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Application of High-Cu Compost to Dill and Peppermint

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A controlled environment experiment was conducted to determine the effect of amending soil with various rates of high-Cu compost (0, 20, 40, and 60% compost/soil by volume) on dill (*Anethum graveolens* L.) and peppermint (*Mentha* X *piperita* L.) yields, on fractionation of Cu and Zn in soils, on elemental composition of soil and tissue, and on the essential oils. The compost contained about 2000 mg kg^{-1} of Cu. Dill yields were greatest in the 20 or 40% treatments, but peppermint yields were greatest in the 20% treatment. Compost additions increased soil pH and electrical conductivity (EC), HNO_3 extractable soil B, Ca, K, Mg, Mn, P, S, Na, and Pb. Additions of high-Cu compost to soil increased tissue P, S, and Na in both crops and Mn, Mo, and Zn in dill but decreased tissue Ca, Cd, and Fe in both crops and Mn, Mo, and Zn in peppermint, increased Cu in all soil fractions including exchangeable, and increased tissue Cu of dill and peppermint as compared to unamended soil. Addition of 60% of high-Cu compost to soil resulted in 760−780 mg kg^{-1} Cu in the growth medium. Nevertheless, Cu content in both crops reached only 12 mg kg^{-1} DW in the 60% compost treatment, which is below the toxicity levels for plants and below the upper chronic dietary exposure for animals. The application of high-Cu compost altered chemical composition of dill and peppermint essential oils, but oils were free of Cu, Zn, Cd, Ni, Cr, and Pb. Results from this study suggest that mature composts with concentrations of Cu and Zn of 2008 and 321 mg/kg, respectively, can be used as a soil conditioner without risk for phytotoxicity or risk of increasing the normal range of Cu and Zn in crop tissue. However, the long-term effect of the accumulation of heavy metals in soils following repeated compost applications needs to be carefully considered.

KEYWORDS: Dill; peppermint; compost; copper; zinc; sequential extraction; nutrients; trace elements; essential oils

INTRODUCTION

Compost application to agricultural land has many benefits; it improves the soil's biological, chemical, and physical properties, and it provides nutrients to the soil (*1*). In addition, it may also protect soils from erosion, suppress certain plant diseases, and keep organic wastes out of landfills (*1*, *2*). However, elevated concentrations of trace elements in composts could be a concern (*1*−*3*). Most countries have regulatory limits for maximum trace element concentrations in composts (*1*, *4*−*6*). Copper is one of the elements that causes a concern because of its relatively high content in composts (*7*) and its potential toxicity to plants (*8*). The Canadian guidelines for maximum trace element concentrations in composts allow 100 mg Cu kg^{-1} in type AA and A compost and 757 mg of total Cu kg^{-1} in type B composts (*5*). Type AA and A composts have unrestricted agricultural use. However, the 100 mg Cu kg^{-1} (total Cu concentration) in type AA and A composts is difficult to meet (*7*, *9*, *10*), preventing the use of good quality compost as a soil amendment for field- or for container-grown crops (*11*, *12*). Research has indicated that it is difficult to predict the level of Cu accumulation in crops grown in soils after the application of organic amendments (*9*). The addition of some types of composts may even reduce Cu uptake by plants (*13*, *14*). Further research is needed to estimate the safe levels for Cu and other elements of concern in various composts.

The aims of this study were as follows: (i) to evaluate the application of various amounts of high-Cu compost on growth, productivity, and the tissue content of essential and trace elements of dill and peppermint; (ii) to estimate the effect of high-Cu compost application on metal transfer into the oils and on essential oil quality of dill and peppermint; and (iii) to fractionate Cu and Zn in the compost and in the growth medium and relate their concentrations to Cu and Zn accumulation in dill and peppermint.

To meet these objectives, dill and peppermint plants were grown in pots with high-Cu and soil mixtures of either 0, 20, 40, or 60% compost/soil by volume. Matured backyard wastes/manure compost and a Pugwash sandy loam soil were used. Dill and peppermint were chosen as model plants based on previous research indicating very little or no transfer of heavy

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metals into essential oils of crops grown on heavy metal-polluted soils (*9*, *15*−*17*). Both crops are grown for essential oil production or for dry leaves and herba, which are the final marketable products. Dill and peppermint essential oils are important aromatic agents widely used as flavoring and fragrance agents in an array of products, and they have proven antioxidant and antimicrobial properties (*18*, *19*). After the extraction of the essential oil, peppermint and dill residues are used as an excellent animal feed, mostly for sheep (*20*).

