

# TRACE METAL UPTAKE/AVAILABILITY FROM THREE MUNICIPAL COMPOSTS

K. R. Baldwin and J. E. Shelton, North Carolina State University

## INTRODUCTION

The potential uses of composts as fertilizers and soil amendments have propelled many studies aimed at evaluating responses of crops to these products. One of the greatest concerns about using compost for agricultural purposes is the bioavailability of contaminants contained in the material.

Researchers have worried about Cd in sludges and compost and the food chain risk associated with land application of these materials since the 1970's (Chaney and Ryan, 1993). Excessive dietary Cd can accumulate over one's lifetime in the kidney cortex and cause renal tubular dysfunction (Fanconi syndrome), a disease in which low molecular weight proteins are excreted in urine. Bioavailability of Cd contained in composts to tobacco plants (*Nicotiana tabacum* L.) is a particular concern because tobacco is able to absorb Cd with no major signs of toxicity (Tancogne et al. 1988). Schroeder and Balassa (1961) conducted a survey of sources of Cd intake by humans and found tobacco contained the highest Cd concentration of all products tested ( $1.65 \text{ mg kg}^{-1}$  or  $31.2 \text{ } \mu\text{g pack}^{-1}$ ).

Recently, Jing and Logan (1992) reported on the phytoavailability of Cd from different sludges where equal amounts of Cd were applied in each pot. Crop uptake of Cd increased with increasing sludge Cd concentration. This was explained in terms of the filling of specific Cd binding sites in the sludge: The population of Cd binding sites varies widely in strength of specific Cd adsorption, and as sludge Cd concentration increases, the least strongly bound Cd is more phytoavailable. Evidence indicated plant uptake of Cd from sludge with low Cd content was less than that from sludges with higher Cd content, even if the same total amount of Cd was applied. These findings supported the Corey et. al (1987) hypothesis that plant trace metal uptake is controlled in part by sludge chemistry. Corey et al. (1987) concluded that specific metal adsorption on sludge surfaces would normally be the controlling factor in metal phytoavailability in soil-sludge mixtures. Citing data from many studies, they concluded that sludges with higher metal concentration cause higher metal uptake by plants when equal amount of metals are applied (i.e. different amounts of sludge dry matter and hence adsorption capacity were applied).

The solubility of trace metals increases with decreasing pH. Sanders and Adams (1987) found that as pH was decreased for each sludge and metal, a threshold pH was reached below which metal solubility was sharply increased. Adams and Sanders (1984) found that the higher the sludge metal concentration, the higher the threshold pH point of increasing metal solubility.

## OBJECTIVES

In western North Carolina, burley tobacco is often grown on steep slopes where soil erosion can be a significant problem. Twenty years ago, additions of organic matter, primarily manures, to these soils was a regular management practice which not only provided nutrients to crops but helped to reduce erosion as well. Incorporation of organic matter improved infiltration and drainage potential which reduced runoff. Since that time, chemical fertilizers have replaced manures as principal nutrient sources for burley crops, organic matter levels in soils have decreased, and erosion has increased. The increasing interest in the beneficial reuse and potential availability of composted organic waste materials presents an opportunity for burley tobacco farmers to begin again to regularly apply organic matter to burley fields. There remain concerns, however, about the availability of trace metals contained in composts to burley tobacco because tobacco is known to be a metal accumulator. The objective of this experiment was to determine the plant availability of compost trace metals to a burley tobacco crop.

## METHODS AND MATERIALS

### SOIL

The soil chosen for the field study was a Dyke clay soil (clayey mixed mesic Typic Rhodudults) located on the North Carolina Department of Agriculture Mountain Research Station in Waynesville, North Carolina. An analysis of the soil is presented in Table 1.

## COMPOSTS

Three composts were evaluated in this study: 1) co-composted municipal solid waste and wastewater biosolids compost (COC); 2) municipal solid waste compost (MSWC); and 3) wastewater biosolids compost (WBC). A chemical analysis of the composts is presented in Table 2.

The COC was produced by an aerobic, in-vessel process by Bedminster<sup>®</sup> Bioconversion Corporation in Sevier County, TN. The feedstocks for the material were municipal solid waste and wastewater biosolids; and they were digested for three days in a Eweson<sup>®</sup> digester, a compartmentalized rotary cylinder, and then deposited in windrows on an aerated curing floor. The windrows were turned regularly, and composting continued on the computer-controlled aeration floor for four to six weeks to meet EPA PFRP requirements (Environmental Protection Agency Processes to Further Reduce Pathogens). After composting the compost was cured on a storage floor for over one month.

The MSWC was produced in a pilot project co-sponsored by the Buncombe County, NC, Department of Solid Waste and the North Carolina Cooperative Extension Service. The feedstock was municipal solid waste (MSW) which was minimally presorted to remove undesirable inorganic materials, ground, and placed in windrows. The windrows were turned weekly to provide adequate aeration, and moisture was added when needed. Three month old MSWC was used in the field experiment.

The WBC was produced by the Charlotte Mecklenburg Utility District. Centrifuged, dewatered (18-20% TS) sewage sludge was mixed with wood chips and straw in a ratio of 1:5:1, placed in aerated static piles for a period of 28 days, and then cured for 30 days in an unsheltered area.

Table 1. Experimental Dyke soil chemical characteristics (mg kg<sup>-1</sup>) in May, 1994 before addition of composts.

pH	N	Mn	Cu	Zn	Cd	Cr	Ni	Pb
5.8	1600	870	26	64	<1	81	19	13

Table 2. Elemental composition of composts, COC, MSWC and WBC, applied to a Dyke soil in 1994 at 0, 25, 50 and 100 Mg ha<sup>-1</sup>.

	C	Al	Ca	Fe	K	Mg	N	P	S
COC <sup>ad</sup>	36.41	1.79	2.77	1.42	0.38	0.31	1.31	0.26	0.70
MSWC <sup>bd</sup>	6.41	3.60	0.94	3.40	0.27	0.26	0.30	0.08	0.07
WBC <sup>cd</sup>	35.89	1.37	1.72	1.58	0.35	0.22	2.12	0.96	0.43
	Cd	Cl	Cr	Cu	Mn	Na	Ni	Pb	Zn
COC <sup>e</sup>	2.9	5117	58.0	215.0	370.3	6126	39.7	203.0	737.7
MSWC <sup>e</sup>	1.0	339.3	23.7	52.7	602.7	448.7	18.0	34.0	96.3
WBC <sup>e</sup>	2.1	356.3	140.3	173.3	880.7	325.0	16.3	88.3	499.0

<sup>a</sup> Co-Composted municipal solid waste and wastewater biosolids (Sevier Co., TN)

<sup>b</sup> Municipal Solid Waste Compost (Buncombe Co., NC)

<sup>c</sup> Wastewater Biosolids Compost (Charlotte, NC)

<sup>d</sup> % dry weight basis

<sup>e</sup> mg kg<sup>-1</sup> dry weight basis

## BURLEY TOBACCO CULTURE

1994: A tall fescue/clover crop was plowed and disked in the spring, P was incorporated at 56 kg ha<sup>-1</sup>, and the field was harrowed. Twenty-two plots were arranged in randomized complete blocks with four blocks. Plots measured 3.7 by 10.4 meters. Treatments consisted of: 1) MSWC, COC and WBC applied at 25, 50 and 100 Mg ha<sup>-1</sup> (dw) to N-fertilized plots (224 kg ha<sup>-1</sup> N) without lime (9 plots); 2) MSWC, COC and WBC applied at 25, 50 and 100 Mg ha<sup>-1</sup> to N-fertilized plots (224 kg ha<sup>-1</sup> N) limed to bring the pH to 6.5 (9 plots); 3) N-fertilized

(224 kg ha<sup>-1</sup> N) control plots without lime and limed to pH 6.5 (2 plots); and 4) Unfertilized (0 kg ha<sup>-1</sup> N) control plots without lime and limed to pH 6.5 (2 plots). Lime was applied to amended plots at 4000 kg ha<sup>-1</sup>.

