

many commercial mixes. Development of management strategies to compensate for those possible deleterious effects from the use of peats will require further research.

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# Ferric Ethylenediamine-tetraacetic Acid Photodegradation in a Commercially Produced Soluble Fertilizer Affects Iron Uptake in Tomato

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**ADDITIONAL INDEX WORDS.** plant nutrition, iron chelate, FeDTPA, photochemistry

**SUMMARY.** Irradiating a ferric ethylenediaminetetraacetic acid (FeEDTA)-containing commercially available soluble fertilizer with ultraviolet (UV) and blue radiation from high intensity discharge (HID) lamps caused the photooxidation of the FeEDTA complex, resulting in the loss of 98% of soluble iron. The loss of soluble iron coincided with the development of a precipitate that was mostly composed of iron. The effects of using an irradiated FeEDTA-containing fertilizer solution on plant growth and nutrition under commercial conditions were studied. Application of the irradiated fertilizer solutions to greenhouse grown tomato plants (*Lycopersicon esculentum*) resulted in lower levels of iron (6%) and zinc (9%), and higher levels of manganese (8%) and copper (25%) in leaf tissue compared to control plants that received a nonirradiated fertilizer solution. Leaf macronutrient levels (phosphorous, potassium, calcium, and magnesium), leaf dry weight, leaf number, and plant height was not affected by application of the irradiated fertilizer solution.

We thank Martin Kinnamon and Isagusbel Padilla for technical assistance. Use of trade names does not imply endorsement of the products named nor criticism of similar ones not named.

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The application of soluble fertilizers in greenhouse plant production is necessary to maintain plant growth under optimal environmental conditions. To supply Fe, Mn, Cu, and Zn in a soluble form, these metals are often chelated in the fertilizer solution with EDTA [also DTPA (diethylenetriamine-pentaacetic acid), EDDHA (ethylenediamine-di-o-hydroxyphenylacetic acid), and/or HEDTA (hydroxyethylenediaminetriacetic acid) for Fe]. Iron chelates are chromophores (i.e., chemical compounds that absorb light/colored chemical compounds) that absorb strongly in the UV and blue regions of the spectrum. Absorption of this energy causes the photodegradation (photooxidation) of the Fe-chelate complex (Hamaker, 1956). The chelating agent EDTA when complexed with Fe and exposed to UV and blue radiation, photooxidizes into glyoxylic acid, carbon dioxide ( $\text{CO}_2$ ), formaldehyde, and amine residue(s) (Frissel et al., 1959; Hamaker, 1956). In previous studies, photodegradation of FeEDTA- or FeDTPA-containing nutrient solutions resulted in the loss of soluble Fe and the formation of a precipitate that was mostly composed of Fe (Albano and Miller 2001a, 2001b). Marigolds (*Tagetes erecta*) grown hydroponically in an irradiated FeDTPA-containing nutrient solution had lower levels of Fe and higher levels of Mn in leaf tissue, and greater root-associated ferric-chelate reductase activity than in plants grown in a nonirradiated nutrient solution (Albano and Miller, 2001c). An enhanced ability of roots of dicots and nongraminaceous monocots to reduce ferric chelates when Fe is unavailable in the rhizosphere is a physiological trait associated with strategy I Fe efficiency (Bienfait, 1988). The effects, however, of using a commercially available Fe-chelate-containing fertilizer solution that has been irradiated on greenhouse plant production under commercial-type conditions is unknown. Therefore, the objective of this study was to determine under commercial-type conditions the effects of an irradiated FeEDTA fertilizer solution on leaf nutrient levels and plant growth.

## Materials and methods

**TREATMENTS.** Plantex Florida Special [20-10-20 (20N-4.3P-16.6K); Plantex Corp., Brampton, Ontario, Canada] water-soluble fertilizer was

