

EVALUATION OF ORGANIC MATTER AND HEAVY METALS DERIVED FROM THE TANNERY SLUDGE COMPOST

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Abstract

This study focused on the characterization of organic matter (OM) and heavy metals during the composting of tannery sludge. Total concentrations of chromium, zinc, copper, lead, and cadmium in dry compost were reduced and complied with the standards of the Canadian Council of Ministers of the Environment (1995) making the compost suitable for use as a fertilizer and soil conditioner. The composting process significantly produced stable and mature compost and the concentration of the compost organic matter fractions generally decreased during the composting time while the C/N ratio exhibited a decreasing pattern throughout the composting process. Water and solution (0.01 M CaCl₂) soluble organic carbon concentrations rapidly increased to a maximum on the 20th day and declined thereafter. The changes in the humic fractions reveal that the amount of humic acids decreased during the first 20 days and increased thereafter. Reverse trends were observed in the case of fulvic acids (FA). The amounts of FA reached the maximum value on day 20 and declined thereafter.

Keywords: Tannery sludge, compost, humic matter, heavy metals

Introduction

Leather processing is a major industry that produces up to 600 tons of sludge annually in Kenny Leather Sdn Bhd (Melaka-Malaysia). Sludge from leather processing contains large concentrations of inorganic nitrogen (N) and N-rich organic residues (INE-DGMRAR, 1999). Apart from organic material, which releases valuable nutrients upon decomposition, tannery sludge may contain pathogens mainly

of faecal origin, as well as chromium (Cr) and toxic organic components, all of which pose a serious threat to the environment.

The process of tanning consists of the transformation of animal skin to leather. Animal skin (cow, goat, sheep and other animals) is subjected to different processes to eliminate meat, fat and hair, involving different chemicals such as sodium hydroxide, sodium hypochlorite,

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enzymes, lime, chlorides, sulfuric acid, formic acid, ammonium salts, kerosene, and other compounds (SEMARNAP, 1995). The resulting hide is then treated with Cr^{+3} or tannins, mineral salts and colors to obtain leather.

Direct agricultural use of tannery sludge is limited due to the presence of pathogens, fermentation of any unstable organic matter and the organic and inorganic pollutants it contains (Dudka and Muller, 1999). To overcome the risks incurred by the direct use of this waste in agriculture, treatment is required to minimize and eliminate the undesirable effects and to optimize the efficiency of the materials once applied to the soil.

Compost production is the process whereby thermophilic, aerobic microorganisms convert organic materials into a hygienic, biostable product. The microorganisms must be provided with nutrients, water and oxygen supply to thrive. Although composting is a process comprising of natural biodegradation of organic matter, it involves a variety of physical parameters varying simultaneously (Agnew *et al.*, 2003). During composting, part of the organic matter is mineralized to carbon dioxide, ammonia and water, whereas the other part is transformed into humic substances which are structurally very similar to those present in soils (Hernandez-Apaolaza *et al.*, 2000). The exogenous natural organic matter (a material that growing or originating from outside or due to external factors), e.g. of the compost origin, introduced to soils may strongly influence the behavior of organic xenobiotics (a compound that is foreign to a living organism e.g. drugs; carcinogens; and various compounds that have been introduced into the environment by artificial means) (Iglesias-Imenez *et al.*, 1997). The sorption capacity of hydrophobic pollutants (pesticides) by soils amended with exogenous organic matter containing humic-type compounds increases with the degree of evolution (humification) of amendments. In addition, non-composted or immature compost or other manure applied to agricultural soils may also cause phytotoxicity to plants, nitrogen and oxygen deficiency, and adversely affect the environment e.g. Nitrogen immobili-

zation is a major problem for immature composts derived from low N content feedstock such as MSW. Plant grown in composts that immobilize N are often yellow and stunted because of N deficiency, in addition if the $\text{NH}_4\text{-N}$ concentration is above $1,000 \text{ mg kg}^{-1}$, it can produce enough water-soluble NH_3 to be toxic to plant roots) (Garcia *et al.*, 1992; Veeken *et al.*, 2000).