MATERIAL AND METHODS

Plant and Growth Conditions. The container experiment was conducted in an Econair Plant Growth Room GR-192 at the Nova Scotia Agricultural College, NS, by using dill (*Anethum graveolens* L. cv. Mesten) and peppermint (*Mentha* X *piperita* L. cv. Black Mitchum). Dill seeds were purchased from Johnny's Selected Seeds, Albion, ME, while peppermint planting material was kindly provided by Dr. Lyle Craker, University of Massachusetts, Amherst, MA. Plants were grown in 20 cm diameter plastic containers in four replicates. Dill plants were started as seeds by placing 20 seeds in each container. After emergence, the number of plants was reduced to 10 per container, and after a week, the number was reduced further to six plants per container. Peppermint plants were started from rhizomes cut in 2−3 cm pieces to provide 1−2 plantlets per rhizome piece. After emergence, plants were reduced to three per container. Dill and peppermint plants were grown for 12 weeks in a 14 h day and 10 h night regime, with day temperatures of 25 °C and night temperatures of 18 °C. Irrigation of dill and peppermint was conducted once every 24 h by using an automatic watering drip emitter system. To prevent leaching of Cu and other nutrients and trace elements out of containers, plastic trays were placed under each container. Dill and peppermint plants were fertilized every week with 300 mL of fertilizer solution (60 g 20:20:20 $N-P_2O_5-K_2O$ fertilizer dissolved in 10 L of water). Harvesting of both plants was done at the same time, at the beginning of seed formation of dill, and at 50% blossoming of peppermint when the essential oil quality was highest (*23*, *30*).

The compost treatments comprised of 0 (100% soil), 20, 40, and 60% high-Cu compost to soil by volume. The above application rates by volume translate into 0, 125, 250, and 375 g air-dry compost per pot. Depending on the amount of compost, each container weighed between 2150 and 2650 g. A Pugwash sandy loam (Humo-Ferric Podzol) (water, pH 6.0, 3.1 mg^{-1} C g) was used in this study. Results from this study would be relevant to both greenhouse (where growth medium constituents are added by volume) and to field production systems (where compost is added by weight). The high-Cu compost was prepared from 100 kg of feedstock including yard waste, grass clippings, straw, and manure in 1999, and it was left to mature for 6 months with turning every 15 days. To produce unnaturally high-Cu compost, copper(II) nitrate [$Cu(NO_3)_2 \cdot 3H_2O$] dissolved in water was added to the feedstocks at the beginning of composting and mixed thoroughly. At maturity, the compost had a moisture content of 66%: 7.7% C and 0.628% N, C:N ratio 12.3:1, H_2O pH 7.6, and 2.4 mmhos/cm (dS m^{-1}) electrical conductivity (EC) on a wet weight basis. Nutrient and trace elements concentrations in the compost were as follows (in mg kg^{-1} dry weight): Cu, 2008; Ca, 47 000; P, 8750; Na, 2370; K, 15 028; Mg, 9878; Fe, 5975; Mn, 927; Zn, 321; S, 2871; B, 34; Cr, 33; Ni, 29; Pb, 32; and Mo, 5. The National Standard of Canada for compost quality (*5*) specifies that a compost could be considered mature if it meets two of the following three requirements: (i) C/N ration ≤ 25, (ii) oxygen uptake rate shall be less than or equal to 150 mg kg^{-1} of oxygen of volatile solids/h, and (iii) germination of cress (*Lepidium sativum* L.) seeds and radish (*Raphanus sativus* L.) seeds in compost must be greater than 90% of the germination rate of the control sample, and the growth rate of the control sample and the growth rate of plants grown in a mixture of compost and soil must not differ by more than 50%. The high-Cu compost used in this experiment met two of the above requirements, and it was left to mature for 6 months after the thermophilic stage.

A seed germination phytotoxicity test was used to evaluate compost maturity by using cress and marigold (*Calendula officinalis* L.) seeds and a direct seed test (*22*). Two controls (peat and water) were used for seed germination. In each Petri dish, 15 cm^3 of compost or peat was added, and deionized water was added to moisten the material to saturation. Ten seeds were placed in every Petri dish, and the lids were left on until 3 days after the germination. The germination tests were replicated five times.

Sequential Extraction. To fractionate Cu and Zn in the growth medium, a sequential extraction was performed using the BCR method (*23*, *24*), which is recommended by the Community Bureau of Reference (BCR) and the International Organization for Standardization (*25*). The BCR sequential extraction procedure has been extensively used outside Europe as well (*26*−*28*). The BCR method is a three step procedure. The BCR sequential extraction method was developed to recover the following chemical forms of metals in each of the steps: (i) water soluble, exchangeable (EXCH), and weekly bound to organic matter (OM) denoted as EXCH; (ii) occluded in Fe or Mn oxides (FeMnOx); (iii) organically bound and sulfides (OM); and (iv) structurally bound (SB) (residual fraction recovered decomposition of the residue remaining from step iii with aqua regia). Also, the residue remaining after step iii was decomposed in aqua regia to determine the amount of residual Cu. In addition to the four steps, soil samples were decomposed in aqua regia to calculate the percentage of Cu and Zn recovery of the sequential extraction steps i−iv relative to the aqua regia extractable Cu and Zn.