prepared as a 100× stock solution based on a 1× concentration of 200  $\text{mg}\cdot\text{L}^{-1}$  (ppm) N by dissolving one 11.3-kg (25 lb) bag of dry fertilizer into 114.4 L (30.22 gal) of distilled-deionized (DDI) water. After thoroughly mixing, 5 L (1.3 gal) of the 100× fertilizer solution was poured into a 50-L (13.2-gal) Nalgene low-density polyethylene (LDPE) carboy (Nalgene Co., Rochester, N.Y.), brought to volume with DDI water (yielding a 10× fertilizer solution), mixed, and then divided into two 25-L (6.6-gal) LDPE Nalgene carboys. Fertilizer solutions (25 L) were irradiated or nonirradiated for 15 d with 1400  $\mu\text{mol}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$  (400 to 700 nm), 24  $\mu\text{mol}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$  (250 to 400 nm) measured at the external container surface with a radiometer-quantum sensor (LI-250; LI-COR, Lincoln, Nebr.) and UV meter (Apogee Instruments Inc., Logan, Utah), respectively. Carboys were placed on their sides for irradiation and nonirradiated containers were covered with aluminum foil. Irradiance intensity was varied by adjusting lamp-bank distance from solution containers. The radiation source was a combination of 400-W HID metal halide (Sylvania M400/U; OSRAM Sylvania LTD, Danvers, Mass.) and high-pressure sodium lamps (Sylvania LU400) (one lamp of each type above each container). The study was conducted in a controlled environment growth chamber and solution temperature was maintained at 20 °C (68.0 °F) by adjusting air temperature. Treatments derived from the irradiated and nonirradiated fertilizer solutions consisted of two unaltered solutions: 1) nonirradiated (NI) and 2) irradiated with precipitate (that formed when solution was irradiated) remaining in solution (I+P); and one altered solution: irradiated with precipitate removed (I-P) by centrifugation as previously described (Albano and Miller, 2001a). Elemental concentration [(P, K, Fe, Mn, Cu, and Zn) determined by ICP (inductively coupled argon plasma-atomic emission spectrometry)], and pH of the 1× NI and I-P treatment solutions are presented in Table 1. FeEDTA was qualitatively measured in the NI and I-P fertilizer solutions (10× concentration) spectrophotometrically at 258 nm (the wavelength that FeEDTA maximally absorbs) (Hill-Cottingham, 1957).

**GROWING CONDITIONS.** ‘Florida 91’

tomato seeds were sown in Fafard 4P soilless medium (Fafard Inc., Anderson, S.C.) in six-celled grow packs [40  $\text{cm}^3$  (2.4  $\text{inch}^3$ ) per cell, three seeds per cell, 30 Apr. 2001] in a greenhouse at the U.S. Horticultural Research Laboratory, Fort Pierce, Fla. with a heating/venting temperature of 16/27 °C (60.8/80.8 °F), respectively. A pack of six plants constituted a single replication and six replications of each treatment (NI, I+P, and I-P) were made. Plants were thinned to one plant per cell at the emergence of cotyledons, and treatments were initiated 19 d after sowing when the first leaf was expanding. Treatment solutions were prepared from the 10× fertilizer stock solutions previously described with DDI water, and were applied [325 mL (11.0 fl oz)] per six-cell grow pack every other day, avoiding any application to foliage. Carboys containing treatment solutions were agitated prior to drawing 325 mL of solution for application to grow packs. A total of seven treatment applications were made to plants in grow packs with treatment applications 1 to 4 and 5 to 7 formulated as 0.5× (100  $\text{mg}\cdot\text{L}^{-1}$  N) and 1× (200  $\text{mg}\cdot\text{L}^{-1}$  N) solutions, respectively. Leaching fraction averaged 25% over the course of the study. Thirteen days after initiating treatments (32 d after sowing), five of the six plants per grow pack were randomly selected for harvest. At harvest, leaf number and stem length (measured from cotyledon node to apical meristem) were recorded. Leaves were harvested, washed, dried, dry weight recorded, and prepared for elemental analysis by ICP as described previously (Albano et al., 1996). The study was repeated with the following modifications: 1) treatments were initiated 15 days after sowing (seeds were sown on 9 July 2001), and 2) DDI water (325 mL) was applied to grow packs on days 9 and 11 after initiating treatments to prevent wilting. The average greenhouse min/max temperatures over the course of the initial and repeat experiments were 22/30 °C (71.6/86.0 °F) and 23/30 °C (73.4/86.0 °F), respectively.

**MEDIUM ANALYSIS.** Soluble minerals in Fafard 4P medium were determined by ICP on the extract obtained using a modified 1:2 dilution method as described by Lang (1996). Medium [200  $\text{cm}^3$  (12.2  $\text{inch}^3$ )] from a newly opened bag of medium was diluted with 400 mL (13.5 fl oz) of DDI

**Table 1.** Phosphorous, K, Fe, Mn, Zn, and Cu concentration of supernatant and pellet fractions derived from nonirradiated (NI) and irradiated-precipitate removed by centrifugation (I-P) of Plantex 20-10-20 (20N-4.3P-16.6K) fertilizer solutions (1× concentration). pH of the NI and I-P fertilizer solutions (1× concentration). FeEDTA in the NI and I-P fertilizer solutions (10× concentration) was qualitatively measured spectrophotometrically at 258 nm (absorbance). Fertilizer solutions were formulated as a 10× stock based on a 200 mg L<sup>-1</sup> (ppm) nitrogen (N) 1× concentration and were irradiated or nonirradiated with 1400 μmol m<sup>-2</sup> s<sup>-1</sup> for 15 d.