Many experimental parameters have been proposed to assess compost maturity and stability. The degree of organic matter transformation during composting can be predicted with the C/N ratio, concentration of water soluble fraction of organic carbon and humification indices (Drozd *et al.*, 1997; Hsu and Lo, 1999). It has been also proposed that the amounts of humic substances and their fractions (fulvic acids, humic acids, and humins) can be used as indices of compost maturity (Senesi, 1989). However, the extraction and fractionation techniques of humic substances are non-specific, and many non-humic substances may be extracted during alkali treatment of soils or compost. The compost organic matter is very rich in non-humic substances which are also soluble in alkali solutions. Hence, a simple and common extraction procedure based on NaOH solution without any pre-extraction steps may lead to serious errors in compost maturity assessment and structure investigations of humic macromolecules (Adani *et al.*, 1995). The objective of this work was to investigate some of the chemical changes in organic matter fractions and heavy metals during composting of tannery sludge, paying special attention to the properties of humic acids and heavy metals during maturation.

Materials and Methods

Composting Process Design

The tannery sludge used in this study was collected from the Kenny Leather Sdn Bhd in Melaka, Malaysia. Windrow type composting was carried out on tannery sludge mixture consisting of approximately 200 kg of the following materials: sludge (100 kg) was

mixed with sawdust (50 kg), chicken manure (30 kg), and rice bran (20 kg) in a pile 2.5 m long, 1.0 m wide, and 1.5 m high. The mixture was prepared so as to optimize the composting parameters, i.e. 60% humidity and a C/N ratio of about 30 (Paola Castaldi *et al.*, 2005). Table 1 shows the main physico-chemical characteristics of the raw materials. The pile was turned manually every 10 days to maintain aerobic conditions during the process. Temperature was measured daily at a depth of 50 cm at 15 different positions inside the pile (bottom, surface, side and centre). The composting cycle lasted for sixty days. Samples were taken periodically: before composting (T_0), after 5 days (T_5), at 20th day (T_{20}), at 40th day (T_{40}), and at 60th day of composting process

(T_{60}). Each sample was air-dried for a period of ten days. The dried samples were ground into a fine powder.

Chemical Analysis

Representative samples of size 250 g were taken from the homogenized composting materials' mixture for determination of heavy metals and other analyses. Samples were taken from 10 different points of the windrows (bottom, surface, side, and centre) at the beginning as well as at different stages of composting process (0 days, 5th, 20th, 40th and 60th day of composting).

On aliquots (sample solution) of these representative samples, the following physico-chemical analyses were made: the pH was

Table 1. Physico-chemical properties of raw materials used in composting experiments

Paramters	Tannery Sludge	Sawdust	Chicken Manure	Rice bran	Typical soil*
Moisture, %	60.60	65.70	50.60	66.90	-
pH	7.36	5.90	7.93	7.20	6.80
E.C. (mS cm ⁻¹)	9.00	15.00	7.00	6.00	-
Organic -C, %	20.03	57.00	30.40	49.33	3.90
TKN %	0.9996	0.30	4.00	1.10	0.40
C/ N	20.038	190.00	7.60	45.72	9.75
Ash	65.00	80.00	45.00	30.00	25.00
Micronutrients					
Potassium, %	0.415	0.02	1.23	0.99	0.4-3.0
Phosphorus, %	0.097	1.17	3.02	0.23	0.05-0.20
Calcium, %	7.70	0.02	1.99	0.30	0.70
Magnesium (mg kg ⁻¹)	1,190.00	0.004	1.05	236.33	0.06-6.00
Sodium (mg kg ⁻¹)	1,006.00	64.00	123.00	98.00	3.00
Iron (mg kg ⁻¹)	1,062.00	402.00	1,738.00	142.33	55.00
Heavy metals					
Chromium (mg kg ⁻¹)	1,404.00	14.60	16.60	6.30	100.00
Lead (mg kg ⁻¹)	93.00	16.00	1.30	1.20	15.00
Cadmium (mg kg ⁻¹)	3.00	6.50	0.50	0.20	0.50
Copper (mg kg ⁻¹)	41.40	4.80	329.67	24.33	200.00
Zinc (mg kg ⁻¹)	44.67	8.20	634.67	127.00	50.00
Manganese (mg kg ⁻¹)	70.00	4.60	34.00	24.00	850.00

