

EVALUATION OF THE COMPOSTING PROCESS IN DIGESTED SEWAGE SLUDGE FROM A MUNICIPAL WASTEWATER TREATMENT PLANT IN THE CITY OF SAN MIGUEL DE ALLENDE, CENTRAL MEXICO

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Key words: fecal bacterias, thermophilic phase, humic substances, size exclusion chromatography

ABSTRACT

The purpose of this work was to examine the feasibility of using municipal sewage sludge from San Miguel de Allende city WWTP, located at central Mexico, for compost production. Experimental piles were prepared with an aerobic digested activated sludge from a municipal WWTP dewatered in a filter press, grass was added as a nitrogen source and bulking was increased by mixing with either crushed porous volcanic material called tezontle (SGT) or wooden sticks wastes collected from municipal gardening of green areas (SGW). The specific composition of the compost piles was as follows: 30 % municipal sewage sludge, 60 % grass clippings and 10 % small wooden sticks (compost SGW); 30 % municipal sewage sludge, 60 % grass clippings and 10 % tezontle (compost SGT); 100 % sewage sludge (control). The efficient reduction of fecal bacteria was achieved in both piles yielding the USEPA class A compost, yet better process performance was obtained in SGW. In this pile the thermophilic phase lasted seven days longer and higher temperatures were reached, for which the desired removal of coliforms was achieved at two weeks of composting versus three weeks required in SGT. The analysis of typical spectrophotometric assays used in the compost characterization, confirmed the decomposition of organic matter and generation of humic substances. The fractionation of humic extracts by size exclusion chromatography with spectrophotometric detection indicated the increase of humic substances amount, and also the increase of molecular mass with the time of composting process. Overall, this work demonstrates the feasibility of municipal sewage sludge compost production for soil fertilization and provides new data about the changes of the molecular mass in humic fraction during composting.

Palabras clave: bacterias fecales, fase termofílica, sustancias húmicas, cromatografía de exclusión por tamaño

RESUMEN

El propósito de este trabajo es examinar la viabilidad de usar lodo residual municipal de la PTAR en San Miguel de Allende, ciudad localizada en la parte central de México, con la finalidad de producir composta. Las pilas experimentales contuvieron lodo residual proveniente de una planta de tratamiento de aguas residuales de lodos activados que tuvieron una digestión aerobia y un secado en filtro prensa, el cual fue mezclado con césped de corte empleado como fuente de nitrógeno y un material volcánico poroso llamado tezontle (SGT) o desechos de maleza de las áreas verdes municipales (SGW). Las pilas se prepararon de acuerdo a las siguientes proporciones: 30 % de lodo de la planta de tratamiento, 60 % de pasto y 10 % de tezontle (composta SGT); 30 % de lodo de la planta de tratamiento, 60 % de pasto y 10 % de pequeños troncos (composta SGW); 100 % de lodo de la planta de tratamiento. La reducción eficaz de bacterias fecales se logró en ambas pilas, dando lugar a compostas de Clase A de acuerdo con la clasificación de la USEPA, la pila que obtuvo mayor eficiencia de reducción de bacterias fecales fue la SGW. En esta pila la fase termofílica duró siete días y se alcanzaron las más altas temperaturas, provocando la reducción de bacterias fecales deseadas en un periodo de dos semanas contra tres semanas que requirió la pila SGT. Los análisis típicos espectrofotométricos fueron usados para la caracterización de las compostas, confirmando la descomposición de la materia orgánica y generación de sustancias húmicas. El fraccionamiento de extractos húmicos por cromatografía de exclusión por tamaño con detección espectrofotométrica indicó el incremento de su cantidad total y también del incremento de masa molecular con el tiempo del proceso de compostaje. En conjunto, este trabajo demuestra la viabilidad de la producción de composta a partir de lodos residuales municipales y proporciona datos sobre los cambios de la masa molecular de fracciones húmicas durante el compostaje.

INTRODUCTION

The installation of wastewater treatment plants (WWTP) is a common strategy to treat municipal wastewater, yet an undesirable side effect is the generation of relatively large amounts of sewage sludge that contain important pollutants such as pathogenic microorganisms, heavy metals and potentially toxic organic compounds. Despite obvious health risks imposed by municipal biosolids (McFarland 2000, Metcalf and Eddy 2003), this type of waste is attractive due to the large amount of organic matter and nutrients, allowing it to be used as a soil amendment or a final cover for municipal solid waste landfills, only if the sludge has been treated adequately (Girovich 1996, Grigatti *et al.* 2004).

