THE CAUSE OF YELLOW TIPPING IN CITRUS LEAVES

IVAN STEWART AND C. D. LEONARD

Florida Citrus Experiment Station

Lake Alfred

Yellowing tipping of citrus leaves has been observed in Florida for over 20 years. The present investigation has disclosed that the trouble is caused by perchlorates that occur as impurities in fertilizers. The symptoms of this toxicity shows up first as a bright yellow-orange color on the tip of leaves. As the condition becomes worse, the orange color may spread over the entire tip half of the leaves (FIG. 1). The leaf veins usually remain green; only in advanced stages do they become chlorotic. In severe cases the trees partially defoliate.

Yellow tipping is often confused with boron toxicity. In both instances the leaf tips are first to be affected. However, trees that have taken up excess boron usually – but not always – show gumming on the lower leaf surfaces; while the chlorosis is usually a dull yellow color. It is believed that yellow tipping shows up first on the parts of trees most exposed to the sun. This accounts for the fact that the yellow leaves are always found first in the outer exposed rows and in the tops of the trees in thick groves. The chlorosis generally shows up only on the upper leaf surface; however, if the leaves are turned over the bottom surface, exposed to the sun, will show yellowing.

In 1932, Haas (2) published photographs of orange and lemon leaves with symptoms resembling yellow tip. The leaves were collected from California groves that had received large applications of calurea. The only known mention in the literature of yellow tipping in Florida citrus was a photograph published by Bryan (1) and called unidentified leaf symptoms. He reported that the leaf condition was somewhat similar to the symptoms of boron toxicity, and was thought to be associated with excess boron in certain fertilizers. It was pointed out, however, that this assumption had no experimental proof.

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At the Citrus Experiment Station, Lake Alfred, chemical analyses made on leaves having yellow tipping were compared with those from trees showing boron toxicity (Table 1). The highest amount of boron found in the yellow tip leaves was 49.0 ppm. This is considerably below the level found in leaves showing boron toxicity.

In Florida, yellow tipping has been associated with the use of natural nitrate of potash derived from Chilean sources. The symptoms have not been reported in groves that have used the synthetic salt. When studies were started on this problem, applications of 22 pounds of nitrate of potash were made to single trees. In approximately three months, yellow tipping was prevalent in the leaves. From the observations made it was concluded that a toxic substance must be present in the fertilizer.

A study of a commercial chemical analysis disclosed that the fertilizer contained halogenates – chlorates, perchlorates and iodates. Applications of each of these were made as soil treatments to healthy grapefruit trees. Approximately one month later trees receiving chemically pure potassium perchlorate ($KClO_4$) showed symptoms similar to yellow tipping. Applications of 1, 1/2, and 1/4 pounds of potassium perchlorate were then made to grapefruit trees and, in addition,

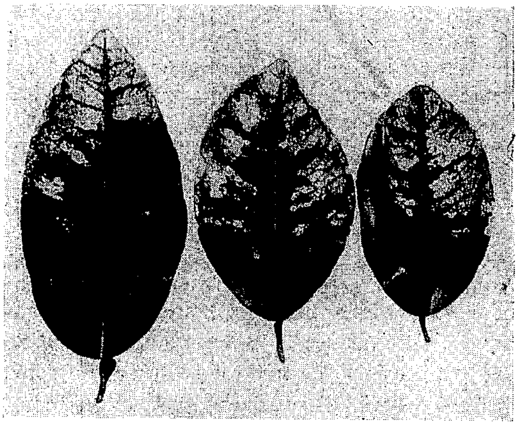

Fig. 1. Grapefruit leaves showing yellow tipping caused by perchlorate impurities in fertilizer.

equivalent amounts of perchlorates were applied to other trees in the form of the calcium and sodium salts. Seven pounds of nitrogen (N) was applied to each tree in the form of ammonium nitrate.