Compost was distributed as uniformly as possible to plots by hand and the field was then disked. Insufficient MSWC was available for 100 Mg ha<sup>-1</sup> applications to limed and unlimed plots in two replications and data from these plots has been treated as missing data. Burley tobacco (*Nicotiana rustica* L. var TN 90) was mechanically transplanted in three rows per plot with 1.23 meters between rows and 22 plants per row. Missing and/or dead plants were reset within 7 days. Conventional crop management strategies were employed throughout the growing season (NC Cooperative Extension Service, 1994), and B was foliarly applied to burley plants in June. The crop was hand-harvested in September, hung on tobacco sticks, and air-cured in barns at the research station.

1995: Fertilizer and lime treatments were reapplied to respective plots, but compost was not applied. Burley tobacco (TN 90) was planted on May 30th and managed conventionally. Serious blue mold disease pressure required additional sprays of fungicides in 1995. After harvesting the burley leaf was hung on sticks in barns on the research station to cure.

## ANALYSES

Soil: A stainless steel soil probe (2.54 cm diameter) was used to collect a composite sample of forty cores from the research field prior to application of compost, N-fertilizer and lime. This sample was air dried and then ground in a soil grinder to pass a 2 mm sieve. After thorough mixing, a 200 gram subsample was obtained for use in laboratory analyses. Total N was determined by Kjeldahl digestion (Bremner, 1965a), and total Mn, Cr, Cd, Cu, Ni, Pb, and Zn by aqua regia digestion (McGrath and Cunliffe, 1985), pH by 1:1 soil/water volume ratio and CEC by NH<sub>4</sub>OAc at pH 7 (Soil Conservation Service, USDA, 1972).

Soil samples were collected from harvest rows in May, July, and September of 1994 and June, July, and September of 1995. A stainless steel soil probe was used to collect a composite sample of four cores (20 cm long by 2.54 cm in diameter) from each plot. Samples were air-dried, ground to pass a 2 mm sieve and stored at room temperature in soil cartons. Samples were extracted with DTPA at pH 7.3 (Lindsay and Norvell, 1978). Extracts were analyzed for Zn, Ni, Cu, Cd and Pb on a Perkin-Elmer Plasma 2000 System inductively coupled plasma emission spectrometer (ICP).

Compost: Approximately 5 kg of each compost was collected at the time of field application and stored for three weeks at 5° C. Approximately 0.5 kg of each sample was thoroughly mixed, and a 0.1 kg subsample was dried overnight at 70° C and then ground in a stainless steel Wiley mill to pass a 2 mm sieve.

A 2.5 g compost subsample (replicated three times) was ashed in a muffle furnace at 500° C overnight. Two ml of distilled H<sub>2</sub>O and 4 ml 6N HCl was added and the subsample heated on a steam bath. The subsample was brought to volume in a 50 ml volumetric with distilled water and analyzed for elemental content (except N) with a Perkin-Elmer Plasma 2000 inductively coupled plasma emission spectrometer. Compost N concentration was determined with a Perkin-Elmer PE 2400 CHN Elemental Analyzer.

Tobacco: Tobacco leaves were collected from growing plants in June, July, August and September of 1994. Ten most-recently-matured leaves were stripped from "guard rows" adjacent to the center "harvest row" in each 3-row plot. Leaves stripped in June were rinsed with distilled water before drying; leaves stripped in other months were dried without rinsing. Cured leaf samples (composited by weight from all stalk positions) were obtained by subsampling from cured hands at grading. All leaves were dried at 70° C, and then ground in a stainless steel Wiley mill to pass a 1 mm sieve. Ground samples were stored at room temperature in acid-washed glass jars.

Ten leaves were stripped from guard rows in June, July and August of 1995. In June, most-recently-matured leaves were rinsed in distilled water before drying at 70° C. In July and August, 10 plant leaves were stripped from two stalk positions: most-recently matured leaves from the upper part of the plant and mature leaves from the lower part of the plant, near the stalk position where a leaf had been collected in June.. These leaves were not rinsed before drying at 70° C. Cured leaf samples from upper, middle, and lower stalk positions were subsampled from cured hands at grading. Dried samples were ground in a stainless steel Wiley mill to pass a 1 mm sieve and stored in acid-washed glass jars at room temperature while awaiting laboratory analysis. Dried plant samples were analyzed as per 1994.

Statistics: Comparisons between means were made using a general linear models procedure in the Statistical Analysis System (SAS, 1985).

## RESULTS AND DISCUSSION

### Copper

*Leaf Cu:* Variation in trace metal concentration within each pH category was large throughout the experiment. Consequently, the analysis of variance for each compost treatment was conducted using the General Linear Models (GLM) procedure with pH as a continuous rather than a discrete variable (Ray, 1982).

Table 3. Mean tissue Zn and Cu concentration ( $\text{mg kg}^{-1}$ ) of burley leaf samples in September of 1994, June and August of 1995 and cured leaves in both years from three compost treatments, COC, MSWC and WBC applied at 0, 25, 50 and 100  $\text{Mg ha}^{-1}$ . Within columns, means followed by the same letter are not significantly different at  $p=0.05$ .

Compost	Rate	1994 Zn			1995 Zn			
		Fresh <sup>4</sup>	Cured	June	Fresh	Cured		
		Sept.	Composite <sup>5</sup>		Sept.	Lower <sup>6</sup>	Middle <sup>6</sup>	Upper <sup>6</sup>
COC <sup>1</sup>	0	28.1a	44.0a	58.4a	36.4a	38.2a	40.7a	42.9a
	25	33.7b	50.1b	63.1ab	38.3a	41.2ab	46.8b	45.5ab
	50	35.9b	54.6b	71.0b	39.5a	46.1bc	50.5bc	49.4b
	100	37.5b	78.6c	71.1b	41.3b	48.8c	53.1c	55.8c
MSWC <sup>2</sup>	0	29.7ab	38.9a	58.8a	36.4a	38.9a	41.1a	42.5a
	25	27.9a	40.1a	64.7ab	38.7a	40.4a	41.5a	43.9a
	50	29.5ab	45.2a	66.7b	38.8a	41.1a	47.0a	46.0a
	100	35.4b	61.8b	68.0ab	35.6a	50.0a	49.3a	50.2a
WBC <sup>3</sup>	0	29.8a	38.9a	59.8a	37.8a	40.0a	41.9a	43.7a
	25	29.9a	46.1a	66.3a	41.5a	47.0ab	54.6b	52.7bc
	50	29.4a	45.3a	67.7a	37.8a	42.7a	44.9ac	46.4ab
	100	33.4a	49.8b	69.4a	41.7a	57.7b	52.3bc	53.9c

Compost	Rate	1994 Cu			1995 Cu			
		Fresh	Cured	June	Fresh	Cured		
		Sept.	Composite		Sept.	Lower	Middle	Upper
COC	0	8.0a	8.8a	16.9a	13.9a	10.2a	11.3a	14.9a
	25	12.0b	12.1b	19.0ac	15.3a	13.7ab	14.3b	14.4a
	50	12.5b	15.8b	21.8b	15.5a	15.2b	14.6b	15.6a
	100	13.5b	28.7c	20.6bc	16.4b	17.2b	16.5b	24.1b
MSWC	0	8.8ab	7.3ab	17.7a	14.0a	10.9a	11.7a	15.1ab
	25	8.1a	5.1b	18.4a	14.6a	12.2a	11.2a	12.9a
	50	9.7b	8.0a	19.9a	14.3a	13.0a	14.0a	15.6ab
	100	9.1ab	7.8ab	19.9a	15.9a	17.9b	21.9b	20.2b
WBC	0	8.6a	6.9a	18.0a	14.1a	11.9a	11.5a	15.0ab
	25	8.2a	7.0a	17.7a	14.7a	12.5a	15.9b	18.3a
	50	8.3a	7.3ab	17.2a	13.7a	12.4a	11.0a	13.4b
	100	9.4a	10.3b	16.7a	14.2a	21.3a	12.6a	16.2ab

