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Research Article

Trace Elements Characterization in Fresh and Composted Livestock Manures

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Abstract

Animal manure is identified as the alternative of fertilizers to enhance the soil fertility and crop production. But the application of animal manure may contaminate the environment with heavy metals; thus it is imperative to determine the heavy metal contents of manures. The present study was aimed to compare the trace elements in fresh and composted animal manures using the sequential fractionation method. Composted and fresh animal manures from four different animals, [buffalo (BF), cow fresh (CW), goat (GT) and poultry (PL)], were selected to determine various fractions of iron (Fe), zinc (Zn), nickel (Ni), manganese (Mn) and mercury (Hg). Metals were stepwise fractionated into exchangeable, adsorbed, organically bound, carbonate precipitated and residual forms. The extractability of elements considerably varied depending on the type of livestock and extraction method. The composted and fresh manure exhibited the lower concentrations of water soluble extractable metals as compared to other fractions. A great proportion of metal was detected in carbonate and residual forms. Irrespective of the form of element, the reagents were varied in order of HNO_3> EDTA >NaOH> KNO_3> H_2O, while metal species were found in the order of Fe> Hg >Mn> Zn > Ni. Iron, Mn and Hg were found in higher concentration in composted manures whereas Ni and Zn concentrations were sparingly higher in fresh manures. Analytical results indicated that composting of animal manures might have encouraged soil microbial activity, which promoted trace mineral supply thus improving the plant nutrition. The variations in the heavy metals could be related to the chemical properties of the individual metals and the characteristics of manure.

Keywords: Composting; Livestock manures; Heavy metals; Fractionation

Introduction

Livestock manure has been used for centuries as a fertilizer for farming, as it improves the soil structure that holds more nutrients and water for better crop production. It also encourages soil microbial activity which promotes the trace element supply thus improving plant nutrition. Animal manure either fresh or composted has been traditionally applied to the agricultural fields for decades as an organic fertilizer [1]. The use of composted manure will contribute more to the organic matter content of the soil. Fresh manure may contain higher amounts of viable weed seeds, which can lead to the weed problems. In addition, various pathogens may be present in fresh manure which can cause various illnesses to individuals eating fresh produce. The residual effects of the manure and compost are important. When manure and compost are used to fertilize crops, soil organic matter will increase with the passage of time and subsequent rates of fertilizer application can generally be reduced because of increased nutrient cycling. Composts are considered as valuable product used for soil amendment of agricultural lands. The application of composted animal manure has increased over the years. This practice not only improves the quality of the crops but also preserves the environment [2,3]. However, non-composted manure or immature compost may impart adverse effects on plant growth and/or seed germination [4,5].

Heavy metal contamination of soil is a major concern because of their toxicity and threat to human life and other biota. Toxic heavy metals entering the ecosystem may lead to geo-accumulation, bio-accumulation and bio-magnifications. Excessive accumulation of heavy metals in soil and other media may eventually enter food chain [6,7]. Consequently, many countries in the world have established specific guidelines and standards for the application of composts in agricultural lands.

Trace metal composition of composts widely varied depending on the sources, composting process [6] and geographical location [8]. Like soils sediments and sludge, trace metals can exist in a variety of forms in composts. These forms include water soluble, exchangeable, bound to organic substances, occluded or co-precipitated with oxides, carbonates and phosphates, or other secondary minerals and ions in the crystalline lattices of the primary minerals [7,9,10,11]. Total trace metal composition of composts is of little importance in determining the total metal uptake by plants and consequently contamination of the food chain since different forms have variable mobility, bioavailability and potential environmental contamination. It is therefore, important to know the distribution of each trace metal form [10,11]. Conceptually, sequential fractionation categorizes metals associated with chemically homogeneous fractions that ultimately affect metal availability. Sequential extraction (or fractionation) procedures have been predominantly developed to determine the amounts and proportions of metals present as different forms in soil or sediment samples [12,13].

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Table 1: Physico-chemical properties of manure used in this study.