Each of the extraction steps was conducted in 250 mL polypropylene centrifuge tubes with screw caps, unless indicated otherwise, using 2.5 g of soil. The extraction was conducted by shaking in a mechanical, end-over-end shaker at a speed of 30 rpm and room temperature of 22 ± 2 °C. The chemical fractions were operationally defined as follows.

(i) Exchangeable fraction of metals (EXCH): a 100 mL amount of 0.11 mol L^{-1} acetic acid was added to the samples (2.5 g). The samples were shaken for 16 h at room temperature of 22 ± 2 °C (overnight) and centrifuged at 1500*g* for 20 min. The supernatant was filtered through No. 41 Whatman filter paper and stored in a refrigerator at 4 °C for analysis. The residue in each tube was washed by adding 50 mL of deionized water, shaking for 15 min on the end-over-end shaker, and centrifuging for 20 min at 1500*g*. The supernatant was decanted and discarded, taking care not to discard any of the solid residues. The extract was preserved by adding 1 mL of concentrated HNO_3.

(ii) Metals bound by reducible iron and manganese oxides (FeMnOx): a 100 mL amount of 0.5 mol L^{-1} hydroxylammonium chloride (hydroxylamine hydrochloride) was added to sample residues from step i. The samples were resuspended by manual shaking and then extracted by mechanical shaking for 16 h at a room temperature of 22 ± 2 °C (overnight) and centrifuged at 1500*g* for 20 min. The supernatant was filtered through No. 41 Whatman filter paper and stored in a refrigerator at 4 °C for analysis. Then, the samples were washed with deionized water.

(iii) Metals bound to organic matter and sulfides (OM): to the residue from step ii, 25 mL of 8.8 mol L^{-1} hydrogen peroxide was added in small aliquots and carefully to avoid losses due to violent reaction. The vessels were loosely covered with the caps, and the samples were decomposed at a room temperature of 22 ± 2 °C for 1 h with occasional manual shaking. Decomposition was continued for 1 h at 85 ± 5 °C in a water bath. The volume was further reduced to 3 mL by further heating of uncovered vessels. Further aliquot of 25 mL was added to each sample. Covered vessels were heated again at 85 ± 5 °C in a water bath and decomposed for 1 h. After that, the vessels were uncovered, and the volume was reduced to about 1 mL. A 100 mL amount of 1.0 mol L^{-1} ammonium acetate was added to the cool wet residues, and the samples were shaken for 16 h at 22 ± 2 °C (overnight). The extract was separated from the solid residue by centrifugation and decantation as in step i and further stored in a refrigerator for analysis.

(iv) Structurally bound (SB) or residual fraction recovered with aqua regia: the soil samples from step iii were transferred to 250 mL Pyrex decomposition tubes. The predecomposition step was performed at room temperature (at 22 ± 2 °C) for 16 h (overnight) in 28 mL of 37% (concentrated) HCl:70% (concentrated) HNO_3 mixture per tube. Each suspension was decomposed at 130 °C for 2 h using a reflux condenser. Samples were filtered through Whatman No. 41 filter and diluted to 50 mL with 0.5 mol L^{-1} HNO_3 and stored in a refrigerator for analyses.

Table 1. Cress and Marigold Seed Germination and Height for Evaluation of Compost Maturity by Using the Direct Seed Test

	control H_2O		control peat		high-Cu compost	
species	germ (%)	height (cm)	germ (%)	height (cm)	germ (%)	height (cm)
Cress	75a[a]	1.4c	75a	4.2a	68a	2.9b
Marigold	100a	1.3b	93a	3.1a	80b	1.6b

[a] Means with the same letter within a crop and index are not significantly different at $P \leq 0.05$.

(v) Aqua regia extraction of soil samples: soil samples (3 g) from all treatments were decomposed in aqua regia using the same decomposition procedure as in step iv to estimate the total amount of Cu and Zn recovered in the sequential extraction.

All samples from the sequential extraction plus the aqua regia decomposed soils samples were analyzed for Cu and Zn on a Varian Spectra AA-20 flame atomic absorption spectrophotometer (FAAS). Separate standards were prepared for each batch of samples in the appropriate matrix, to account for possible interferences.

Trace Element Analyses. Dry soil, compost, and tissue samples were decomposed with concentrated HNO_3 (*7*). Immediately after the harvest, plant subsamples (above ground shoots) from all of the treatments were oven-dried at 60 °C for 72 h, then ground, and sieved through a 2.0 mm screen. Soil and compost samples from all the pots were taken after the harvest, dried at 60 °C for 72 h, then ground, and sieved through a 2.0 mm screen. Plant, compost, and soil samples were decomposed in concentrated nitric acid by using 4 g of soil or tissue as described previously (*17*).

