approximately equal to that collected in 100 hours at 110kgN/ha. The 200-hour NH₃ losses were approximately 89, 89, and 72% of applied NH⁺-N at 12, 22, and 32°C, respectively.

Influence of Depth of Placement on NH₃-N Loss

The depth from which significant NH_3 -N can be lost from applied N fertilizers is important in determining remedial cultural practices. The water content of this soil cover could also be managed to some extent if it saved N for the plant environment. Since a soil cover is the most practical means of reducing NH_3 -N losses, additional knowledge in this area is needed.





MOIST SOIL COVER

The influence of soil CEC on NH₃-N retention with respect to depth of placement of the NH₄⁺-compound is shown in Fig. 2 and Table 3. Placement of $(NH_4)_2SO_4$ in sand at a depth of 7.5 cm resulted in an NH₃-loss reduction from 75 to 60% of applied NH₄⁺-N. In Ha sicl at the same depth NH₃-N losses were reduced from 69 to 25% with $(NH_4)_2SO_4$ and from 51 to 10% of applied NH₄⁺-N with NH₄NO₃. A 10 to 20% lower NH₃-N loss value was observed for NH₄NO₃ than for $(NH_4)_2SO_4$ at all placement depths (Fig. 2). Even though the volatile NH₃ from the $(NH_4)_2SO_4$ and NH₄NO₃ treatments moved through 7.5 cm of soil, NH₃ losses from $(NH_4)_2SO_4$ remained greater than from NH₄NO₃.

Houston Black clay with the same pH as Ha sicl lost less of the applied NH₄⁺-N with increasing depth of placement. Less than 2% NH₃-N was lost when $(NH_4)_2SO_4$ was placed at 5.0 cm and none at 7.5 cm in HBc. Cation exchange capacity effects on NH₃ loss were more effective when the NH₄⁺-compounds were incorporated into the soil (Fig. 2, Table 3). Houston Black clay has a CEC of 58 meq/100 g and Harkey sicl has a CEC of 22 meq/100 g. If the NH₃ has to diffuse through the soil, it is more likely to be adsorbed in a high CEC soil. If the soil has a low CEC, the NH₃-N losses can be large with compounds such as NH₄NO₃, and even greater with compounds like (NH₄)₂SO₄.

The uniform mixing of $(NH_4)_2SO_4$ into Ha sicl simulating field incorporation by disking, increased NH₃-N losses (Table 3). A fertilizer placement depth of 2.5, 5.0, and 7.5 cm resulted in NH₃-N losses of 52, 35, and 26%, respectively, whereas N losses from mixing the fertilizer into the 2.5, 5.0, and 7.5 cm of soil were 60, 45, and 29%, respectively. The NH₃-N losses are 3 to 10% greater from mixing the fertilizer into the soil than from the placement of all fertilizer at 2.5, 5.0, and 7.5 cm depth.

DRY SOIL COVER

A dry soil cover was more effective in reducing NH₃-N losses than a moist soil cover (Table 3). Ammonia-nitrogen losses with a cover of 2.5 cm (0- to 4.5-mm aggregates) of HBc reduced the NH₃-N loss by 49% of applied NH⁺-N. A coarser HBc soil aggregate (2.5- to 4.5-mm) placed 2.5 cm deep over (NH₄)₂SO₄ resulted in a 17% reduction in NH₃ loss when compared to surface-applied (NH₄)₂SO₄. Placement of (NH₄)₂SO₄ under a dry soil cover of Ha sicl with a water-saturated sand subsoil, however, resulted in large NH₃-N losses. The NH₃-N losses with 2.5 cm air dry Ha sicl over $(NH_4)_2SO_4$ on a water-saturated sand subsoil was 52% of applied N, identical to the loss with a wet soil cover (Table 3). Ammonium-nitrogen applied at the same depth, but with a drier subsurface sand (6% water by weight), lost only 32% of the applied NH4+-N. An increased placement depth of 5.0 cm of air dry Ha sicl over the same drier subsoil completely eliminated NH₃-N loss. McDowell and Smith (11) and Jackson and Chang (8) also noted that drier soils lost less NH₃-N. If evaporation of water from the soil was largely stopped, as was probably the case with a dry soil cover in this experiment, adsorption of NH₃ and water by the dry soil could explain our observations.

Physical movement of NH₃ to the soil surface through

capillary water movement may be a major factor causing NH_3 losses from subsurface applied NH_4^+ -salts. Without capillary water movement, large quantities of NH_3 - NH_{4+} may never reach the soil surface.

A REGRESSION EQUATION MODEL FOR PREDICTING NH₃-N LOSSES

Two regression equations predicting NH₃-N loss from surface-applied NH₄⁺-compounds were derived using the following variables: time (t), temperature (T), ammoniumnitrogen application rate (U), cation exchange capacity (CEC), average soil pH for 0 and 120 hours after fertilizer addition (pH), NH₃-N loss as a percent of added NH₄⁺-N (X). Separate and highly significant regression equations were derived for (NH₄)₂SO₄ and NH₄NO₃. The regression equation for (NH₄)₂SO₄ is as follows:

 $X = 786.995 - 0.132U + 0.585 T + 0.541 \text{ CEC} + 1.923t + 103.087 \text{ pH} + 2.112 \times 10^{-4}U^2 - 8.838 \times 10^{-3} \text{ CEC}^2 - 0.0295t^2 + 2.145 \times 10^{-4}t^3 - 5.694 \times 10^{-7}t^4$. $R^2 = 0.86$

The corresponding regression equation for NH_4NO_3 is as follows:

 $X = -192.800 - 0.0264U + 0.659T - 0.658 \text{ CEC} + 0.582t + 26.430 \text{ pH} + 4.613 \times 10^{-5}U^2 + 8.027 \times 10^{-3} \text{ CEC}^2 - 5.48 \times 10^{-3}t^2 + 2.906 \times 10^{-5}t^3 - 5.363 \times 10^{-8}t^4$. $R^2 = 0.81$.