Manure type	Livestock Sources	Water Soluble				
		P	K	Ca	Mg	pH
Fresh	GT	127	239	174.6	419.5	7.7
	BF	90	163.8	217.2	447.8	7.6
	CW	119	81.6	263.2	708.3	7.5
	PL	154	119.9	153.3	618.4	7.6
Compost	GT	90	77.5	194.8	586.4	7.3
	BF	89	114.4	333.3	717.6	7.4
	CW	104	80.3	224	630.8	7.6
	PL	141.5	157.3	296.8	690	7.4

Note. *For this and subsequent figures BF= buffalo fresh and composted manure, CW= cow fresh and composted manure, GT= goat fresh and composted manure, PL= poultry fresh and composted manure and P,K,Ca,Mg= water soluble phosphorus, potassium, calcium, magnesium.*

Metals fractionation in animal waste can provide useful information to predict their bio-availability and the potential for contamination of soils and waters. Most researches on metal contamination of soils have been conducted in relation to municipal sewage sludge application and to date; very little work has been done on metal input from animal manure byproducts as fresh and composted ones [14]. Research reports on the concentration of metals in composted and fresh manure samples are particularly lacking. Therefore, the present study was aimed to evaluate the metal constituents (Fe, Zn, Ni, Mn, Hg) in fresh and composted animal manures.

Materials and Methods

Animal composted waste and fresh manure samples were collected from the village Pohar, District Abbottabad. Fresh and composted manure samples of different animals were collected. These included: cow (CW), buffalo (BF), poultry (PL) and goat (GT). The manure samples were air dried, crushed, and sieved (< 0.5 mm) to ensure homogeneity and digested in a mixture of acids (HNO_3 and $HClO_4$). Total elements i.e. nickel (Ni), iron (Fe), zinc (Zn) and mercury (Hg) in the extract of digested manure samples were determined by atomic absorption spectrophotometer (*IBSRAM*.1994). The sample weighing 0.25 g was digested with 5 mL concentrated $HClO_4$ over a hot plate for 1 h. After drying 20% HNO_3 was added to the sample and it was heated again for 1h. The solution was diluted to 50 mL with deionized water and passed through a 0.22 µm filter. The P was determined by a spectrophotometer at 710 nm [15]. The manure samples were extracted for water soluble P, K, Ca, Mg and other metals using deionized distilled water.. Potassium, Ca, Mg, and other metals were determined by an atomic absorption spectrophotometer (Model Z-2300, Hitachi, Japan). Soil pH were measured in soil-water (1:5; w: v) suspensions (Table 1). Treatments were applied in triplicates and data was statistically analyzed using Stat view software (SAS, 1999).

Metal fractionation

Modified versions of a sequential extraction procedure [16] were employed to fractionate the solid-phase chemical forms of the metals. The modified extraction procedure was developed on the basis of studies with model heavy metal compounds [17]. Sequential extractions were carried out in triplicates using 0.5 g of fresh and composted animal manure samples in 50 mL centrifuge tubes. The trace elements were fractionated into exchangeable, adsorbed, organically bound, carbonate precipitated and residual forms by sequential extractions with 25 mL of the following reagents: 0.5MKNO_3, 16 hours; deionized water, 2 h (extracted three times and combined); 0.5MNaOH, 16 hours; 0.05MNa_2 EDTA, 6 h, and 4MHNO_3 16 h at 80°C. During each extraction, the sample suspension was centrifuged at 10000 rpm for 10 min and the resulting supernatant solution was decanted and filtered through 0.22 µm filter. The metal content in the filtered solution was determined by an atomic absorption spectrophotometer, using standards with the extract to form the background matrix. A complete mass balance system was used to determine the amounts of metal extracted. Taking into account the volume of water carried over in the next extraction, each metal fraction was corrected for the aqueous phase elements as suggested by Stover et al. [16].