Composting of sewage sludge has often been performed by mixing the biosolid with easily degradable organic materials, and formed into piles under controlled conditions regarding moisture, pH, oxygenation and carbon/nitrogen ratio, resulting in an aerobic degradation of organic matter. During the thermophilic phase, the temperature inside the piles oscillates between 40 and 60 °C (Epstein 1997, Lemunier *et al.* 2005, Heinonen *et al.* 2006, Bustamante *et al.* 2008, Hachicha *et al.* 2009), phenomena which aides in the destruction of pathogens.

At the end of the composting process, a stabilized organic matter rich in humic substances (HS) is obtained (Spaccini *et al.* 2000, Sullivan and Miller 2001). The term HS refers to a group of heterogeneous and polyelectrolytic organic compounds with a wide range of molecular masses (up to 100 kilodaltons (kDa)), that are considered the main and more stable components of the soil organic matter. HS contribute to soil fertility since they act improving structure, oxygenation and water holding capacity as well as buffer and cationic exchange capacity (Stevenson 1994). The HS can also bind and potentially immobilize important xenobiotics as pesticides (Conte *et al.* 2005) or heavy metals (Tipping 2002, Weng *et al.* 2002, Halim *et al.* 2003, Sadi *et al.* 2002, Wrobel *et al.* 2003).

Based on the pH dependent solubility, HS are classified as humic acids (HA), fulvic acids (FA) and humins (HU) (Stevenson 1994, McCarthy 2001, Sutton and Sposito 2005).

Values of E4/E6 (the ratio of absorbance at 465 and 665 nm), as well as evaluation of functional groups (total acidity, carboxylic and phenolic groups) and fractionation by size exclusion chromatography have been used to compare the maturity and stability of organic matter of diverse origins like peats (Janos 2003, Shirshova *et al.* 2006, Fong and Mohamed 2007), compost from municipal

solid waste (Campitelli *et al.* 2006), sewage sludge (Zbytniewski *et al.* 2002, Réveillé *et al.* 2003), poultry manure, pig slurry, cotton waste, maize straw and sweet sorghum bagasse (Sanchez *et al.* 2003). In regard to compost, the humification process is studied in order to understand and evaluate the transformations of organic matter initially present in the biosolids (Veeken *et al.* 2000). In some previous works on the compost production from sewage sludge and solid waste, HA characteristics have been determined at the beginning and at the end of the process. According to reported results, total acidity of the final product increases together with the amount of phenolic and carboxylic groups as compared to the initial biosolid (Zbytniewski and Buszewskie 2005). At the same time, E4/E6 ratios decrease and the molecular mass of the humic acids increases slightly (Janos and Zatrepáková 2007). However, the modifications of HA characteristics during the composting process has not been studied in detail.

San Miguel de Allende is a touristic city at central México, with about 140 000 habitants. Considerable amounts of municipal wastewater are produced and these are treated in the local WWTP with a flow rate of 70 L/s. The safe disposal of biosolids generated by the WWTP has become an issue to solve; and the aim of this work is to examine the feasibility of utilizing sewage sludge for compost production. Two bulking materials, necessary for compost aeration were tested, both of them abundant and available at negligible cost. The performance of the composting process was evaluated in each case, using typical measurements and assays. Additionally, the evolution of molecular mass in the humic substances fraction was studied by size exclusion chromatography with spectrophotometric detection. Obtained results provided an insight on the humification process and also supply regionally useful data regarding about the possibility of compost production from the municipal sewage sludge.

MATERIALS AND METHODS

Composting of sewage sludge

The experimental system consisted of three windrows (piles) (1m³) prepared with the sewage sludge, grass and two different bulking materials: (T) tezontle, which is a soft porous material of volcanic origin, abundant in the region of San Miguel de Allende and (W) small wooden sticks – also abundant as wastes from municipal green areas gardening. The sewage sludge was obtained from an activated

sludge unit of the municipal WWTP; it was used after receiving an aerobic digestion treatment and thickening on a filter press. The specific composition of the compost piles was as follows: 30 % municipal sewage sludge, 60 % grass clippings and 10 % small wooden sticks (compost SGW); 30 % municipal sewage sludge, 60 % grass clippings and 10 % tezontle (compost SGT); 100 % sewage sludge (control). The piles were maintained for a period of three months mixing the materials every seven days; temperature inside the piles was measured at several sites and the reported values are the average of the three successive measurements.