Essential Oil Analyses. Dill and peppermint plants were dried in a shaded well-aerated place at 22 °C to preserve the essential oil. Steam distillation was used to extract the essential oil of dill and peppermint from air-dried herbage. Distillation was carried out on 400 g of dried plant material (leaves, flowers, and stems) in 2 L round-bottom flasks for 3 h in a Clevenger type apparatus purchased from Quickfit, England (*21*, *29*). The extracted peppermint and dill oils were measured by volume and stored at −80 °C until further quality analyses. The dill and peppermint were diluted in hexane (0.05 mL of oil into 0.95 mL of GC standard grade hexane) the day the samples were analyzed on a Varian 6500 gas chromatograph (GC) as described elsewhere (*17*).

All of the data sets were analyzed using two way analysis of variance with SAS (*30*) at $\alpha = 0.05$; where the interactions or main effects were found significant, mean separation of data was performed. Constant variance and normality of residuals were tested, and some transformations were necessary for the normal distribution of residuals.

RESULTS AND DISCUSSION

The seed germination phytotoxicity test (**Table 1**) suggests that marigold seed germination was more sensitive to the high-Cu content of compost as compared to the seed germination of cress. The high-Cu compost had also reduced the height of both cress and marigold (measured 10 days after germination), as compared to the peat control. Results suggest that initial plant growth (plant height 10 days after seed germination) may be a more sensitive index to the high-Cu content of compost as compared to seed germination.

Soil pH and EC measured at the end of the experiment (**Table 2**) indicated that soil pH and EC were increased with increasing rates of compost application. Application of high rates of mature plant waste/manure compost to the mostly acidic Nova Scotia soils would be beneficial, since it may reduce the need for lime application.

Table 2. Soil pH, EC, Plant Yields, and Height from Dill and Peppermint as a Function of the Rate of High-Cu Compost Application to the Soil

		pH	EC ($dS\ m^{-1}$)	yields (DW g/pot)	height (cm)
species	treatment	$n = 4$	$n = 4$	$n = 4$	$n = 24$
dill	0% compost	5.34c[a]	3.9	77.6b	63b
	20% compost	5.42bc	4.5	133.0a	86a
	40% compost	5.45b	4.6	141.3a	77ab
	60% compost	5.73a	5.8	91.8b	72b
peppermint	0% compost	5.36c	3.8	70.1bc	65bc
	20% compost	5.36c	4.5	107.5a	83a
	40% compost	5.52ab	4.8	85.8ab	70b
	60% compost	6.05a	5.0	49.6c	57c

[a] Means with the same letter within a species are not significantly different at $P \leq 0.05$.

Dill yields increased in the 20 and 40% compost treatments, but peppermint yields increased in the 20% treatment relative to the 0% treatment (unamended soil) (**Table 2**). Greater yields of both crops in the 20% compost and for dill in the 40% treatment may have been due to a number of factors such as improved soil physical and biological properties, increased amounts of nutrients, and increased pH fostering better availability of some nutrients. In a similar experiment, we found that yields of basil and swiss chard were increased by some levels of compost even when compost was quite immature and had extremely high-Cu content (*17*). The yield-suppressing effect of compost in the 60% treatment relative to the 20 and 40% compost treatments (**Table 4**) may be due to Cu phytotoxicity (*8*). In the previous study, we also found that high rates of high-Cu compost reduced basil but not swiss chard yields (*17*).

No reports in the literature were found on the effect of high-Cu concentrations on dill; however, there were reports about peppermint (*15*, *16*, *31*). Yields of both crops in the 60% treatment were not different from yields in the unamended soil, although in the 60% treatment, Cu in the growth medium reached 760−780 mg kg^{-1}. In the experiment of other authors (*31*), tissue Cu in the highest sewage sludge treatment reached 44 mg kg^{-1}, but in the 60% treatment of the present study, tissue Cu of both peppermint and dill was about 12 mg kg^{-1}. These results suggest a lower phytoavailability of Cu in compost-amended soils than in sewage sludge-amended soil. Other authors (*15*), in field experiments with peppermint grown in highly polluted soils, reported very similar values for tissue Cu and Zn to Cu and Zn in peppermint reported by the previous authors (*31*). In our experiment, tissue Cu in the 60% treatment reached 12.2 vs 9.3 mg kg^{-1} in the 40% treatment where no adverse affect was found. Our data indicate an increase in menthol and menthyl acetate content in the peppermint oil in the 60% treatment relative to the 40% treatment, which may be evidence for a physiological reaction to Cu phytotoxicity. Perhaps, the critical tissue Cu concentration in peppermint might be around 10 mg kg^{-1}, which is one-half the value of the respective critical concentrations reported for most other crops (*8*, *32*). Further research is needed to establish the critical tissue Cu concentration for peppermint and dill with respect to yield reduction.