The trees receiving the highest rates of perchlorates showed yellow tipping in approximately one month; while those that received the intermediate and low rates showed yellow leaves in two and three months, respectively, following the applications. Trees treated at the same time with 50 pounds of natural nitrate of potash had symptoms of about the same severity as those that had received the 1/2 pound application of potassium perchlorate. In every way the symptoms appeared identical.

Perchlorate toxicity symptoms were more severe in the absence of nitrogenous fertilizer. Trees treated with one pound of potassium perchlorate, without the addition of nitrogen, developed yellow tipping. Later the entire leaves became copper colored and severe defoliation followed. Nitrates apparently have an inhibiting effect on the action of perchlorates in plants. This inhibition of perchlorate toxicity by nitrates has also been observed in Germany (3) on field crops.

Chemical analyses for perchlorates were made on the leaves. It was found necessary to develop a new procedure for this determination. Tests showed that, when chlorine was supplied to trees as sodium chloride or sodium chlorate, chloride could be extracted from the dry leaves with hot water. This indicated that the sodium chlorate was reduced either before it entered the trees or within the tree itself. However, perchlorates are more stable than chlorates. The only common procedure for reducing them to chlorides is with heat. Indications are that the perchlorate ion is not reduced but is taken up by the plant per se.

Leaf samples were collected from trees that had received soil treatments of perchlorates. The highest amounts of perchlorates were found in leaves from trees that had received the higher soil applications, and the amount found in the leaves was correlated with the leaf symptoms. Leaves taken from the tops of trees where the toxicity was severe contained higher concentrations of perchlorates than those collected from the lower limbs where very little yellow tipping was found (Table 2).

The symptoms were much more severe on trees that received sodium perchlorate than on trees receiving the calcium or potassium salts. Leaves from the old flush contained almost twice as much perchlorates as leaves from the new flush (Table 3). This is in agreement with the leaf symptoms; in that yellow tipping can be found on the old leaves first. Since the chlorosis is almost always present on the tips of leaves and usually absent on the basal half, chemical analyses were made on the tip one-third of affected leaves and compared with the base one-third. The perchlorate content in the tips was over five times that found in the base (Table 3).

A sand-culture study was made to observe the effect of different concentrations of perchlorate on citrus seedlings. Three seedlings each of sweet orange, grapefruit, and Cleopatra mandarin were put in pots. They were given one liter of nutrient solution daily containing 0, 5, 10, 20, 50, 75, and 100 ppm. ClO_4 supplied as $NaClO_4$. In less than one month, the grapefruit seedlings receiving 100 ppm. ClO_4 showed extensive yellow tipping. One leaf on a sweet orange seedling showed a yellow pattern. The leaves on the Cleopatra did not show any yellow tipping, but the leaf tips became burned and turned black. The growth was reduced in all the seedlings at the highest perchlorate rate. Two months after the experiment was started, grapefruit seedlings in pots given rates of 20 ppm. and over showed yellow tipping. The sweet orange and Cleopatra seedlings developed toxicity symptoms only at the highest rate. This experiment would tend to confirm field observations that varieties may differ in sensitivity to perchlorates.

It has been known for more than 50 years that Chile caliche (from which natural nitrate fertilizers are derived) contains up to seven percent potassium perchlorate (5). Considerable trouble developed in Europe on field crops from using sodium nitrate that contained perchlorates as impurities. It was observed (4) that sodium nitrate did not cause injury to field crops when less than one percent perchlorates were present.

The test used in this study was not sensitive enough to detect any perchlorates in the natural sodium nitrate now being produced.

However, chemical analyses made on samples from current production of natural nitrate of potash showed that it contains more than 0.5 percent halogenates (Table 4). Field studies indicate that practically all of the halogenates found are perchlorates. The amounts present are enough to cause yellow tipping in citrus when sufficiently large applications of nitrate of potash are made. It is understood that, on the basis of this work, steps have been taken to lower the concentration of perchlorates in natural nitrate of potash.