Biological and chemical analyses

The determination of fecal coliform bacteria (MPN) was accomplished according to the USEPA Method 1681 (Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A-1 medium) using culture-specific media and elevated temperature to isolate and enumerate fecal coliform organisms. Obtained compost with the three piles at different time periods was evaluated for MPN per g dry mass (dm), in terms of USEPA classification of compost (EPA 1994). According to this classification, a class A compost should contains less than 1000 MPN fecal coliforms per g of dm and can be used as a safe soil amendment for food and non food plants; class B compost is a safe soil amendment only for ornamental plants and it cannot contain more than 2×10^6 MPN fecal coliforms per g dm. Samples with a MPN value higher than 2×10^6 fecal coliforms per g dm are classified as class C compost.

The amount of organic matter was assessed by two techniques, first as the water-soluble organic carbon (TOC) and second the total organic matter (OM). For TOC determination, the aliquot of compost was suspended in water (1:10 sample: water proportion) and the mixture was stirred for 2 h, then the sample was centrifuged at 4000 g (Hermle Z 206A centrifuge) for 20 min and filtered through a 0.45 µm cellulose syringe filter. The filtrate was introduced into a reaction chamber, at 680 °C, which is packed with a catalytic oxidizer for carbon oxidation; the generated CO₂ was transported by the air flow to a non dispersive infrared analyzer (Shimadzu TOC-5050aM analyzer). The OM was assessed after ignition of the dry sample at 550 °C for an hour in a furnace (Felisa Fe-361) according to Standard Methods SM 2540G.

The ratio between absorbance measured at 465 nm and 665 nm was assessed in humic acid extracts. To do so, three aliquots of each compost sample (10 g) were extracted with 100 mL of 0.1 M Na₄P₂O₇ in

sealed bottles with stirring for 24 h. The extracts containing humic substances were centrifuged for 20 min at 4000 g, and the supernatants were passed through a 0.45 μm cellulose syringe filter and acidified to pH=2 with 5 M HCl. To accomplish total HA precipitation, the samples were maintained at 4 $^{\circ}\text{C}$ during 24 h and then centrifuged (20 min, 4000 g); the pellets were then washed twice with 0.1 M HCl and with deionized water. The HA were further purified by stirring with a 0.06 M HCl solution for 12 h, centrifuging and washing with deionized water. The HA precipitates were dried at room temperature. An aliquot of each sample (3 mg) was dissolved in 10 mL of NaHCO_3 0.05 M adjusted to pH 8.3 with NaOH 0.02 M. (Chen *et al.* 1977), the absorbance was measured at 465 nm and 665 nm using a spectrophotometer HACH DR/4000 and the value of E4/E6 ratio was calculated.

Total acidity and carboxylic groups in aliquots HA were measured by titration with barium hydroxide and calcium acetate respectively, according to conventional methods described by Schnitzer and Khan (1972). The content of phenolic groups was calculated as the difference between total acidity and carboxyl groups.

Size exclusion chromatography of humic fraction of compost

Humic substances were extracted from compost samples (10 g) with 100 mL of $\text{Na}_4\text{P}_2\text{O}_7$ 0.1 M in sealed bottles with stirring for 24 h. Extracts were centrifuged for 20 min at 4000 g. Fractionation according to the molecular mass was performed using size exclusion liquid chromatography (SEC) with DAD detection, as described by Sadi *et al.* (2002). The column (750 \times 9 mm) was packed with Toyopearl HW-50F gel; the injection volume was 100 μL , the mobile phase was 10 mM sodium pyrophosphate (pH 10.3) at a flow rate of 0.5 mL/min; and DAD detection was carried out in the wavelength range 254 - 600 nm. The column calibration was performed using bovine serum albumin (66 kDa), carbonic anhydrase from erythrocytes (29 kDa), cytochrome c from equine heart (12.4 kDa) and aprotinin from bovine lung (6.5 kDa) ($r^2 = 0.9978$).

RESULTS AND DISCUSSION

In this work, feasibility of using municipal sewage sludge in a composting process is evaluated, performing amendment of the biosolids with additional organic matter and two different bulking materials.

For this purpose, temperature and fecal coliforms were monitored in the two compost piles containing sewage sludge with grass and tezontle (SGT) or wooden sticks (SGW) respectively. A pile containing sewage sludge without any addition was used as a control. Structural changes of organic matter were also characterized.

In **figure 1**, a plot of temperature inside the piles over time is presented; as can be observed similar changes occurred in the SGW and SGT piles over six weeks, while environment temperature was maintained for the control pile.