<sup>1</sup> Co-Composted municipal solid waste and wastewater biosolids (Sevier Co., TN)

<sup>2</sup> Municipal Solid Waste Compost (Buncombe Co., NC)

<sup>3</sup> Wastewater Biosolids Compost (Charlotte, NC)

<sup>4</sup> most-recently-matured leaves

<sup>5</sup> weighted composite sample

<sup>6</sup> stalk position of leaves sampled

Miner and Tucker (1990) reported a North Carolina nutrient sufficiency range for Cu in the uppermost fully developed tobacco leaf prior to flowering of 5 to 10  $\text{mg kg}^{-1}$ . Collins et al. (1961) reported that flue-cured tobacco contained 14.9 to 21.1  $\text{mg kg}^{-1}$  Cu. Robson and Reuter (1981) found that for the majority of crop plant

species. Cu causes toxicity when the foliar Cu content is 20 to 30 mg kg<sup>-1</sup>. Mean leaf Cu concentrations at various sampling dates for differing rates of COC, MSWC and WBC application are shown in Table 3.

There was no effect of pH on leaf Cu concentration in COC and WBC treatments in September of 1994 (Figure 1: rates are pooled for WBC), nor in MSWC treatments (data not shown). Indeed, 1994 late season leaf Cu concentration in 25 and 100 Mg ha<sup>-1</sup> COC treatments increased with increasing pH (rate X pH interaction). There is usually a negative correlation between Cu uptake and soil pH (Tiwari and Kumar, 1982).

There was no pH effect on 1994 cured leaf Cu concentration in any compost treatment. Cured leaves in COC and WBC treatments in 1994 (Figures 2 and 3) showed a rate effect, while MSWC treatments (data not shown) did not. Leaf Cu concentration in the 100 Mg ha<sup>-1</sup> rate COC treatment was in the toxicity threshold range reported by Robson and Reuter (1981). High leaf Cu concentration at high pH: 1) supported the theory that with high rates of addition of metals, the pH threshold for diminishing availability occurs at higher soil pH (Adams and Sanders, 1984); 2) suggested the Jing and Logan (1990) conclusion that "the population of Cd binding sites in sludge varies widely in strength of specific Cd adsorption, and as sludge Cd concentration increases, the least strongly bound Cd is more phytoavailable" can be applied to compost Cu as well; and 3) supported the hypothesis of Corey et al (1987) that sludge chemistry (in this case compost chemistry) controls the activity of Cu in the soil solution.

Leaf Cu concentration in MSWC and WBC treatments was not related to pH and leaf Cu concentration was relatively low in both cases at low pH. In these cases, adsorptive capacity of compost may control Cu availability rather than low soil pH.

Early leaf Cu concentration for all composts in 1995 showed an interaction of rate X pH. The only rates in all three composts which were inversely related to pH were 0 Mg ha<sup>-1</sup> rates for COC and MSWC and 0 and 25 Mg ha<sup>-1</sup> rates for WBC (data not shown).

Figure 4 shows late season upper leaf Cu concentration in COC and WBC (rates pooled) treatments. In COC treatments, upper leaf Cu concentration did not decrease with increasing pH at any rate. Upper leaf Cu in WBC and MSWC treatments was inversely related to pH but not related to rate (data for MSWC not shown). Upper leaves in all compost treatments had a higher Cu concentration than lower leaves (data not shown).

Late season lower leaf Cu concentration showed a rate X pH interaction in COC treatments (data not shown) in which neither 50 nor 100 Mg ha<sup>-1</sup> application rates showed an inverse relationship to pH, but 0 and 25 Mg ha<sup>-1</sup> rates did. Late season lower leaf Cu concentration in MSWC and WBC treatments in 1995 was not related to pH or compost rate (data not shown). Higher expected Cu concentration at lower pH was probably not observed in lower leaves because of translocation of Cu to upper leaves.

Cured leaf Cu concentration in 1995 in all compost treatments and at all leaf positions (data not shown) was not related to pH. Lower, middle and upper leaf Cu concentration was related linearly to rate in COC treatments but not in WBC treatments. Lower and middle leaf Cu concentration was linearly related to rate in MSWC treatments but not upper leaf Cu. Lower and middle cured leaves in COC treatments were significantly lower in Cu concentration than upper leaves. Upper leaves had a higher Cu concentration than lower leaves in MSWC treatments, but WBC treatments did not vary in Cu concentration by leaf position.

*Soil Cu:* Mean DTPA-extractable soil Zn, Ni, Cu, Cd and Pb concentration at various sampling dates for differing rates of COC, MSWC and WBC application is shown in Table 4. Generally, there was no inverse relationship observed between DTPA-extractable metal concentration and pH in September, 1994, except for Ni in MSWC and WBC treatments. No effect of pH was observed on DTPA-extractable Zn, Cu, Cd, Ni or Pb concentration in September, 1995. The response of DTPA-extractable metal concentration to rate of compost addition was generally linear throughout the experiment. In contrast to Valdares et al. (1983) and King and Hajjar (1990), DTPA-extractable trace metal concentration was not well-correlated with amount of trace metal applied.

Lindsay and Norvell (1978) reported a critical DTPA-extractable Cu value of 0.2 ppm. Follett and Lindsay (1970) reported that DTPA-extractable Cu ranged from 0.14 to 3.18 mg kg<sup>-1</sup> (while total soil Cu ranged from 2 to 92 mg kg<sup>-1</sup>). Walsh et al. (1972) reported significant reductions in snapbean yield at 20 mg kg<sup>-1</sup> DTPA-extractable Cu concentration. DTPA-extractable Cu concentration for compost treatments ranged from 0.44 to 9.33 µg g<sup>-1</sup>.

DTPA-extractable soil Cu concentration in September, 1994, was linearly related to pH in COC-treated soil (Figure 5), contradicting reports in the literature (Locascio, 1978; Cavallaro and McBride, 1980). DTPA-extractable Cu concentration was linearly related to rate and unrelated to pH in WBC treatments (Figure 6) and not related to rate or pH in MSWC treatments (data not shown). The increasing extractability of Cu with increasing

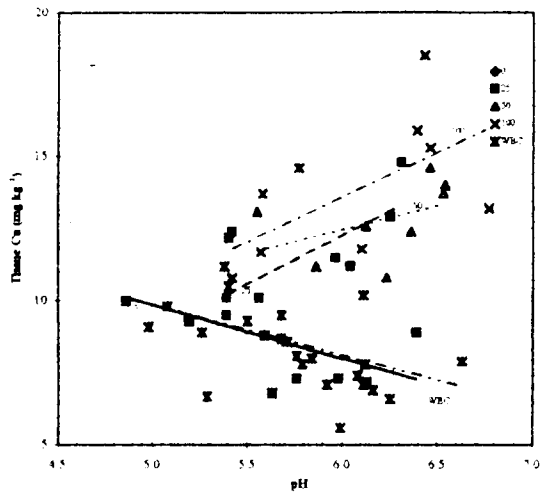


Figure 1. Effect of pH and rate of application ( $\text{Mg ha}^{-1}$ ) on 1994 late season leaf Cu concentration of burley tobacco grown on COC- and WBC-amended soil. Rates pooled for WBC.