* Mokhtaruddin *et al.* (2001); E.C. = Electrical conductivity

* Values on dry basis

determined on a suspension of sample in water (10 g/15 ml), the total organic carbon (TOC) was measured according to the ANNE method (Aubert, 1978), and the total nitrogen (Kjeldahl method) and inorganic nitrogen were measured (Bremner, 1965). The rate of decomposition was calculated after ignition of the dry sample at 550°C (16 h). For P available the Olsen method was used (Hafidi *et al.*, 1994). Available Ca, Na, K and Mg were determined using ammonium acetate. Total P, Ca, Na, K, Mg, Fe and Mn were determined after the ash process. Phosphorus was measured colorimetrically and other elements were analysed in the extracts using atomic adsorption and flame photometry (De Souza, 1998). According to the French Association of Normalizations (AFNOR, 1993) standards, to obtain the total Cr, Zn, Cu, Pb and Cd content, 1 g of each sample was mineralized for 4 h at 550°C, and then dissolved in 5 ml of hydrofluoric acid. The solution obtained was evaporated to dryness, the residue was then dissolved with concentrated HNO₃/HCl (1:1) solution and the acid solution was diluted to analysis.

Extractable Organic Carbon

One gram of each sample was weighted into a 250-ml polyethylene flask and extracted with 100 ml of deionized water (WEOC-water extractable organic carbon) or with a 0.01 M CaCl₂ solution (SEOC-solution extractable organic carbon) by shaking for 24 h. Subsequently the suspension was transferred into centrifuge tubes and centrifuged at 3,000 rpm for 25 min and filtered through 0.45 µm filter membranes. The extracts were immediately analyzed for organic carbon using the TOC analyzer (Bernal *et al.*, 1996).

Fractionation of Humic Substances and Extraction of Pure Humic Acids

For the determination of the amount of humic fractions in compost, the Kononova–Bielczikova method was applied (Kononova, 1966). One gram of compost was extracted with 100 ml of a mixture of 0.1 M Na₂P₂O₇ + 0.1 M NaOH for 24 h. The supernatant solution was separated by

centrifugation (3,500 rpm, 30 min) and the carbon content was determined in the solution (corresponding to fulvic-FA and humic acids-HA). Fifty milliliters of the supernatant solution was acidified to pH below 2 with 6 M HCl, heated to 80°C for 2 h and allowed to stand at room temperature overnight. Soluble FA was then separated from the precipitated HA by centrifugation (3,500 rpm, 30 min). The organic carbon that corresponds to FA carbon was determined in the supernatant. The content of HA was calculated as (HA + FA) - (FA) = (HA). To obtain pure samples of humic acids (HA) from compost, an original method was applied. A compost sample (about 50 g) was extracted in a Soxhlet extractor with 250 ml of n-hexane for 24 h and then with 250 ml of chloroform also for 24 h to remove non-humic large molecules (bitumens, waxes, lipids, lignins, and tannins). The residual compost was shaken overnight with 0.1 M HCl (1:10 soil to solution ratio) to extract small molecules and the movable fraction of FA. The residual solid was shaken with 0.1 M NaOH under N₂ for 24 h (1:10 soil to solution ratio) and centrifuged (3,500 rpm, 30 min). The supernatant solution was acidified with 6 M HCl to pH below 2, heated to 80°C for 2 h and allowed to stand in at room temperature overnight. Soluble FA were then separated from the HA by centrifugation (3,500 rpm, 30 min). The precipitated HA were cleaned from clay fraction by shaking for 24 h with 100 ml of dilute HF–HCl solution (5 ml 52% HF + 5 ml conc. HCl + 990 ml H₂O).

The cleaning procedure was repeated twice. The HA sample was also washed to completely remove chlorine (Cl), and air-dried.

Results and Discussion

Characterization of Tannery Sludge

The tannery sludge was alkaline in nature (pH, 7.36). More than 83% fraction of the sludge had the equivalent particle size < 50 µ and its equivalent texture was silty clay. The wet bulk density was close to that of mineral soil, but when dry, it was relatively light with a bulk density equal to 0.14 g cm⁻³.

The sludge was rich in organic matter. The nitrogen content was higher than that of the typical soil. It also had a high content of sodium and calcium.