Results and Discussions

The physico-chemical properties of the manures were shown in Table 1. Metal fractionation in animal manure provides useful information to predict their bioavailability and potential for contamination of soil and water. During the extraction process, the metal concentrations varied based on extraction method. The overall metal fractions extracted by reagents varied in the order HNO_3> EDTA >NaOH> KNO_3> H_2O (Table 2,3). Emmerich et al. [9] reported that like soils, sediments and sludge, trace metals can exist in a variety of forms in both composted and fresh animal manures. These forms include water soluble, exchangeable, linked to organic substances, co-precipitated with oxides, carbonates and phosphates, or other secondary minerals and ions in the crystalline lattices of the primary minerals. The extractability of elements considerably varied depending on type of livestock and extraction method. The principle metal forms in manure and manure treated soil are soluble, exchangeable, absorbed, organically bound, oxide bound and precipitated [18,19]. During the study, Iron (Fe) fractions extracted by reagents varied in the order of HNO_3> EDTA > KNO_3> H_2O >NaOH (Table 4). Regardless of extractant, composted manures differed for Fe as BF>GT>PL>CW; whereas fresh manures varied as GT>BF>PL>CW. The maximum Fe concentration was found in composted BF manure (2174 mg kg^{-1}) and the minimum Fe concentrations was detected in fresh CW manure (1664 mg kg^{-1}) (Table 4). Milan et al. [20] reported lower ratios of composts

Table 2: Zn fractions (mg kg-1) in fresh and composted animal manures.

Manure type	Animal manure	KNO_3	H_2O	NaOH	EDTA	HNO_3	HNO_3 $HClO_4$
Composted manure	BF	8.2	2.5	15.6	86.1	70.1	145
	CW	4.1	3.7	17	66.4	25.7	163
	GT	5.9	1.8	25.2	102.3	52.8	135
	PL	10.7	4.5	40.3	148.9	71.2	187.5
Fresh manure	BF	9	3.5	40.7	49.9	30.7	170
	CW	4	3.4	33.7	62.2	35.6	150.5
	GT	1.2	5.4	41.6	82.8	31.7	185
	PL	7.5	4.1	41.8	139.2	63.4	160
	LSD (0.05)	0.63	0.35	0.63	3.75	0.63	0.22

Table 3: Mn Fractions (mg kg-1) in fresh and composted animal manures**.**

Manure type	Animal manure	KNO_3	H_2O	NaOH	EDTA	HNO_3	HNO_3 $HClO_4$
Composted manure	BF	5.9	7.6	22.6	450.7	248.6	475.3
	CW	1.3	9.7	13.9	346.5	273.6	430.9
	GT	3.6	10.3	21.7	402.6	223.7	434.1
	PL	1.4	7.7	9.2	505	168.2	466.1
Fresh manure	BF	4.6	9.1	16.4	277.7	136	456.7
	CW	2.9	9.8	14.7	282.5	86.7	437
	GT	1.9	8.7	24.1	307	105.9	450.8
	PL	4.9	7.3	12.7	494	183.6	438
	LSD (0.05)	0.14	0.16	0.16	1.01	0.59	3.64

Table 4: Fe Fractions (mg kg-1) in Fresh and Compost of different animal sources.

Manure type	Animal manure	KNO_3	H_2O	NaOH	EDTA	HNO_3	HNO_3 $HClO_4$
Composted manure	BF	26.8	4.3	0.8	564.8	1781	2147
	CW	32.4	4.85	0.45	520.8	1572	1825.5
	GT	15.1	5.75	0.85	222.2	1632	2058.5
	PL	8.1	2.5	0.2	843.3	1562	1873
Fresh manure	BF	14.2	3.35	0.6	217	1145	1821.2
	CW	24.8	1.15	0.2	742.3	1308	1664
	GT	12.3	0.85	0.4	112.4	1509	1855.2
	PL	7	1.1	0.5	621.4	1474	1806.3
	LSD (0.05)	0.191	0.337	0.147	2.325	12.06	38.92