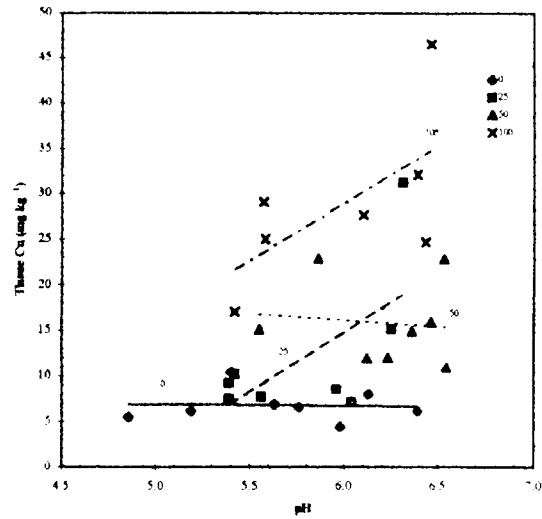


Figure 2. Effect of pH and rate of application ( $\text{Mg ha}^{-1}$ ) on 1994 cured leaf Cu concentration of burley tobacco grown on COC-amended soil.

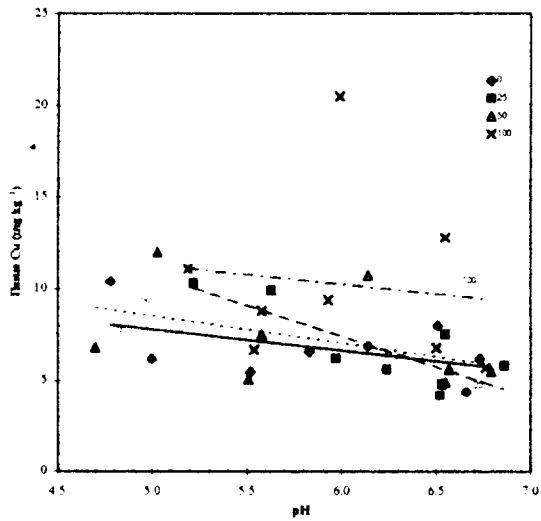


Figure 3. Effect of pH and rate of application ( $\text{Mg ha}^{-1}$ ) on 1994 cured leaf Cu concentration of burley tobacco grown on WBC-amended soil.

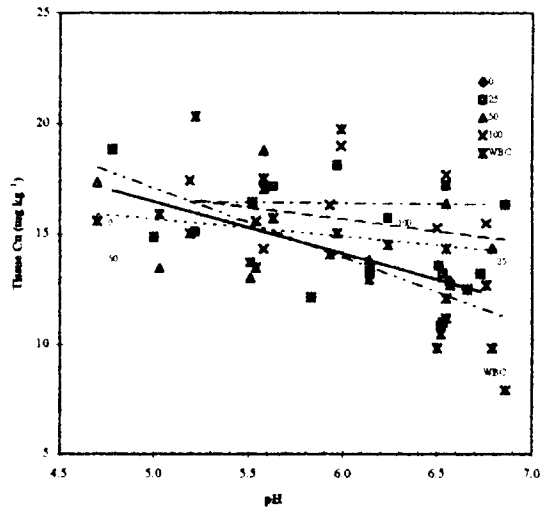


Figure 4. Effect of pH and rate of application ( $\text{Mg ha}^{-1}$ ) on 1995 late season upper leaf Cu concentration of burley tobacco grown on COC- and WBC-amended soil.

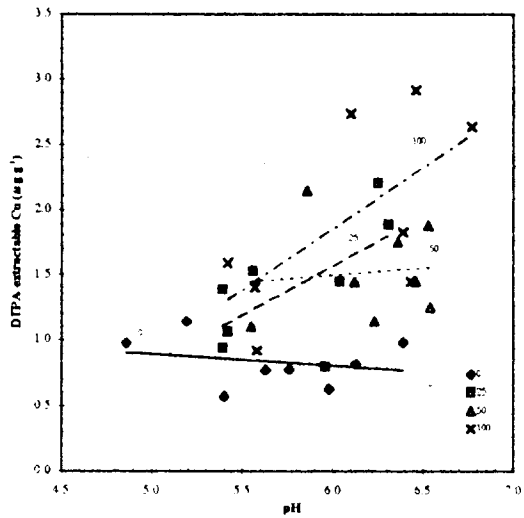


Figure 5. Effect of pH and rate of COC application ( $\text{Mg ha}^{-1}$ ) on 1994 late season DTPA-extractable soil Cu concentration.

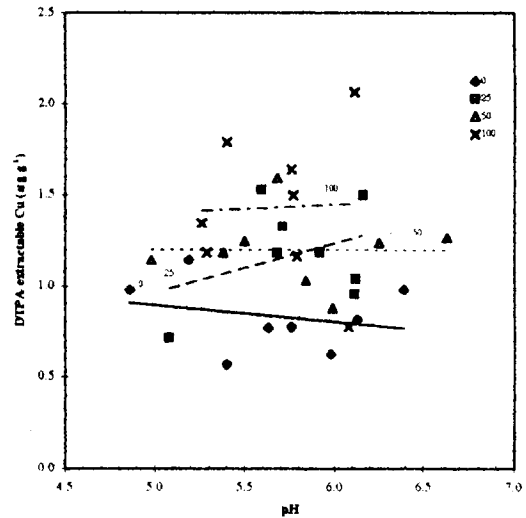


Figure 6. Effect of pH and rate of WBC application ( $\text{Mg ha}^{-1}$ ) on 1994 late season DTPA-extractable soil Cu concentration.

pH could have contributed to differences in leaf Cu concentration between COC and WBC treatments in 1994. DTPA-extractable Cu concentration was unrelated to pH in all compost treatments in 1995.

Table 4. Mean DTPA-extractable soil Zn, Ni, Cu, Cd and Pb concentration ( $\mu\text{g g}^{-1}$ ) determined in September of 1994 and 1995 for three compost treatments, COC, MSWC and WBC, applied at 0, 25, 50 and 100  $\text{Mg ha}^{-1}$ . Within columns, data followed by the same letter are not significantly different at  $p=0.05$ .

Compost	Rate	1994					1995				
		Zn	Ni	Cu	Cd	Pb	Zn	Ni	Cu	Cd	Pb
COC <sup>1</sup>	0	1.58a	.24a	0.95a	.03a	1.18a	1.50a	.34ab	0.91a	.04a	1.33a
	25	2.97a	.34a	1.60b	.05ab	1.72a	2.14a	.33a	1.18ab	.04a	1.72b
	50	3.32a	.30a	1.32ab	.06bc	1.70a	3.85b	.41ab	1.55bc	.06b	2.44c
	100	5.62b	.33a	1.71b	.07c	2.54b	4.23b	.44b	1.71c	.06b	2.58c
MSWC <sup>2</sup>	0	1.15a	.23a	0.80a	.03a	1.08a	1.62a	.33a	0.95a	.04a	1.35a
	25	1.76b	.31a	1.09a	.05b	1.45b	1.91ab	.39a	1.03a	.04a	1.56a
	50	1.67b	.28a	1.00a	.04ab	1.29ab	1.98ab	.43a	1.02a	.03a	2.13a
	100	1.50ab	.37a	0.97a	.03ab	1.38ab	3.50b	.71a	2.01b	.09b	1.59a
WBC <sup>3</sup>	0	1.24a	.26a	0.84a	.03a	1.11a	1.76a	.35a	0.98a	.04a	1.51a
	25	2.44b	.33a	1.15ab	.05b	1.47b	2.86a	.44ab	1.26ab	.04ab	2.69a
	50	2.65bc	.35a	1.19b	.05b	1.31ab	2.70a	.44ab	1.22a	.04ab	1.81a
	100	3.61c	.33a	1.45b	.06b	1.40b	4.97b	.52b	1.77b	.06b	1.89a

<sup>1</sup> Co-Composted municipal solid waste and wastewater biosolids (Sevier Co., TN)

<sup>2</sup> Municipal Solid Waste Compost (Buncombe Co., NC)

<sup>3</sup> Wastewater Biosolids Compost (Charlotte, NC)

**Cu Comparisons Among Composts:** Because individual composts varied in trace metal content, differing amounts of trace metals were applied at each compost application rate. Neither leaf Cu, nor DTPA-extractable soil Cu concentration, therefore, could be directly contrasted among composts by rate of compost application. When concentration was regressed against rate of metal application for each compost, plotted regression lines did not overlap and a comparison of composts remained inappropriate.