Element	NI <sup>z</sup>	I-P	Loss due to irradiation		Recovered in pellet	
	(mg L <sup>-1</sup> ) <sup>y</sup>	(mg L <sup>-1</sup> )	(mg L <sup>-1</sup> )	(%)	(mg L <sup>-1</sup> )	(%)
P	54.40	51.13	3.27	6.0	0.23	7.0
K	143.33	139.30	4.03	2.8	0.07	1.7
Fe	0.47	0.01	0.46	97.9	0.39	84.8
Mn	0.28	0.26	0.02	7.1	0.01	50.0
Zn	0.40	0.35	0.05	12.5	0.02	40.0
Cu	0.27	0.26	0.01	3.7	0.00	0.0
pH	5.2	5.8	---	---	---	---
Absorbance	1.107 AU <sup>x</sup>	0.373 AU <sup>x</sup>	---	---	---	---

<sup>z</sup>No precipitate formed in the nonirradiated fertilizer solution.

<sup>y</sup>1 mg L<sup>-1</sup> = 1 ppm.

<sup>x</sup>absorbance units.

**Table 2.** Effects of irradiation of fertilizer solutions [nonirradiated (NI), irradiated with precipitate retained (I+P), and irradiated with precipitate removed by centrifugation (I-P)] on leaf Fe, Mn, Cu, and Zn concentration of 'Florida 91' tomato plants (n = 6).

Treatment	Leaf mineral concentration (mg·g <sup>-1</sup> ) <sup>z</sup>			
	Fe	Mn	Cu	Zn
NI	90 a <sup>y</sup>	75 b	12 b	74 a
I+P	84 b	79 ab	16 a	67 b
I-P	85 b	84 a	16 a	67 b

<sup>z</sup>1 mg·g<sup>-1</sup> = 1 ppm.

<sup>y</sup>Mean separation within columns by LSD, *P* ≤ 0.05.

water, stirred, allowed to equilibrate for 45 min, and then filtered [gravity filtered (Whatman no. 41; Whatman Paper Ltd., Maidstone, Kent, England)].

Statistics. Data were analyzed by analysis of variance (ANOVA) to determine the effect of experiment and treatment. Calculations were performed with the general linear model (GLM) procedure of SAS (SAS Institute, Inc., Cary, N.C.). Means were separated and planned comparisons were made using LSD or pairwise *t* tests.

## Results and discussion

At the end of the irradiation period, only 2% of Fe remained soluble in the irradiated fertilizer solution (Table 1). Consistent with previous studies, a precipitate formed in the irradiated fertilizer solution associated with a decrease in absorbance at 258 nm and a rise in solution pH, indicating the destruction of FeEDTA with irradiation

(Table 1) (Albano and Miller, 2001a, 2001b). Analysis of the precipitate indicated that it was primarily composed of Fe, accounting for 84% of the soluble Fe lost due to irradiation with smaller amounts of other elements (Table 1). These data are consistent with previous studies with other commercially produced soluble fertilizers that contained FeEDTA and lab-prepared nutrient solutions that contained FeDTPA that were similarly irradiated (Albano and Miller, 2001a, 2001b).

At harvest, plants had 5 to 6 leaves. Leaves appeared normal with no signs of symptoms and/or abnormalities associated with nutrient deficiency or toxicity. As an average of both experiments, leaf dry weight per plant and plant height varied slightly between treatments and averaged 0.85 g (0.030 oz) and 12.80 cm (5.039 inches), respectively (data not shown).

Foliar levels of calcium (Ca), magnesium (Mg), Cu, Fe, K, Mn, P, and

Zn were, as an overall average, 20% higher in experiment 1 than in experiment 2. This is most likely due to greater medium leaching with the additional applications of DDI water in experiment 2. There were, however, no experiment by treatment interactions, therefore data are presented as an average of both experiments. The levels of Ca, K, P, and Mg in leaf tissue were not affected by treatment and averaged 17, 25, 9, and 5 mg·g<sup>-1</sup> (1.7%, 2.5%, 0.9%, and 0.5%), respectively (data not shown). These levels are considered adequate for tomato plants at first flowering stage (Hochmuth et al., 1999).