The results of the physicochemical characterization of the sludge presented in Table 1 showed that it was low in C/N ratio (20) and high in nitrogen content (0.99). Apart from the plant nutrient, the analysis of the sludge showed that it contained high amounts of trace elements like chromium, cadmium, sodium and lead, which all have a negative impact on plant growth (Lisk *et al.*, 1992). From the above results it appears that the general characteristics of the sludge from the tannery were at an allowable limit, except for chromium, cadmium and lead.

Compost Analysis

The main physico-chemical properties of the composted materials at different times of the process are presented in Table 2. The pH values were within the optimal range for the development of bacteria 6.0 - 7.5 and fungi 5.5 - 8.0 (Zorpas *et al.*, 2003). Two phases of the composting process were recorded: the first phase of stabilization (about 30 days), where temperature peaked at 64°C after 30 days of

processing and pH was slightly increased (7.5); the second phase of maturation (about 30 days), characterized by a temperature plateau at 35°C and slight acidification of the medium (6.6). The change in the C/N ratio from 24.5 to 15.6 and the amount of ash reflect microbial decomposition of organic matter and stabilization during composting. Electrical conductivity in a water extract of the final product did not exceed the salinity limit value of 3 mS cm⁻¹ to be used in good fertilizers (Soumare *et al.*, 2003).

The increase in total nitrogen during composting was caused by the decrease of substrate carbon resulting from the loss of CO₂ (Soumare *et al.*, 2002; Zorpas *et al.*, 2003) (Table 3). Inorganic nitrogen, N-NH₄ and N-NO₃, are usually affected by the action of proteolytic bacteria and partly incorporated into stable organic forms such as amide and heterocyclic nitrogen. Available and total P, Ca, K, Mg and Na as well Fe and Mn were more important for the use of such material as mineral fertilizers (Soumare *et al.*, 2003). Therefore, application of this material will increase the stable organic N and humic carbon and improve mineral elements necessary for plant growth.

Table 2. Physico-chemical properties of sludge materials at different times of composting

Properties	0 days	5 days	20 days	40 days	60 days	European standards applicable to composts*
Moisture, %	58.40	63.60	68.60	64.10	60.10	< 75
pH	7.30	7.40	7.50	6.90	6.60	5.5-7.0
E.C. (mS cm ⁻¹)	2.00	1.80	1.50	1.70	2.00	3
Ash, %	63.00	68.00	75.00	83.00	88.00	-
C/N	24.50	21.33	19.11	17.40	15.60	< 25

* Values on dry basis

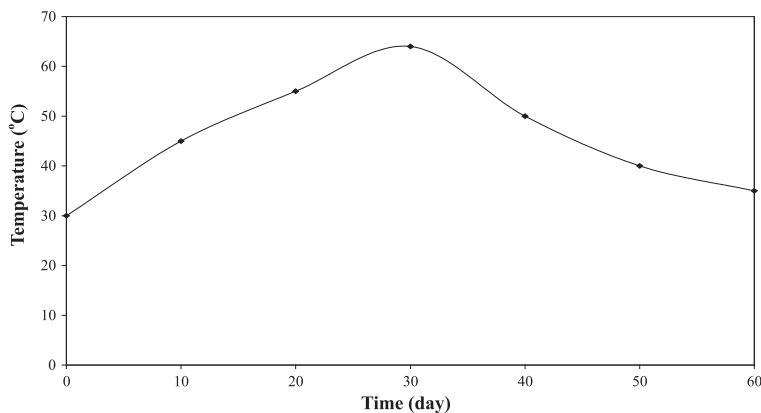
Table 3. Total content of various nutrients in compost samples at different stages of maturation