Log transformation of Cu, Zn, Pb and Ni application rates and corresponding tissue or soil concentrations of these metals allowed a direct comparison of COC and WBC treatments because log transformed regression lines overlapped. Log transformation of Cd and Ni application rates and corresponding tissue or soil concentrations of these metals allowed a direct comparison of all three composts.

Comparisons of leaf Cu concentration were made with log-transformed data. At equal rates of Cu application, leaf Cu concentration was greater in "higher Cu" COC ( $215 \text{ mg kg}^{-1} \text{ Cu}$ ) treatments than in "lower Cu" WBC ( $173.3 \text{ mg kg}^{-1} \text{ Cu}$ ) treatments in late-season and cured leaves in 1994 and in June and August upper leaves in 1995. (data shown for 1994 cured leaves in Figure 7 and June, 1995, upper leaves in Figure 8). This suggested the Corey et al. (1987) conclusion that "sludges with higher metal concentration could cause higher metal uptake by plants when equal amount of metals were applied" could also be applied to composts.

The significant interaction of rate X pH for September 1994 COC leaf Cu concentration shown in Figure 1 may also help to explain why leaf Cu concentration in COC and WBC treatments differed significantly at that time. Leaf Cu concentration was unrelated to pH and rate in WBC treatments, whereas in 25 and 100  $\text{Mg ha}^{-1}$  COC treatments it was linearly related to pH.

The difference in 1994 cured leaf Cu concentration may be explained by examining Figures 2 and 3. Mean cured leaf Cu concentration (Table 3) in 100  $\text{Mg ha}^{-1}$  COC treatments ( $28.7 \text{ mg kg}^{-1}$ ) was over twice that of 100  $\text{Mg ha}^{-1}$  WBC treatments ( $10.3 \text{ mg kg}^{-1}$ ). This difference supports the concept that in higher metal composts, trace metals can occupy low energy binding sites and be more available for plant uptake.

Differences in 1995 late season Cu concentration (Figure 4) may have occurred because leaf Cu concentration was unrelated to pH in COC treatments and inversely related to pH in WBC treatments.

Comparisons of DTPA-extractable soil Cu were made with log-transformed data and showed significant differences in September, 1994, DTPA-extractable Cu concentration between COC and WBC treatments. Once again, at equal rates of metal application the compost with the higher metal concentration had more "plant

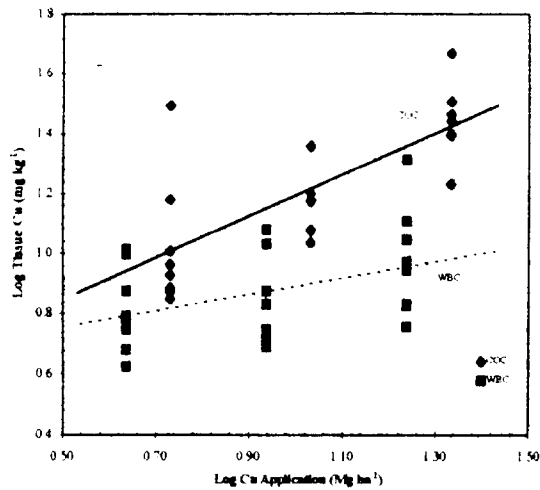


Figure 7. Effect of rate of application ( $\text{kg ha}^{-1}$ ) of COC and WBC compost Cu on 1994 cured leaf Cu content of burley tobacco

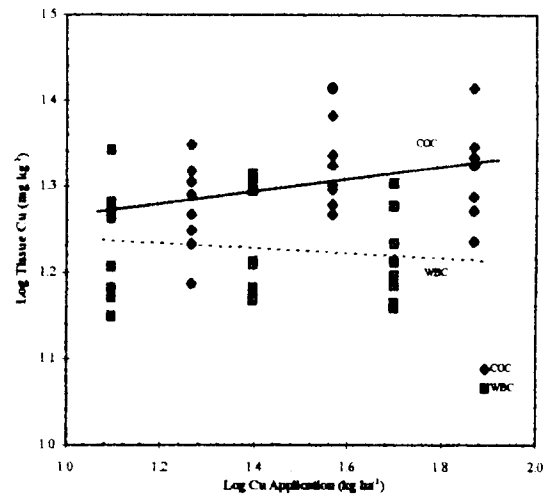


Figure 8. Effect of rate of application ( $\text{kg ha}^{-1}$ ) of COC and WBC compost Cu on 1995 early season leaf Cu content of burley tobacco

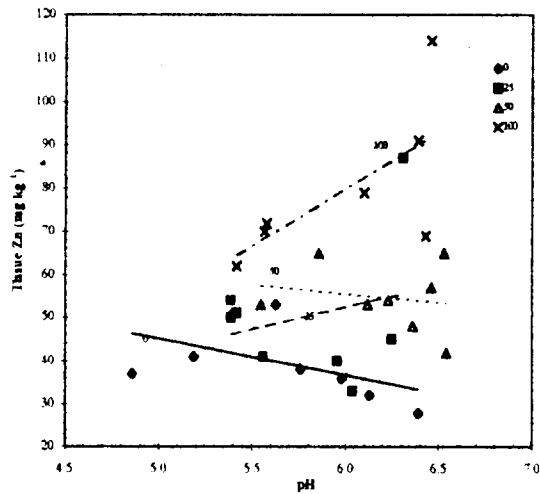


Figure 9. Effect of pH and rate of application ( $\text{Mg ha}^{-1}$ ) on 1994 cured leaf Zn concentration of burley tobacco grown on COC-amended soil.

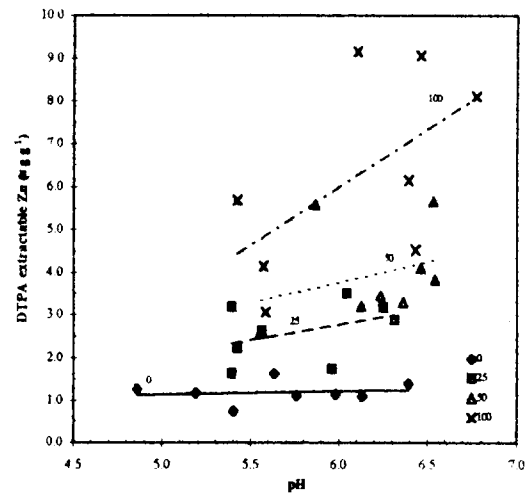


Figure 10. Effect of pH and rate of application ( $\text{Mg ha}^{-1}$ ) on 1994 late season DTPA-extractable soil Zn concentration.

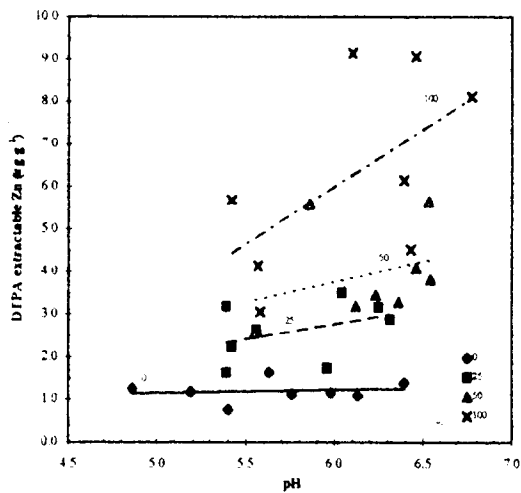


Figure 11. Effect of pH and rate of COC application ( $\text{Mg ha}^{-1}$ ) on 1994 late season DTPA-extractable soil Zn concentration.