Compost addition to the soil increased HNO_3 extractable B, Ca, K, Mg, P, S, Na, and Pb in soil; it had no effect on HNO_3 extractable Cr, Hg, and Ni, and it decreased HNO_3 extractable As, Cd, Co, Fe, Mo, and Se (**Table 3**) relative to the unamended soil. The addition of compost increased tissue P, S, Na, and Mo (in dill); increased K in some treatments; had no effect on tissue B, Co, Hg, Ni, Pb, and Se; decreased tissue Ca, Cd, and Fe in both crops; and decreased Mg and Mo in some treatments. Tissues As and Cr in all treatments were below the detection

Table 3. Mineral Composition of Soils and Plant Tissue and Plant Uptake of Macro- and Microelements as a Function of the Rate of Compost Application and Plant Species

treatments element		dill				peppermint			
		0%	20%	40%	60%	0%	20%	40%	60%
Ca[b]	soil concn	0.2d[a]	5.1c	9.7b	11.7a	0.2d	4.2c	10.5b	16.1a
	plant concn	11.9a	8.7b	8.7b	7.2c	7.7a	5.9b	4.7c	4.6c
	plant uptake	0.9	1.2	1.2	0.7	0.5	0.6	0.4	0.2
K	soil concn	1.0c	1.4bc	1.9b	4.2a	1.2c	1.5c	2.2b	3.7a
	plant concn	32.8b	32.9b	38.2a	39.4a	30.7b	24.1c	24.1c	35.8a
	plant uptake	2.5	4.4	5.4	3.6	2.2	2.6	2.1	1.8
Mg	soil concn	1.2d	1.9c	2.6b	3.6a	1.2c	1.9bc	2.8b	3.5a
	plant concn	2.4a	1.9b	2.1b	2.1ab	2.3a	1.7b	1.6b	1.8b
	plant uptake	0.2	0.3	0.3	0.2	0.2	0.2	0.1	0.1
P	soil concn	0.4d	1.3c	2.2b	4.0a	4.5d	1.1c	2.1b	3.6a
	plant concn	3.8b	4.7a	3.7b	4.6a	3.2c	3.4bc	3.8b	4.5a
	plant uptake	0.3	0.6	0.5	0.4	0.2	0.4	0.3	0.2
S	soil concn	0.2d	0.3c	0.5b	0.8a	0.2d	0.3c	0.8b	0.8a
	plant concn	0.8c	1.4b	1.8a	1.8a	0.86b	1.02b	1.1b	1.6a
	plant uptake	0.2	0.6	0.8	0.5	0.2	0.3	0.3	0.2
Fe	soil concn	17.8a	16.7ab	14.8b	13.5b	19.2a	17.9b	16.0c	14.5c
	plant concn	0.1a	0.03b	0.04b	0.04b	0.2a	0.05b	0.04b	0.03b
	plant uptake	8.4	3.4	5.3	3.9	14.4	5.9	3.6	1.5
Mn	soil concn	201c	262c	308b	416a	226c	257c	341b	411a
	plant concn	90c	36e	92c	116b	164a	30e	46e	65d
	plant uptake	6.9	4.8	12.9	10.6	11.5	3.2	3.9	3.2
Na	soil concn	88.4d	201.6c	295.4b	571.8a	80.0d	178.1c	283.5b	517a
	plant concn	309.6c	395.2c	807.6b	1266.9a	79.5c	100.2bc	117.1b	402.8a
	plant uptake	24.1	52.6	114.1	116.3	5.6	10.8	10.0	20.0
As	soil concn	0.26a	0.07b	0.08b	0.05b	0.22a	0.07b	0.07b	0.07b
	plant concn	ND[c]	ND	ND	ND	ND	ND	ND	ND
	plant uptake	NA[d]	NA	NA	NA	NA	NA	NA	NA
B	soil concn	1.4d	2.5c	3.6b	5.8a	2.0c	2.3c	4.6b	5.3a
	plant concn	22.1b	23.9a	22.4ab	23.6ab	18.9a	17.9a	17.6a	18.4a
	plant uptake	1.7	3.2	3.2	2.2	1.3	1.9	1.5	0.9
Cd	soil concn	1.2a	1.1a	0.9a	0.1b	1.1a	1.1a	1.1a	0.1b
	plant concn	0.4a	0.1b	0.2b	0.2b	0.7a	0.3c	0.4b	0.3c
	plant uptake	27.2	16.0	28.3	18.4	46.3	33.3	35.2	12.4
Co	soil concn	54.3a	51.0a	46.6b	44.4b	58.4a	52.2b	53.0b	45.0c
	plant concn	0.1a	1.6a	1.2a	1.5a	0.6a	1.0a	1.6a	2.0a
	plant uptake	10.1	214.1	175.2	141.4	43.5	106.4	139.0	98.7
Cr	soil concn	10.2a	9.8ab	9.2b	9.2b	11.0a	10.3ab	10.2ab	9.6b
	plant concn	ND	ND	ND	ND	ND	ND	ND	ND
	plant uptake	NA	NA	NA	NA	NA	NA	NA	NA
Hg	soil concn	0.08a	0.09a	0.10a	0.11a	0.08a	0.08a	0.16a	0.14a
	plant concn	0.25a	0.29a	0.32a	0.28a	0.39a	0.37a	0.32a	0.45a
	plant uptake	19.4	38.6	45.2	25.7	27.3	39.8	27.5	22.3
Mo	soil concn	8.7a	8.0b	7.9b	7.7b	9.4a	8.3a	8.5ab	8.3b
	plant concn	0.7b	1.1a	0.7b	1.2a	1.3ab	1.4a	1.1b	0.4c
	plant uptake	53.6	145.0	100.3	106.5	89.0	151.6	97.8	19.8
Ni	soil concn	4.5a	4.4a	4.6a	5.0a	4.6b	4.6b	4.8ab	5.2a
	plant concn	0.3ab	0.2b	0.3a	0.2ab	0.2a	0.2a	0.2a	0.3a
	plant uptake	21.0	23.9	41.0	20.2	10.5	23.7	12.9	12.9
Pb	soil concn	5.5c	7.5bc	7.9b	10.7a	6.1c	7.3bc	8.6b	11.2a
	plant concn	0.3ab	0.2b	0.2b	0.2b	0.4a	0.3ab	0.4ab	0.3ab
	plant uptake	22.5	31.9	32.5	20.2	30.1	34.4	33.5	13.9
Se	soil concn	0.21a	0.12ab	0.05b	0.03b	0.37a	0.16b	0.06c	0.01c
	plant concn	0.012	0.01	0.01	0.02	0.01	0.01	0.02	0.02
	plant uptake	0.93	1.7	1.7	1.4	0.9	1.4	1.3	0.8