versus fresh manure for Fe. This suggests that Fe could be more resistance to acid leaching or become fixed upon composting. Zinc (Zn) fractions extracted by reagents varied in the order of EDTA>HNO_3>NaOH>KNO_3> H_2O (Table2). Composted manures differed for Zn as PL>CW>BF>GT whereas fresh manures variated as: GT>BF>PL>CW (Table 2). The maximum Zn concentration was found in PL compost (187.5 mg kg^{-1}) and the lowest concentration was found in GT compost (135 mg kg^{-1}) (Table2). Relatively higher Zn concentrations of compost (largely derived from animal feeds) were used as additives Sims and Wolf, 1994 [21]. In addition, most of the dietary Zn is not absorbed in animal bodies but excreted in animal manures. Qiao et al. [22] showed that the application of biosolids increased the Zn concentration in the reducible fraction of paddy soil. Nickel (Ni) fractions extracted by reagents varied in the order HNO_3>EDTA>NaOH>KNO_3> H_2O (Table 5). Composted manure samples differed for Ni in the order of BF > GT > CW > PL, while the order of fresh manure samples was: GT>BF>CW>PL. The maximum Ni concentration was found in fresh manure of GT (92.25 mg kg^{-1}) and the minimum Ni was present in PL compost (66.15 mg kg^{-1}) (Table 5). The results of Walter et al. [23] showed that the application of biosolids to agricultural soil resulted in significantly higher concentrations of the total trace metals as compared to control. Rajaie et al. [24] found that Ni was predominantly bound to oxides and in the residual fractions. Manganese (Mn) fractions extracted by reagents appeared in order HNO_3>EDTA>NaOH>H_2O>KNO_3 (Table2). Composted manure samples differed for the release of Mn as BF>PL>GT>CW whereas the Mn concentrations in the fresh manure samples were: BF>GT>PL>CW (Table 3). The maximum concentration of Mn was

Table 5: Ni Fractions (mg kg-1) in Fresh and Compost of different animal sources.

Manure type	Animal manure	KNO_3	H_2O	NaOH	EDTA	HNO_3	HNO_3 $HClO_4$
Composted manure	BF	9.3	10.1	16.3	20.4	41.9	83.85
	CW	12.5	14.8	17.4	18.6	40	71.65
	GT	9.7	9.5	14.7	21.4	35.6	81.95
	PL	12.2	14.2	20.1	19.7	33.6	66.15
Fresh manure	BF	10.4	5.9	9.8	19.3	31.7	81.5
	CW	8.1	12.3	13.5	17.5	30.1	74.2
	GT	14.4	9.9	11.9	16.6	30.8	92.25
	PL	10.7	7.5	16.1	15.8	34.8	74
	LSD (0.05)	0.276	0.391	0.493	0.163	0.687	1.929

Table 6: Hg Fractions (mg kg-1) in Fresh and Compost of different animal sources.

Manure type	Animal manure	KNO_3	H_2O	NaOH	EDTA	HNO_3	HNO_3 $HClO_4$
Composted manure	BF	270.4	197.6	317.6	161	60.4	788.3
	CW	238.4	274.9	332	140	44.9	771.4
	GT	181	146.5	275.5	240.2	70.6	799.3
	PL	141.4	245.3	272.1	156.4	39.8	787
Fresh manure	BF	240.8	191.3	189.9	150.9	37.5	728.3
	CW	297.4	256.2	341.4	179.3	43.2	783.7
	GT	206.7	212.5	296.7	183.2	32.9	835.9
	PL	238.6	283.7	320.3	254.3	50.8	758.5
	LSD (0.05)	1	1.9	1.25	2.05	1.2	3.84