Properties	0 days	5 days	20 days	40 days	60 days	Typical compost*
TKN %	0.80	0.88	0.90	0.92	0.95	0.90
NH ₄ ⁺ -N (mg kg ⁻¹)	3.70	3.00	2.80	2.00	1.40	1.40
NO ₃ ⁻ -N (mg kg ⁻¹)	3.40	2.90	2.60	1.50	0.09	0.05
N-org. (mg kg ⁻¹)	5.40	5.90	6.10	6.80	8.20	7.30
P total (mg kg ⁻¹)	7.50	6.30	5.90	5.00	3.90	1.80
P available (mg kg ⁻¹)	4.20	3.80	3.20	2.60	1.90	0.40
Ca total (mg kg ⁻¹)	720.00	610.00	530.00	300.00	150.00	71.70
Ca available (mg kg ⁻¹)	105.00	90.000	70.00	59.00	45.00	13.90
K total (mg kg ⁻¹)	38.00	25.00	18.00	12.00	8.00	6.10
K available (mg kg ⁻¹)	12.00	10.00	8.00	5.20	2.50	2.60
Na total (mg kg ⁻¹)	980.00	740.00	540.00	290.00	140.00	4.50
Na available (mg kg ⁻¹)	250.00	180.00	120.00	80.00	50.00	4.00
Mg total (mg kg ⁻¹)	990.00	730.00	530.00	310.00	130.00	8.1.00
Mg available (mg kg ⁻¹)	100.00	80.00	60.00	30.00	10.00	1.20
Mn (mg kg ⁻¹)	85.00	80.00	76.00	50.00	60.00	59.90
Fe (mg kg ⁻¹)	1,200.00	1,900.00	2,876.00	3,850.00	5,674.00	3,768.00

* Soumia *et al.* (2005)

Figure 1 shows the temperature profile with time during the composting process. It was characterized by increasing the temperature up to 64°C, and then decreasing it to 35°C, which indicated the end of the composting process. It was observed that tannery sludge composting was able to reach high thermophilic tempera-

tures, which was due to its high nutrients content from the organic materials (Mays and Giordano, 1989). To maintain a high temperature within the windrow, the compost heap should be large enough to allow heat generated by metabolic processes to exceed the heat lost at the exposed surfaces (Eweis *et al.*, 1998).

**Figure 1. Temperature profile throughout the composting process**

Heavy Metals Analysis

Composting can concentrate or dilute heavy metals present in tannery sludge (Zorpas *et al.*, 2003). Decrease in the amounts of heavy metal depends on metal loss through leaching. The increase of metal level is due to weight loss of organic matter in the course of composting following organic matter decomposition, release of carbon dioxide and water and mineralization processes (Canarutto *et al.*, 1991).

Figure 2 shows the changes in the concentrations of metals (Cr, Zn, Cu, Pb, Cd) during composting. The order of total metal content in the final composted sludge was Zn > Cr > Cu > Pb > Cd. During composting, total content of all metals decreased. This could be explained by metal loss through leaching in the course of composting. This loss mainly occurred during the thermophilic phase and could be related to metal release from decomposed organic matter, an increase of moisture from 58.4% to 73.5% (due to release of water upon the decomposition of organic matter), or a change of other oxidic and anionic conditions in the medium, so increasing the solubility of metals (Hsu and Lo, 2001; Soumare *et al.*, 2003; Zorpas *et al.*, 2003), although some authors suggest that where the potential toxic metal concentrations of compost are high, the leachability of metals associated with compost is of concern (Hsu and Lo, 2001).

The determination of the total content of heavy metals in the tannery sludge compost showed values significantly lower than those of compost authorized for agricultural use as determined by the Canadian Council of Ministers of the Environment (CCME, 1995), (Table 4), as available recommendation for sludge compost, as well as from the values obtained for other sludge composts (Zorpas *et al.*, 2003).

The results of the metals analysis on the compost at different stages of composting show that the main part of the metal elements, concentrated in the most resistant fractions, are either not, or are weakly, bioavailable to plants, and only a weak proportion represents the unstable fraction easily bioavailable (exchangeable + soluble).

Water and CaCl₂ Extractable Fractions of Organic Carbon

Water extract from compost is the source of the most readily biologically active organic carbon (Chefetz *et al.*, 1998). The water soluble organic carbon (C) level increased from 1.8% in the compost on day 5 to 2.7% on day 20 and then gradually decreased to 1.7% at the end of the process (Figure 3). The increasing phase of the water soluble carbon content during the first 20 days of composting was probably caused by the appearance of soluble organic matter (e.g. sugars, proteins, hemicellulose, organic and