[a] Means with the same letter within a fraction (column) and species are not significantly different at $P \leq 0.05$. [b] Ca, K, Mg, P, S, and Fe are in mg/g, while the concentration of other elements is in mg kg^{-1}. [c] ND, values below the detection limit. [d] NA, not applicable. Not possible to calculate due to undetected concentrations in plant tissue. Plant uptake of Ca, K, Mg, P, and S is g/pot, while plant uptake of other elements is in mg/pot.

limit of VGA-AAS and ICP (0.05 As and 0.005 Cr in solution, which corresponds to 0.3 As and 0.03 Cr mg/kg in plant tissue). Various factors such as soil pH might have influenced the accumulation of essential and trace elements in dill and peppermint tissue. For instance, decreased tissue Fe in compost-amended soils could be due to decreased HNO_3 extractable soil Fe and increased pH, as Fe is reduced at lower pH (depending on redox potential) and becomes more phytoavailable. Other ions such as Cu and Zn may also have influenced Fe uptake, and antagonistic interactions between Fe and Cu and Fe and Zn have been reported in the literature (*8*, *33*). Lower Mg accumulation in plants may have been affected by the increase in K and Ca in the compost-amended soils. Most probably, B accumulation by plants in the 60% compost-amended soils was limited by higher Ca concentration in these treatments (*34*). Mo content in the high-Cu compost was 5 mg kg^{-1}, equal to the maximum permissible Mo concentrations in type A compost (*5*). However, addition of 60% compost to the soil increased

Table 4. Fractionation of Cu and Zn in the Soils, Their Concentrations in Plants, and Plant Accumulation

			fraction concn in mg kg^{-1} air-dried matter						tissue accumulation	
metal	species	treatment	Exch	FeMnOX	OM	SB	AR soil[d]	recovery[c] (%)	tissue (mg kg^{-1})	uptake (μg/pot)
Cu	dill	0%	bd[b]	1.8d	1.2d	5.6c	10.0d	86	2.6d	0.2
		20%	29.2c[a]	113c	56c	15b	297cc	72	8.8c	1.2
		40%	51b	203b	91b	35a	565b	67	15.0a	2.1
		60%	54b	315a	135a	44a	761a	72	12.0b	1.1
	mint	0%	bd	1.1d	0.9d	4c	6.8d	88	5.8c	0.4
		20%	19d	73c	41c	16b	206c	72	8.2b	0.9
		40%	43b	174b	75bc	44a	508b	66	9.3b	0.8
		60%	64a	280a	102b	45a	782a	66	12.2a	0.6
Zn	dill	0%	bd	bd	2a	26a	44d	64	22.9d	1.8
		20%	29c	8c	5a	20ab	73bc	85	28.1c	3.7
		40%	53b	22b	5a	20ab	97b	103	36.3b	5.1
		60%	77a	38a	2a	16b	117a	114	40.6a	3.7
	mint	0%	bd	bd	3a	23ab	46d	57	21.0a	1.5
		20%	20c	5c	5a	15b	66c	68	13.5b	1.4
		40%	47b	20b	3a	16b	92b	93	15.1b	1.3
		60%	79a	42a	2a	14b	125a	110	16.6b	0.8