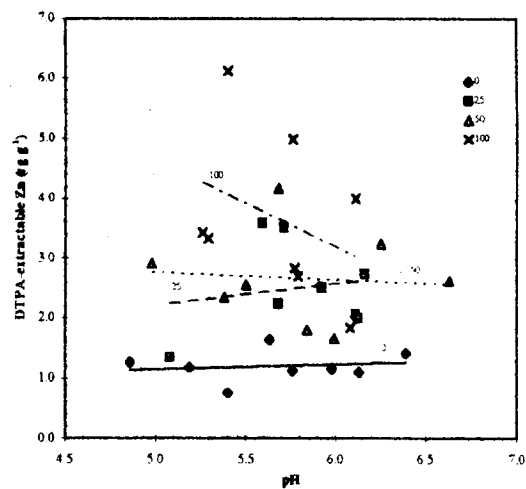


Figure 12. Effect of pH and rate of WBC application ( $\text{Mg ha}^{-1}$ ) on 1994 late season DTPA-extractable soil Zn concentration.

available" Cu than a lower metal compost. Extractable Cu concentration increased with increasing pH and rate in COC treatments and was unrelated to pH in WBC treatments (Figures 5 and 6).

## Zinc

*Leaf Zn:* Normal Zn concentrations in plant tissue ranges from 25 to 150 mg kg<sup>-1</sup> (Adriano, 1986) and mean Zn content in various commercial tobaccos is 51 to 84 mg kg<sup>-1</sup> (Ward, 1941). Miner and Tucker (1990) reported a North Carolina nutrient sufficiency range for Zn in the uppermost fully developed tobacco leaves prior to flowering of 20-60 mg kg<sup>-1</sup>. Mean leaf Zn concentrations at various sampling dates for differing rates of COC, MSWC and WBC application are shown in Table 3. Burley leaf Zn concentration at all sampling dates fell within the reported ranges.

A significant rate X pH interaction for 1994 cured leaf Zn concentration in COC treatments is shown in Figure 9. At the 100 Mg ha<sup>-1</sup> rate, Zn concentration was linearly related to pH and at the other rates of addition of COC, leaf Zn concentration was unrelated to pH. Leaf Zn concentration was unrelated to pH in MSWC (data not shown) and WBC (Figure 10) treatments in 1994 cured leaf samples. The highest rate of addition of Zn in the experiment (100 Mg ha<sup>-1</sup> COC) resulted in increasing leaf Zn concentration as pH increased and supported the Adams and Sanders (1984) finding that higher metal concentrations (in sludges) resulted in increased metal solubility and hence uptake at higher pH values.

Late 1995 leaf Zn concentration was inversely related to pH in lower and upper leaves in COC treatments (data not shown). Upper leaves demonstrated a rate X pH interaction. Late 1995 leaf Zn concentration in MSWC and WBC treatments was unrelated to rate but was inversely related to pH in upper leaves in MSWC and WBC treatments and in lower leaves in WBC treatments (data not shown). There were no significant differences in Zn concentration between upper and lower leaves in any compost treatment at that sampling date.

Cured upper, middle and lower leaf Zn concentration in COC treatments in 1995 was inversely related to pH and linearly related to rate. A significant interaction (rate X rate X pH) was observed in middle leaves, with all rates inversely related to pH. Upper leaves had significantly higher Zn concentration than lower leaves (data not shown). This contradicted data presented by King and Hajjar (1990) and Frank et al. (1977). Upper, middle and lower cured leaf Zn concentration in MSWC treatments was inversely related to pH, and no interactions were significant (data not shown). Upper cured leaves had significantly higher Zn concentration than lower leaves (data not shown). Cured upper, middle and lower leaf Zn concentration in WBC treatments was inversely related to pH and linearly related to rate but there were no significant differences in leaf Zn concentration among leaf positions at that time (data not shown).

*Soil Zn:* In COC treatments, DTPA-extractable soil Zn concentration showed a rate X pH interaction in September of 1994 (Figure 11). Zinc concentration was linearly related to pH at the 100 Mg ha<sup>-1</sup> rate and was not related to pH at other rates, contradicting many reports, including those of MacLean (1974), Friesen et al. (1980) and Gupta et al. (1971). DTPA-extractable Zn concentration was unrelated to pH but linearly related to rate in both WBC (Figure 12) and MSWC treatments (data not shown). All three composts showed a linear response to rate in 1995, but no response to pH.

*Zn Comparisons Among Composts:* Comparisons of composts were made with log-transformed data. There were significant differences in leaf Zn concentration between COC (737 mg kg<sup>-1</sup> Zn) and WBC (499 mg kg<sup>-1</sup> Zn) treatments in both late and cured leaves in 1994. Differences in 1994 late season leaf Zn concentration are shown in Figure 13 (data for 1994 cured leaf concentration not shown). At equal rates of Zn loading, leaf Zn concentration in "higher Zn" COC treatments was significantly greater than leaf Zn in "lower Zn" WBC treatments. This suggested the Corey et al. (1987) conclusion that "sludges with higher metal concentration could cause higher metal uptake by plants when equal amount of metals were applied" could be applied to composts. Leaf Zn concentration was inversely related to pH in WBC treatments while in COC treatments it was not, and that contributed to the difference in 1994 late season leaf Zn concentration between the two compost treatments. The difference in cured leaf Zn concentration may be partly explained by the interactions of pH and rate in COC treatments in which both leaf Zn and DTPA-extractable soil Zn concentration increased with increasing pH at the 100 Mg ha<sup>-1</sup> rate. In both cases, compost chemistry rather than soil chemistry seemed to be controlling plant Zn uptake. However, there was no difference in DTPA-extractable soil Zn concentration between COC and WBC treatments.

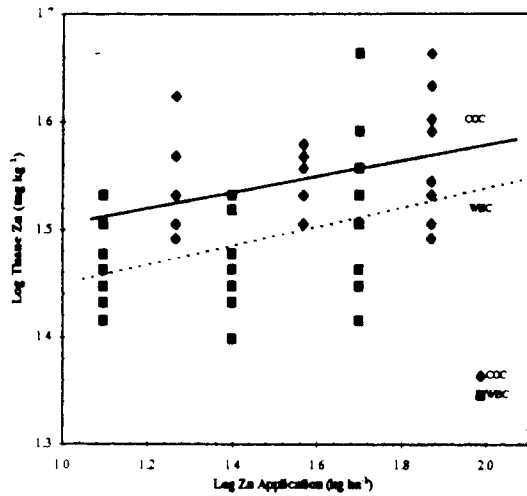


Figure 13 Effect of rate of application ( $\text{kg ha}^{-1}$ ) of COC and MSWC compost Zn on 1994 late season leaf Zn content of barley tobacco.

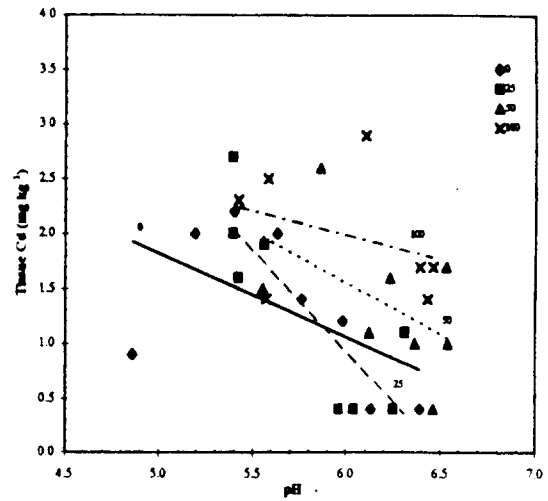


Figure 14 Effect of pH and rate of application ( $\text{Mg ha}^{-1}$ ) on cured leaf Cd concentration of barley tobacco grown on COC-amended soil.