found in BF compost (475.3 mg kg^{-1}) and the lowest in CW compost (430.9 mg kg^{-1}) (Table3). The results of Zeng-Yie [25] showed that Mn was relatively abundant in the composts. The Mn recovery from the composts is affected not only by the digestion method but also by the type of compost. Mercury (Hg) fractions extracted by reagents varied in order of NaOH>KNO_3>H_2O>EDTA>HNO_3 (Table 6). The composted manure samples differed for Hg as GT>BF>PL>CW, while for fresh manure samples differed as GT>CW>PL>BF (Table 6). The maximum value of Hg was found in fresh manure of GT (835.9 mg kg^{-1}) and the minimum was of observed in fresh manure of BF (728.3 mg kg^{-1}) (Table 6). El-Arhaf et al. [26] found that the reduction of Hg in the samples of dairy cattle and chicken feed rations and manures prepared by acid digestion was rapidly, smoothly, and quantitatively affected by any reducing agent. Ure [27] reported that the EDTA method extracts the elements from all the non-silicate bound soils. Bolan et al. [28] reported that the metal concentration considerably varies among animal manures. Additional variation is associated with the age of the animal, type of ration, housing type and waste management practices. Increase in the metal concentration in animal feed have often resulted in the metal concentrations of the manure by-products. The type of bedding material in animal wastes units may influence the litter dry matter and other chemical properties [21]. Eneji et al. [29] reported that the major concern of soil pollution via available heavy metals of manures can be minimized through composting under aerobic conditions. Pare et al. [30] reported decreases in the extractability of some heavy metals during composting of biosolids and municipal solid wastes, indicating a reduced risk of their entry into food chain through crops and water. According to Pare et al. [30], proportionally greater increase in humic compounds during composting would cause more heavy metals to interact with sites of greater binding strengths where they become less accessible and available to plants and extracting agents. Sequential extraction of metals indicates a decline in soluble components and thus an increase in residual, organically bound forms.

Irrespective of manure, trace elements varied in order of Fe>Hg>Mn>Zn>Ni (Table 2-6). Previous studies indicated that metals in the sable organically bound, carbonated and residual forms of animal wastes were highly variable [31]. The comparison of fresh and composted manures on elemental basis showed that the Iron was greater in composted manures than the fresh manures. Zinc concentration was greater in fresh manures than the composted manures. Manganese concentration was maximum in composted manures than the fresh animal manures. Nickel showed greater concentration in fresh manures as compared to composted manures and mercury resulted in greater concentration in the composted manures. Faridullah et al. [31] reported substantial impact of ashing on the fractions of the trace elements in chicken and duck litter. They observed a decreased solubility of the metals in the incinerated samples to the formation of insoluble metals complexes accompanied by enhanced alkaline pH of ash. Trace elements precipitation is highly pH dependent and results in the increase in pH for many trace elements. Lindsay [32] also reported trace elements from soluble precipitates with phosphates, sulfides and other anions. The metal content of manure by-product primarily depends on the amount used in the feed and health remedies. Increases in metal concentration in animal feed have often resulted in corresponding increase in their concentration in the manure by-product [32]. Heavy metals in higher concentrations have the potential to be both phytotoxic and zootoxic [33]. Since the metals phytotoxicity in soils is determined by their bioavailable fractions; thus, the lower concentrations of soluble and exchangeable elements in particular manure may produce less concern about potential effects of associated metals in agro-ecosystem.

Long-term application of manure simply changes the levels and forms of cations. Inorganic fertilizers also enhance the amount of cations in the soil since the nature and composition of manures varied considerably by origin, the observed changes in the soluble cations could be related to the mineral composition of manure. Lindsay [32] reported that trace elements form sparingly soluble precipitates with phosphate, sulfides, and other anions. Trace element precipitation is highly pH dependent and results in an increased pH for many trace elements. Water-soluble and exchangeable fractions of the metals are readily released to the environment; whereas, the residual fractions are immobile under natural conditions. The available pool of metals is also the most labile form in the soil environment and has greater leaching potential than the other forms [10,13,29] reported decrease in the extractability of some heavy metals during composting of biosolids and municipal solid wastes, indicating a reduced risk of their entry into the food chain. Metal differences in manures may reflect the variability in the total amount of element in a particular waste.

Conclusions

The study concluded that acid extracted metals were higher in the composted manure than fresh manures. The metals varied in order of Fe> Hg>Mn>Zn>Ni and the amount of these metals in reagents varied in the order HNO_3>EDTA>NaOH>KNO_3>H_2O but the absolute amounts differed for composted and fresh manures. The composted and fresh manure exhibited the lower concentrations of extractable metals as compared to other fractions. This may reduce the risk of metal transfer from soil to surface waters. Fe, Mn and Hg concentrations were higher in composted manures whereas Ni and Zn showed their maximum concentrations in fresh manures. This research predicted that composting animal manures before application could affect the forms and concentrations of trace elements in the fields. The use of composted animal manures as soil amendment could be useful and would dilute the major concerns of animal waste management.

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