Leaf Fe, Mn, Cu, and Zn levels were not different between the two irradiated treatments (I+P and I-P), indicating that the insoluble Fe supplied in the I+P treatment was not readily available for uptake (Table 2). Leaf Fe, Mn, Cu, and Zn levels were different from the NI treatment except for Mn in the I+P treatment, which was not different from the NI treatment (Table 2), indicating that soluble Fe derived from the medium (Table 3), while apparently sufficient to maintain normal plant growth, did not supply enough Fe to counter the effects of the irradiated solutions [i.e., supplying no Fe (I-P) or insoluble Fe (I+P)]. As an average of both irradiated treatments (I+P and I-P), foliar levels of Fe and Zn were 6% and 9% lower, and Mn and Cu were 8% and 25% higher, respectively, in irradiated than in NI treated plants (Table 2). For all treatments, foliar levels of Fe, Mn, Cu, and Zn were considered adequate [40 to 100 μg·g<sup>-1</sup> (ppm) Fe, 30 to 100 μg·g<sup>-1</sup> Mn, 5 to 10 μg·g<sup>-1</sup> Cu] or high (25 to 40 μg·g<sup>-1</sup> Zn) for tomato plants at first flowering (Hochmuth et al., 1999). These data are consistent with a previous study by Welch et al. (1993) where pea (*Pisum sativum*) grown hydroponically in a minus Fe nutrient solution had higher levels of Mn and Cu and lower levels of Fe and Zn in leaf tissue compared to the control (Fe sufficient) 14 d after seed imbibition. These data are also consistent with our previous study where hydroponically grown marigolds in a similar irradiated nutrient solution containing FeDTPA had higher levels of Mn (I-P) and lower levels of Fe (I-P and I+P) in leaves compared to plants grown in nonirradiated solutions (Albano and Miller, 2001c). In that study, mari-

Table 3. Phosphorous, K, Ca, Mg, Fe, Mn, Zn, and Cu concentration of a newly opened bag of growing medium (Fafard 4P) extract. Extracts were acquired by 1 medium : 2 water dilution method on 200 cm<sup>3</sup> (12.2 inch<sup>3</sup>) of medium and a 45 min equilibration time.

P	K	Mineral concn of medium extract (mg L <sup>-1</sup> ) <sup>z</sup>					
		Ca	Mg	Fe	Mn	Zn	Cu
4.69 ± 0.25 <sup>y</sup>	88.26 ± 2.45	118.27 ± 2.70	75.38 ± 2.46	0.33 ± 0.06	0.23 ± 0.02	0.23 ± 0.01	0.00 ± 0.00

<sup>z</sup>1 mg L<sup>-1</sup> = 1 ppm.

<sup>y</sup>Values are means ± SE (n = 3).

golds grown in irradiated FeDTPA nutrient solutions had higher root-associated ferric (Fe<sup>3+</sup>)-chelate reduction rates than plants grown in the nonirradiated nutrient solutions, a physiological trait common to Fe-efficient, strategy I (dicots and nongraminaceous monocots) plants under Fe deficiency stress. The Fe<sup>3+</sup>-chelate reductase system might also have activity for Mn (Marschner et al., 1982) and Cu (Welch et al., 1993). Root-associated Fe<sup>3+</sup> reduction was not determined in the present study, but other studies have documented higher rates of Fe<sup>3+</sup> reduction of tomato plant roots under Fe deficiency (Zaharieva and Römhelt, 2000). Therefore, it is possible that the elevated levels of Mn and Cu in leaf tissue of plants receiving the irradiated fertilizer solutions may be the result of enhanced Fe<sup>3+</sup>-chelate reductase activity.

In summary, although the foliar levels of Fe, Mn, Cu, and Zn between the nonirradiated and irradiated (I+P and I-P) treatments were found to be statistically different, these differences are most likely not of commercial significance. It appears, at least for short-term greenhouse crops, under conditions similar to those we used, that a commercial medium mix containing a complete nutrient starter charge will supply sufficient levels of soluble Fe to maintain normal plant growth of plants receiving a liquid fertilizer containing insoluble Fe, as was the case with the irradiated fertilizer solutions in this study.

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