The program used for multiple linear regression analysis (4), in this study did not permit forcing of the curves through zero. Consequently, the equation will not predict NH_3 losses for the first 6 hours after solubilization of the NH_4^+ -compound. The regression equation tends to overpredict many values at 6 to 10 hours and underpredict the 90- to 100-hour NH_3 loss values. The errors in predicted NH_3 loss values at 6 to 10 hours varied from 25% to 40%; beyond 90 hours it varied from 10 to 12% from measured NH loss values.

The poorer performance of the regression equation for NH_3 volatilization from NH_4NO_3 probably resulted from a poor approximation of the effect of added acidity. Some other means for determining the effect of acidity might improve the final equation. The principal limitation of these equations are (i) an air flow in excess of 0.16 km/hour (0.1 mph) near the soil surface, (ii) all experimental soils have been of montmorillonitic mineralogy, (iii) lack of NH_3 -N loss predictability during the first 6 hours, and (iv) the fact that these are laboratory data which have not been tested under field conditions.

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A Technique to Determine Iron Efficiency in Plants¹

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ABSTRACT

Soil tests should predict crop response to fertilization. In the case of Fe, plant factors seem to affect the use of Fe by the plant; thus, soil tests may not be reliable. Use of soil Fe by plants is genetically controlled by an adaptive mechanism which is activated in Fe-efficient plants in response to Fe-stress, but remains inactive if Fe is sufficient. Fe-inefficient plants develop less Fe-stress response than Fe-efficient plants. Using this range of Fe-stress response in plants as a basis, we developed a technique to screen plants for Fe efficiency.

A limited supply of Fe and some control of pH in the growth meflum were required in this technique. The pH was controlled by using matrient solutions, N only as No₃, and N as NH₄ and NO₃, and by varying the CaCO₃ and P concentrations in the solutions. The optimum supply of Fe (0.2 mg Fe/liter) was determined by growing the plants at different Fe concentrations.

Four Fe-inefficient and four Fe-efficient plant varieties, representing four plant species, were used as test plants. The plants were screened in mixed cultures and were rated in order of greatest Fe efficiency as follows:

T3238FER tomato (Lycopersicon esculentum Mill.) \geq Hawkeye soybean (Glycine max (L.) Merr.) > W59 Corn (Zea mays L.) > Plomeer 846 sorghum (Sorghum bicolor (L.) Moench) > PI-54619-5-1 soybean > Wheatland sorghum > ys₁/ys₁ corn > T3238fer tomato.

Green plants contained more and chlorotic plants contained < 43 µg Fe/g, but the degree of Fe-chlorosis was the best index of Feefficiency. A suggested sequence for screening plants for Fe-efficiency is presented.

Additional Index Words: Fe-stress response, Fe-inefficient plants, Fe-efficient plants, Fe-stress.

THE ROLE of a soil test is to predict crop response to fertilization (11). Cox and Kamprath (11) discussed several soil tests for Fe that use ammonium acetate (15, 1), EDDHA [ethylenediamine di (*o*-hydroxyphenylacetic acid)] (1), and DTPA (diethylenetriaminepentaacetic acid) as soil entractants. The DTPA procedure was used by Lindsay and Norvell.³ Cox and Kamprath (11) concluded that the availability of Fe seems to depend on many factors aside from an extractable amount of Fe in the soil. They indicated that some of these factors may be inherent in the plant and that until these plant factors are understood, soil tests for Fe may not be reliable.

We attempted to develop a soil test for available Fe in soils (3, 6, 4) and also learned that available Fe in a soil depends on the crop grown. For example, when Millville, Tripp, and Quinlan soils (3) were extracted with DTPA (3, 6) and EDDHA (4), the available Fe in Quinlan < Tripp < Millville. When Wheatland sorghum (Sorghum bicolor (L.) Moench) was used as a test plant, the degree of Fe deficiency was related to the amount of chelate-extractable Fe in the soil. But when PI-54619-5-1 (PI) and Hawkeye (HA) soybeans (Glycine max (L.) Merr.) were used as test plants, PI-soybeans developed Fe chlorosis and HA-soybeans were green, respectively, on all three soils. These observations showed that the plant varieties were affecting the uptake of Fe from the soil (8).

The total quantity of Fe in a soil is usually sufficient for plant growth, but the use of this Fe by plants is genetically controlled by an adaptive mechanism (8) which is activated in Fe-efficient plants in response to Fe stress, but which remains inactive if Fe is sufficient.

Iron-efficient plants release H ions and "reductants" from their roots when they are under Fe stress. The pH at their root zone is lowered which favors Fe^{3+} solubility and reduction of Fe^{3+} to Fe^{2+} . This Fe is reduced externally by the root because it can be prevented from entering the plant

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³W. L. Lindsay, and W. A. Norvell. 1969. Development of a DTPA micronutrient soil test. Agron. Abstr. p. 84.