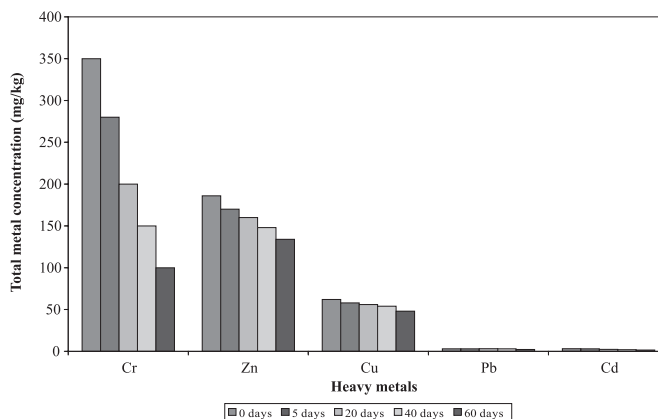


Figure 2. Changes in the total amount of heavy metals during composting of tannery sludge

amino acids) due to microbial-stimulated transformations of compost organic matter. The decomposition and breakdown products were continuously released, resulting in an increase in water-soluble organic carbon. The organic matter was rapidly and easily biologically degraded during the first period. From day 20, the amount of organic carbon in the water extract was decreasing, probably as the result of reducing activity of microbes and the beginning of organic matter humification. A decline in water soluble organic carbon is often used as an indicator of compost maturity.

Organic matter of fresh compost consists of many easily degradable and water soluble substances, whereas mature compost is characterized by rich, and is in stable, decomposition-resistant, high molecular mass humic-like substances which have limited ability to dissolve in water. A similar trend concerning the water soluble organic C during composting was observed in the composting of pig manure (Hsu and Lo, 1999).

Humic substances formed during composting show colloidal properties and may cause aggregation of organic and inorganic

Table 4. Total heavy metal contents in the final compost (60 days) and allowable limits for different classes of compost according to Canadian limits (CCME, 1995)

Heavy metal	Content in final mature compost (mg kg ⁻¹)	Allowable limit (mg kg ⁻¹ dry wt) Class A	Allowable limit (mg kg ⁻¹ dry wt) Class B
Cr	100.00	210.00	1,060.00
Zn	148.00	500.00	1,850.00
Cu	54.00	100.00	757.00
Pb	3.20	150.00	500.00
Cd	1.60	3.00	20.00

Class A compost (which has no restrictions in use), and Class B Compost (which can be used on forest lands and roadsides and for other landscaping purposes).

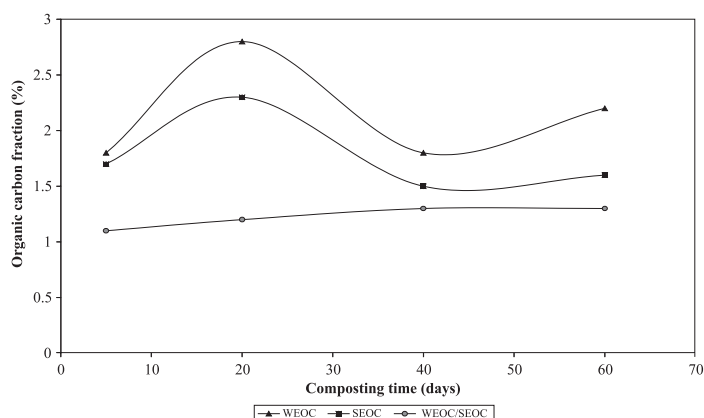


Figure 3. Fractions of soluble organic carbon of tannery sludge compost; water extractable organic carbon (WEOC), solution extractable organic carbon (SEOC)

matter. This is a very desirable feature of organic matter because it influences many physicochemical properties of compost and soils. To investigate the colloidal properties of compost organic matter, a 0.01 M CaCl_2 solution was used instead of water in the same way as in the study on water extractable fractions. The aggregation of humus origin substances takes place in the presence of polyvalent cations like Ca^{+2} . In this study, during the composting time the amount of extractable carbon found in the 0.01 M CaCl_2 solutions was decreasing faster in relation to the amount of carbon found in water extracts (Figure 3).

This implies transformation of low molecular organics to humic-like substances of higher molecular weight during composting. The ratio of water extractable organic carbon and solution extractable organic carbon (WEOC/SEOC) slowly decreased during the maturation time. The aggregation ability of compost organic matter was increasing during the process. The ratio of water extractable carbon to 0.01 M CaCl_2 solution extractable carbon may be treated as another maturity index, reflecting the properties of humic substances formed during composting.