[a] Means with the same letter within a fraction (column) are not significantly different at $P \leq 0.05$. [b] Below the detection limit. [c] Recovery is the % of the sum of all fractions relative to the aqua regia extractable (total) concentrations of the respective metal. [d] Concentration of Cu and Zn in aqua regia decomposed soil. Exch, exchangeable fraction of Cu or Zn in soil. FeMnOX, Fe and Mn oxides fraction of Cu or Zn in soil. OM, organic matter bound fraction of Cu or Zn in soil. SB, structurally bound fraction of Cu or Zn in soil. AR, aqua regia extractable fraction of Cu or Zn in soil.

slightly tissue Mo of dill but reduced tissue Mo of peppermint. In all treatments, tissue Mo of both dill and peppermint was within the normal range for Mo in plants (*8*). Our results suggest that dill may be placed in the group of crops with medium uptake of Na, while peppermint may be in the group of plants with low and very low ability to accumulate Na (*35*). Some of the trace element concentrations in dill and peppermint tissue were not affected by the treatments.

Distribution of Cu and Zn in Different Soil Fractions. The forms of heavy metals in soil greatly influence their availability to plants and other biota. Sequential extractions provide an understanding of the distribution of bound heavy metals in soil (*48*) and are used to predict phytoavailable amounts of metals in both short- and long-term time periods. Measurements of total amount of metals in soil may be less important than an estimation of available and potentially available forms such as exchangeable and FeMnOx. Knowledge on the amount of OM metals is also important, since OM may decompose in time and release the OM-bound metals. Sequential extractions have the ability to determine the association of heavy metals with different mineralogical phases and are accepted methods to evaluate solid speciation of Cu, Zn, and Mn in soil.

Most of the Cu in the unamended soil was found in the SB fraction, while EXCH-Cu was below the detection limit of FAAS (**Table 4**). The addition of the high-Cu compost to both crops increased the EXCH-Cu in the 60% treatment 2−4 times relative to the EXCH-Cu in the 20% treatment. In the 60% compost treatment, the FeMnOx-Cu and OM-Cu were increased by more than 100 times and SB-Cu by eight times relative to the respective Cu concentrations in unamended soils. Our results are relatively close to the results reported by other authors (*36*), who found that the application of composted sewage sludge (44 Mg/ha) increased Cu in all fractions in three loamy soils, but the biggest increase was detected in the organic fractions. In the same study (*36*), it was reported that the EXCH-Cu was increased by 50−60% in the three soils relative to the unamended soil, although the OM-Cu increased more than twice and the total Cu increased twice relative to the unamended soil.

Interestingly, extremely high total Cu concentrations in soil of the 20, 40, or 60% treatments did not result in corresponding increases in tissue Cu (**Table 4**). Although the addition of high-Cu compost to soil resulted in a significant increase of all Cu fractions in the soil, Cu concentrations in both crops were below toxicity levels for other plants (*8*). Tissue Cu concentrations in peppermint from our experiment were below the values reported by others (*15*, *31*), probably due to different conditions in the three experiments. It has been demonstrated that a small but significant increase in tissue Cu of peppermint was correlated with increased HNO_3 extractable Cu in the soil (*16*). However, other workers (*31*) did not find an increase in tissue Cu as a result of increased application of sewage sludge that increased Cu in the soil (up to 182 mg kg^{-1}). Results from this study are not in agreement with other studies reporting no increase in tissue Cu as a result of increased Cu content in soil (*3*, *37*−*39*). Different plant species have different responses to Cu and other heavy metals (*36*, *39*−*41*). Other investigations, however, have reported increased levels of bioavailable Cu in soils and increased Cu content in plant tissue as a result of compost application (*42*−*45*).

With the increasing rate of compost application, aqua regia extractable Zn, EXCH-Zn, and FeMnOx-Zn fractions increased significantly, but OM-Zn and SB-Zn did not change. Interestingly, in the 0% compost treatment, both crops had similar tissue Zn levels, about 20 mg kg^{-1} dry matter, which is regarded as sufficient for normal plant growth and development (*8*). Our results suggest dissimilar species response to elevated Zn content of the growth medium. Peppermint tissue Zn did not correlate well with Zn concentration in any particular soil fraction. Other authors (*36*) did not find an increase in EXCH-Zn fraction in one of the soils they investigated; however, the authors reported an increase in EXCH-Zn for two other soils. Research has demonstrated that Cu normally has a higher adsorption affinity than Zn in soils and biosolids-amended soils (*46*, *47*). Results from our experiment support the above understanding.