SUMMARY

Yellow tipping of citrus leaves, which has been observed in Florida groves for many years, has been found to be caused by perchlorate toxicity. Yellow tipping was produced with soil applications of nitrate of potash derived from Chile caliche, which contains perchlorates, and with chemically pure potassium, calcium, and sodium perchlorates. Chemical analysis of leaves from trees treated with perchlorates showed that these salts are taken up in the oxidized state by the trees. A higher concentration of perchlorates was found in leaves from the tops of trees than in leaves from the bottom branches. Grapefruit seedlings grown in sand cultures were found to be much more sensitive to perchlorate toxicity than either Cleopatra mandarin or sweet orange. Chemical analysis of nitrate of potash fertilizers showed the halogenate content to be over 0.5 percent, most of which is believed to be perchlorates.

TABLE 1

Boron Content of Grapefruit Leaves from Trees Showing Boron Toxicity Symptoms and from Trees Showing Yellow Tipping.

Degree of Symptoms	Boron ppm.
Slight Boron Toxicity Symptoms	159.0
Slight Boron Toxicity Symptoms	135.0
Moderate Boron Toxicity Symptoms	290.0
Severe Boron Toxicity Symptoms	675.0
Severe Boron Toxicity Symptoms	510.0
No Boron Toxicity Symptoms	62.5
Slight Yellow Tipping	12.5
Moderate Yellow Tipping	49.0
Moderate Yellow Tipping	27.5
Severe Yellow Tipping	10.0

Table 2

Perchlorate Content of Grapefruit Leaves Taken from Trees Treated with Nitrate of Potash, Sodium Chlorate, and Perchlorate Salts.

Salt Applied	Amount of Salt* Applied per Tree (Pounds)	Position Sample Taken From Trees	ClO_4^- mgm./gm. Dry Wt.
$KClO_4$	1.00	Top	2.15
$KClO_4$	1.00	Bottom	1.78
$KClO_4$	.50	Top	2.54
$KClO_4$	.50	Bottom	1.32
$KClO_4$	.25	Top	1.37
$KClO_4$	.25	Bottom	1.23
$NaClO_4$	.88	Top	5.10
$NaClO_4$	.88	Bottom	2.85
$NaClO_4$	.44	Top	4.12
$NaClO_4$	.44	Bottom	2.83
$NaClO_4$	.22	Top	2.13
$NaClO_4$	.22	Bottom	1.02
$Ca(ClO_4)_2$	.86	Top	4.57
$Ca(ClO_4)_2$	.86	Bottom	2.54
$Ca(ClO_4)_2$	.43	Top	6.14
$Ca(ClO_4)_2$	.43	Bottom	1.70
$Ca(ClO_4)_2$	.23	Top	.85
$Ca(ClO_4)_2$	.23	Bottom	1.04
$NaClO_3$	1.00	Top	0.00
$NaClO_3$	1.00	Bottom	0.00
KNO_3	25.00	Top	2.13
KNO_3	25.00	Bottom	1.98

*Equivalent amounts of ClO_4^- was applied in the K, Na, and Ca salts at the comparative rates. The high, medium, and low rates contained 0.72, 0.36 and 0.18 pounds of ClO_4^- respectively.

TABLE 3

Perchlorate Content of Grapefruit Leaves from New and Old Flush and of the Leaf Tips and Base.

	ClO_4^- Mgm./gm. (Dry Wt.)
New Flush	1.28
Old Flush	2.56
Leaf Tips (Yellow)	3.71
Leaf Base (Green)	0.69

TABLE 4

Perchlorate Content of Nitrate of Potash and Nitrate of Soda from Natural Sources.

Sample	Fertilizer	ClO_4^-* percent
1	KNO_3	0.58
2	KNO_3	0.51
3	KNO_3	0.66
4	$NaNO_3$	0.06

*The ClO_4 content actually includes all of the halogenates. However, field tests indicate that practically all of the halogenates found are perchlorates.

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