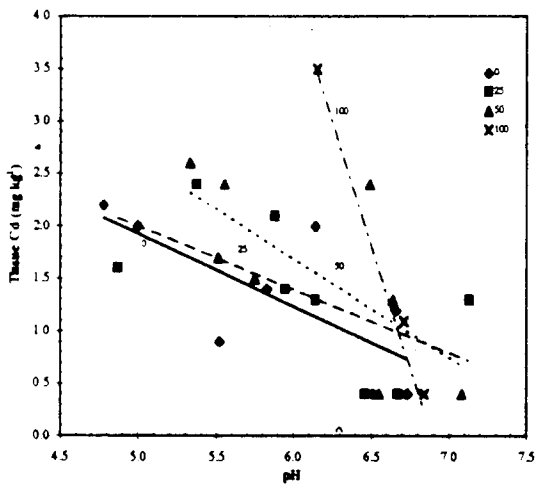


Figure 15 Effect of pH and rate of application ( $\text{Mg ha}^{-1}$ ) on cured leaf Cd concentration of barley tobacco grown on MSWC-amended soil.

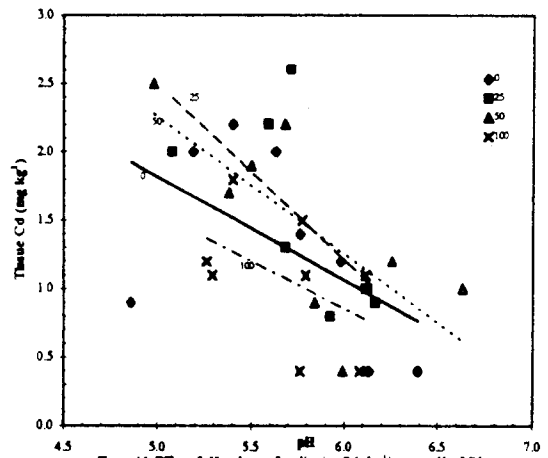


Figure 16 Effect of pH and rate of application ( $\text{Mg ha}^{-1}$ ) on cured leaf Cd concentration of barley tobacco grown on WBC-amended soil.

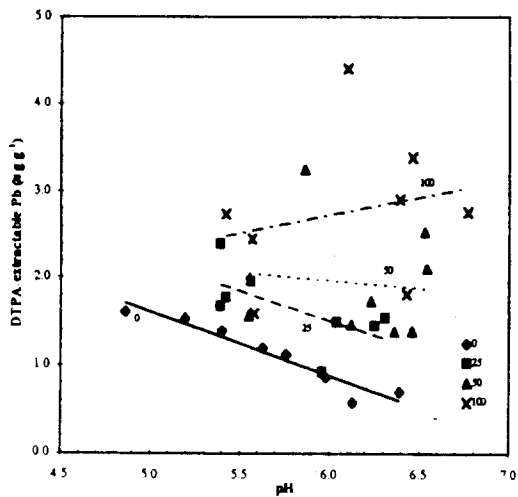


Figure 17 Effect of pH and rate of COC application ( $\text{Mg ha}^{-1}$ ) on 1994 late season DTPA-extractable soil Pb concentration.

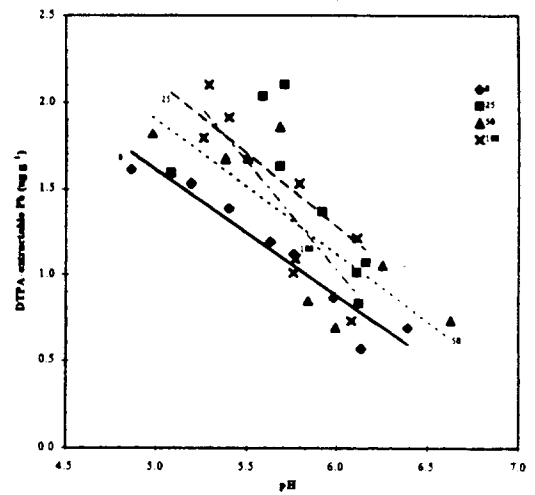


Figure 18 Effect of pH and rate of WBC application ( $\text{Mg ha}^{-1}$ ) on 1994 late season DTPA-extractable soil Pb concentration.

## Cadmium

With the exception of the composite samples taken from 1994 cured burley leaves, there was little detectable Cd in burley tissue at any sampling date. Only 69 out of 87 1994 cured leaf samples contained detectable Cd. For the purposes of statistical analysis, the 18 samples with Cd content below detectable limits were assigned values of  $0.4 \text{ mg kg}^{-1}$  (50% of the detection limit). Fewer than 15 samples at any other sampling date contained detectable Cd. Cured leaf Cd concentration was inversely related to pH in COC MSWC and WBC treatments in 1994 (Figures 14, 15 and 16, respectively).

In contradiction to reports in the literature (Christensen, 1984a; Kuo et al., 1985) extractable Cd concentration in COC treatments increased significantly as pH increased in September, 1994 (data not shown). Available Zn Cu Ca and Mn in solution may have reduced Cd sorption through competition for adsorption sites (Kuo and Baker, 1980; Bittell and Miller, 1974). Considerable Zn Cu and Ca was added to the soil in COC applications and the Dyke soil contained  $500 \text{ mg kg}^{-1}$  Mn (Tables 1 and 2). DTPA-extractable Cd concentration was low in all treatments throughout the experiment (Table 4).

When data was log transformed, there were significant differences in September, 1994, DTPA-extractable Cd concentration among compost treatments (data not shown). At equal rates of Cd application, DTPA-extractable Cd concentration was significantly higher in "higher Cd" COC ( $2.9 \text{ mg kg}^{-1}$ ) and WBC ( $2.1 \text{ mg kg}^{-1}$ ) treatments than in "lower Cd" MSWC ( $1.0 \text{ mg kg}^{-1}$ ) treatments.

## Lead

No Pb was detectable in burley leaf tissue at any sampling date in either 1994 or 1995.

Cox and Rains (1972) and John and Van Laerhoven (1972b) reported that application of lime to Pb-contaminated soils reduced the foliar Pb content of plants but had little effect on Pb in roots. DTPA-extractable Pb concentration in September, 1994, soil samples taken from COC treatments showed a rate X pH interaction in which extractable Pb concentration did not decrease with increasing pH (Figure 17). At the  $100 \text{ Mg ha}^{-1}$  rate, DTPA-extractable Pb concentration in COC treatments increased with increasing pH. Extractable Pb concentration in WBC treatments was inversely related to pH but not related to rate (Figure 18). Extractable Pb concentration was not related to either pH or to rate in MSWC treatments (data not shown).

Log-transformed September, 1994, data showed that at equal rates of Pb application, the "higher Pb" COC ( $203 \text{ mg kg}^{-1}$ ) treatments had higher extractable soil Pb concentration than "lower Pb" WBC ( $88 \text{ mg kg}^{-1}$ ) treatments.

## Nickel

No Ni was detectable in burley leaf tissue at any sampling date in either 1994 or 1995.

DTPA-extractable Ni concentration was low in all compost treatments (Table 4). In contrast to reports in the literature (Harter, 1983; Gerritse et al. 1982), Ni extractability was unrelated to pH in COC treatments. Rate X pH interactions were observed in MSWC treatments for DTPA-extractable Ni concentration, and extractability was inversely related to pH. In WBC treatments, DTPA-extractable Ni concentration was inversely related to pH and unrelated to rate (data not shown).

When September, 1995, data was log transformed (data not shown), "lower Ni" WBC ( $16.3 \text{ mg kg}^{-1}$ ) treatments had higher DTPA-extractable Ni concentration at equal rates of Ni application than "higher Ni" COC ( $39.7 \text{ mg kg}^{-1}$ ) treatments.