The overall proportion of Cu concentrations in various fractions was as follows: FeMnOx > OM > EXCH > SB; however, for the unamended soil, the sequence was SB > FeMnOX = OM > EXCH. We did not find significant positive correlations between Cu amounts in any particular soil fraction and Cu accumulation in dill and peppermint tissue. The recovery percentage of Cu [the sum of Cu amounts recovered in the sequential extraction procedure (steps i−iv) relative to “pseudototal” concentration recovered with aqua regia] was between 66 and 88% for Cu and between 57 and 114% for Zn. These results confirm the findings of Davidson et al. (*26*) about the inconsistent recovery of heavy metals by the BCR sequential extraction procedure. However, other reports suggest that the BCR sequential procedure provides very similar recovery of heavy metals as compared to the traditional Tessier et al. method (*28*, *48*), although the Tessier et al. procedure may recover greater amounts of heavy metals (*27*). However, although a number of authors have used the Tessier et al. procedure or modification of it for acid soils (*17*, *49*, *50*), the Tessier et al. procedure might not be ideal for acid soils, since it includes a step for extracting carbonates. Still, even in acid soils, there are “acetate soluble components”, which may not be carbonates and which may correlate well to uptake by plants.

Research has shown that Cu is strongly bound by soil organic matter (*36*, *51*−*53*). Hence, the addition of compost to the soil may increase the total Cu concentration, but it would not necessarily increase Cu bioavailability. Some studies have shown that organic amendments increased NaOH extractable Cu and decreased nitric acid extractable Cu (*36*, *54*, *55*). Other authors (*36*) reported that compost addition increased the total metal soil concentration; however, the increases were in the less available Cu fractions. In the same study (*36*), authors did not find correlation between total soil and plant concentration of Cu. Our results support the findings of other authors (*36*).

GC analysis of the dill and peppermint essential oils revealed some variation in the content of menthol and mentyl acetate in the oils depending on the treatments, which might be a physiological reaction to initial Cu toxicity. Despite high rates of high-Cu compost applications, dill and peppermint oil composition remained within the range of “typical” oil composition for these crops (*56*). Hence, from a practical point of view, dill and peppermint essential oils produced on growth medium with additions of high-Cu compost may receive the normal market price. The results from this study on alterations in oil constituents are in disagreement with the reports of refs *15*, *16*, and *31*, who did not find alterations in peppermint oil composition due to the elevated metal concentrations in the growth medium and in the tissue. In a similar experiment with basil, we found that application of similar high-Cu compost to basil altered the basil essential oil composition (*17*).

The heavy metal content of the dill and peppermint oils from all of the compost treatments was below the detection limit of AAS. Two studies (*15*, *31*) reported that heavy metal concentrations in peppermint essential oils derived from plants that had been grown under various treatments were similar relative to those plants grown in a control soil, which had normal background levels of metals. Previously, we reported that additions of high-Cu compost to the growth medium of basil did not increase Cu concentration in basil essential oil (*17*). Results from this study suggest that Cu does not accumulate in the final product (i.e., essential oils) of peppermint and dill when plants are grown in a medium with very high content of organically bound Cu. Hence, peppermint and dill might be suitable crops for Cu-polluted agricultural soils, since the essential oil would not be contaminated with Cu. Because essential oils are added in trace amounts to food products, detectable amounts of Cu in the oil would not contribute much to the total Cu uptake by humans. In addition, Cu does not seem to accumulate in these two crops in amounts toxic for plant or mammals, so after the extraction of essential oil, dill and peppermint residue could be safely fed to sheep and other animals. Copper is regularly used in animal diets at concentrations of about 8−11 mg kg^{-1} (*57*). The difference between the sufficient and the toxic levels of Cu in an animal’s diet is narrow. Copper concentrations of 20 mg kg^{-1} may be toxic to sheep; Cu in sheep diet should never exceed levels of 25 mg kg^{-1} (*57*). Further research is needed to assess compost and soil properties that influence the synthesis and accumulation of various compounds in dill and peppermint oil. Also, further research is needed to evaluate the effect of high-Cu compost to plants from other families and to other soil types.

Results from this study suggest that the application of mature high-Cu compost to agricultural crops may not result in phytotoxicity or accumulation of excessive Cu in plant tissue. Our results advocate a need to review current regulatory levels for Cu in composts. Such reevaluation has to include studies on long-term bioavailability and accumulation of Cu following repeated applications and effects on other environmental endpoints. The problem of the soil acting as a “sink” and the distribution of Cu and other heavy metals among various soil pools ought to be considered in the long-run, thereby potentially affecting uptake of plants and other soil biota.

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