Humic Substances Content

Humic substances constitute the most important fraction of compost organic matter. The sum of humic acid (HA) and fulvic acid (FA) fractions in this study did not change considerably during the composting process (Figure 4). However, a small increasing trend concerning the humic content was observed, which is in agreement with other studies dealing with composting of municipal solid wastes (Chefetz *et al.*, 1996). Mature and well humified compost applied to a poor sandy soil may greatly improve its properties, increasing fertility and crop productivity. Moreover, humic substances strongly interact with pesticides to speed up their biodegradation, and with metal ions influence their transport and plant accessibility. They also have ability to buffer pH and to act as a potential source of nutrients for plants. The levels of humic fractions (HA, FA, HM) in compost represent the degree of maturity.

The HA level gradually increased during the process, from 10% of organic carbon to 13%. The FA level gradually decreased from 13% of organic carbon to 8% of total organic carbon at the end of composting. Although a wide

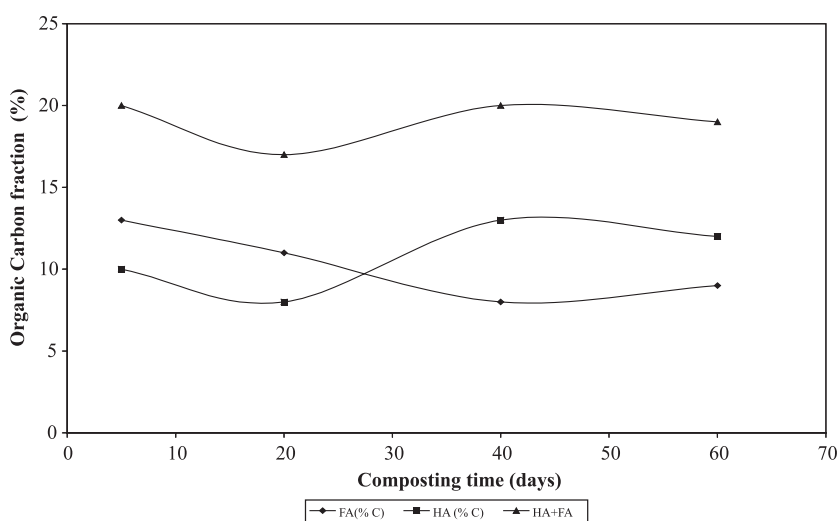


Figure 4. Relative content of fulvic acids (FA), humic acids (HA) and humins (HM) as percentages of compost organic carbon during tannery sludge composting

variation may be observed in the levels of humic fractions, the trends clearly indicate that fulvic acids decrease and humic acids increase during composting. The variations observed in the amounts of humic and fulvic acids as a function of composting time, and especially the substantial decrease of HA during the first 20 days need to be explained. The increasing level of HA represents the degree of humification and maturity of compost (Veeken *et al.*, 2000). In general fresh composts contain low levels of HA and higher levels of FA.

As composting proceeds the HA content increases and the FA level tends to decrease (Drozd *et al.*, 1997). It must be mentioned here that extraction techniques for humic substances are in general non-specific, and non-humic, and easily biodegradable material (lipids, waxes, tannins, cellulose, sugars, and peptides) are isolated during extraction with alkali solutions. Non-humic material precipitates during acidification and precipitation of humic acids (Adani *et al.*, 1995). However, the amount of non-humics decreases during maturation. In this study the humic fraction of fresh compost must have contained a relatively high level of non-humic, easily biodegradable organic matter which decomposed during composting. This influenced the humic acid fraction, lowering its level before day 20. From that day the synthesis of humic acids became the dominant process, and its level started to increase.

Conclusions

The study concludes that the initial concentrations of chromium, cadmium, lead, copper, and zinc all decreased as the composting process progressed. The total concentrations of heavy metals in the compost complied with the compost authorized for agricultural use as determined by the Canadian Council of Ministers of the Environment (CCME, 1995) a available recommendation for sludge compost.

The composting process significantly produced stable and mature compost and the concentration of compost organic matter fractions generally decreased during the

composting time while the C/N ratio exhibited a decreasing pattern throughout the composting process. Water and solution (0.01 M CaCl₂) soluble organic carbon concentrations rapidly increased to a maximum on the 20th day and declined thereafter. The changes in the humic fractions reveal that the amount of humic acids decreased during the first 20 days and increased thereafter. Reverse trends were observed in the case of fulvic acids. The amounts of FA reached the maximum value on day 20 and declined thereafter.

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