## CONCLUSIONS

Trace metals applied in COC, MSWC and WBC did not accumulate in leaf tissue in sufficient quantities to produce toxicity symptoms in burley tobacco. Burley leaf Pb and Ni concentration was below detectable limits and Cd concentration, only detectable in cured 1994 leaf samples, was low. Burley leaf Zn and Cu concentration was linearly related to rate of compost addition, but mean leaf Zn concentration fell within the sufficiency range reported by Miner and Tucker (1990) in all compost treatments. Leaf Cu was above the sufficiency range reported by Miner and Tucker (1990) in most treatments, but within the normal range for flue-cured tobacco reported by Collins et. al (1961). Cured 1994 leaf Cu in  $100 \text{ Mg ha}^{-1}$  COC treatments was within the toxicity range reported by Robson and Reuter (1981). However, no Cu toxicity symptoms were observed.

Taken together, the evidence provided by this experiment indicated that at the same rate of metal application, Zn and Cu in composts are more available from composts containing higher concentrations of these

metals than from lower metal composts. Leaf Cu concentration in 1994 and 1995 and leaf Zn concentration in 1994 was generally not related to pH, and the extractability of trace metals generally did not vary with pH (in some cases increased with increasing pH), even at high rates of addition of compost. These findings indicated that compost chemistry influenced if not controlled the availability of trace metals to burley tobacco.

## REFERENCES

- Adams, T.M. and J.R. Sanders. 1984. The effects of pH on release to solution of zinc, copper and nickel from metal-loaded sewage sludges. *Environ Pollut.* B8:85-99.
- Adriano, D.C. 1986. *Trace Elements in the Terrestrial Environment*. Springer-Verlag, New York.
- Adriano, D.C., A.L. Page, A.A. Elseewi and A.C. Chang. 1982. *Journal of Environmental Quality*. 11:197-203.
- Bittel, J.E., and R.J. Miller. 1974. *Journal of Environmental Quality*. 3:250-253.
- Bremner, J.M. 1965a. Inorganic forms of nitrogen. In C.A. Black et al. (ed.) *Methods of soil analysis*. Part 2. *Agronomy* 9:1179-1237. Am. Soc. Of Agron., Madison, WI.
- Cavallaro, N., and M.B. McBride. 1980. *Soil Science Society of America Journal*. 44:729-732.
- Chaney, R.L. and J.A. Ryan. 1993. Heavy metals and toxic organic pollutants in MSW composts. In *Science and Engineering of Composting: Design, Environmental, Microbiological and Utilization Aspects*. Harry A.J. Hoitink and Harold M. Keener, eds. pp. 451-489. Renaissance Publications, Worthington, OH. 728 pages.
- Christensen, T.H. 1984a. Cadmium soil sorption at low concentrations: I. Effect of time, cadmium load, pH, and calcium. *Water Air Soil Pollut.* 21:105-114.
- Collins, W.K., G.L. Jones, J.A. Weybrew and D.F. Matzinger. 1961. Comparative chemical and physical composition of flue-cured tobacco varieties. *Crop Science*. 1:407.
- Corey, R.B., L.D. King, C. Lue-Hing, D.S. Fanning, J.J. Street and J.M. Walker. 1987. Effects of sludge properties on accumulation of trace elements by crops. In: *Land Application of Sludge*. A.L. Page et al. Eds. pp25-51. Lewis Publishers, Chelsea, MI.
- Cox, W.J., and D.W. Rains. 1972. *Journal of Environmental Quality*. 1:167-169.
- Follett, R.H., and W.L. Lindsay. 1970. In: *Profile Distribution of Zinc, Iron, Manganese, and Copper in Colorado Soils*. Colorado Agricultural Experiment Station Technical Bulletin. 110:1-78.
- Frank, F., H.E. Braun, M. Holdrinet, and K.I. Stonefield. 1977. Metal contents and insecticide residues in tobacco soils and cured tobacco leaves collected in southern Ontario. *Tobacco Science*. 21:74-80.
- Friesen, K.K., A.S.R. Juo, and M.H. Miller, 1980. *Soil Science Society of America Journal*. 44:1221-1226.
- Gerritse, R.G., R. Vriesema, J.W. Dalenberg, and H.P. de Roos. 1982. *Journal of Environmental Quality*. 11:359-364.
- Gupta, S.K., F.W. Calder, and I.B. MacLeod. 1971. *Plant and Soil*. 35:249-256.
- Harter, R.D. 1983. *Soil Science Society of America Journal*. 47:47-51.
- Jing, J. and T.J. Logan. 1992. Effects of sewage sludge cadmium concentration on chemical extractability and plant uptake. *J. Environ. Qual.* 21:73-81.
- John, M.K., and C.J. Van Laerhoven. 1972b. *Journal of Environmental Quality*. 1:169-171.
- King, L.D. and I.M. Hajjar. 1990. The residual effect of sewage sludge on heavy metal content of tobacco and peanut. *Journal of Environmental Quality*. 19:738-748.
- Kuo, S., E.J. Jellum, and A.S. Baker. 1985. Effects of soil type, liming, and sludge application on zinc and cadmium availability to swiss chard. *Soil Science*. 139:122-130.
- Kuo, S., and A.S. Baker. 1980. *Soil Science Society of America Journal*. 44:969-974.
- Lindsay, W.L., and W.A. Norvell. 1978. Development of a DTPA soil test for zinc, iron, manganese, and copper. *Soil Science Society of America Journal*. 42:421-428.
- Locascio, S.J. 1978. *Solutions*. 30-42.
- MacLean, A.J. 1974. *Canadian Journal of Soil Science*. 54:369-378.
- McGrath, S.P. and C.H. Cunliffe. 1985. A simplified method for the extraction of the metals Fe, Zn, Cu, Ni, Cd, Pb, Cr, Co and Mn from soils and sewage sludges. *Journal of Science, Food and Agriculture*. 36:794-798.
- Miner, G., and R. Tucker. 1990. Plant analysis as an aid in fertilizing tobacco. In: *Soil Testing and Plant Analysis*. R.L. Westerman, ed. Pp. 645-657. Soil Science Society of America, Inc. Madison, WI.
- North Carolina Cooperative Extension Service. 1994. *1995 Burley Tobacco Information*. AG 376. North Carolina State University, Raleigh, NC.
- Ray, A.A. 1982. SAS user's guide: Statistics. SAS Inst., Cary, NC.
- Robson, A.D. and D.J. Reuter. 1981. Diagnosis of copper deficiency and toxicity. In *Copper in Soils and Plants*; J.F. Loneeragan, A.D. Robson and R.D. Graham Eds. pp. 287-312. Academic Press, London.
- Sanders, J.R. and T.M. Adams. 1987. The effects of pH and soil type on concentration of zinc, copper and nickel extracted by calcium chloride from sewage sludge-treated soils. *Environ. Pollut.* A43:219-228.
- SAS. 1985. SAS User's Guide: Statistics. SAS Institute, Cary, NC.
- Schroeder, H.A., and J.J. Balassa. 1961. Abnormal trace metals in man: Cadmium. *Journal of Chronic Disorders*. 14:236-258.
- Smith, S.R. 1992. Sewage sludge and refuse composts as peat alternatives for conditioning impoverished soils: Effects on the growth response and mineral status of *Petunia grandiflora*. *Journal of Horticultural Science*. 67(5): 703-716.
- Soil Conservation Service, USDA. 1972. Soil survey laboratory methods and procedures for collecting soil samples. Soil Survey Invest. Rep. 1. U.S. Gov. Print. Office, Washington, DC.
- Tancogne, J., Nguyen Phu Lich, P. Schiltz, R. Truhaut, J.D. Claude, and J. Chouteau. 1988. Influence of various growth medium related factors on the absorption of cadmium. *CORESTA Information Bull.* 1988 Congress. Oct. 9-13. China.
- Tiwari, R.C., and B.M. Kumar. 1982. *Plant and Soil*. 68:131-134.
- Valdres, J.M.A.S., M. Gal, U. Mingelgrin, and A.L. Page. 1983. *Journal of Environmental Quality*. 12:49-57.
- Ward, G.M. 1941. Mineral absorption studies with tobacco. *The Lighter*. 11(1):16-22.