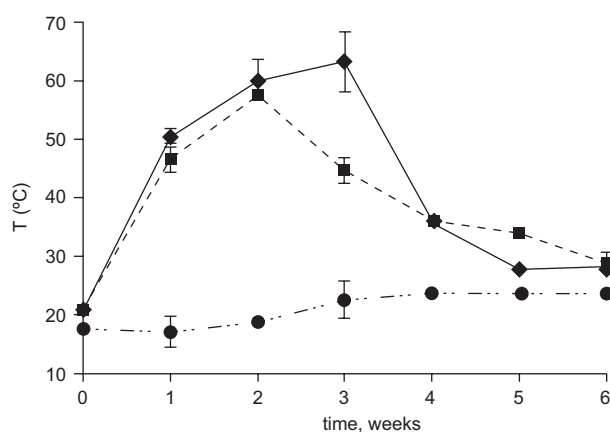


Fig. 1. The evolution of temperature inside the piles during the first six weeks of composting: (—◆—) SGW, (---■---) SGT, (---●---) control pile. Each point is the mean value obtained for three successive days and the respective standard deviation is also marked

The initial increase of temperature indicates microbial activity: in SGW the top value (67 $^{\circ}\text{C}$) was reached at three weeks and in SGT the highest value was 56 $^{\circ}\text{C}$ and was registered after two weeks. Once the thermophilic phase had been completed, the temperature in both piles gradually decreased, reaching steady environment temperature about one week earlier in the SGW as compared to the SGT pile. Obtained results indicate that higher biological activity in SGW compost could be consequence of getting a better oxygenation. The control pile, without additives, did not show any significant variation in temperature.

The microbiological analysis of sewage sludge showed fecal coliforms content higher than 2×10^6 MNP per g, and it was classified as sludge type C according to the USEPA regulations. The high concentration of fecal coliforms makes impossible the direct use of the municipal sewage sludge as soil amendment or fertilizer. The amount of fecal coli-

forms in SGW and SGT piles was monitored during the composting process, and the obtained results are presented in **figure 2**, indicating that both composting processes are quite efficient in the removal of fecal coliforms.

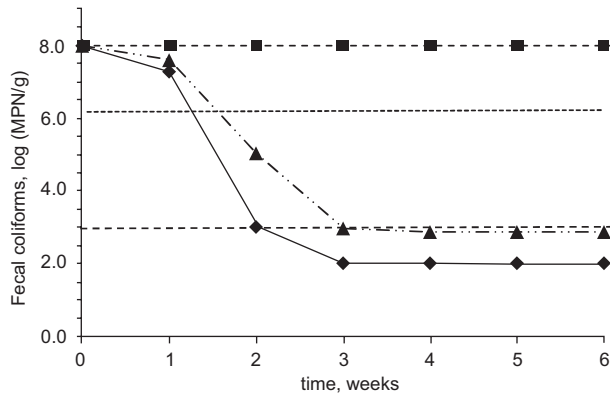


Fig. 2. The most probable number of fecal coliform bacteria (as log(MPN/g), assessed at different time periods of composting: (—◆—) SGW, (—▲—) SGT, (—■—) control pile as compared to EPA regulations of class A and class B compost.

After two weeks of composting, the SGW compost fulfilled the EPA standard for a class A compost (<1000 MPN/g); while the SGT required three weeks to reach the desired MPN level. These results are consistent with temperature profiles registered in the two piles (**Fig. 1**); higher temperature in SGW apparently favored removal of coliform bacteria in a shorter time with respect to SGT, yet in both cases, the final MPN were below 1 % of the initial values.

The pH values in the SGT and SGW composts were monitored over the three months of the experiment and the results obtained are shown in **figure 3**.

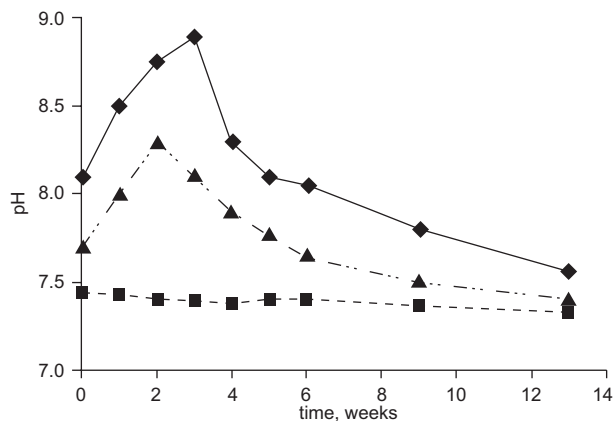


Fig. 3. pH values registered in three piles during composting: (—◆—) SGW, (—▲—) SGT, (—■—) control pile.

At the beginning, a pH raise was observed for the two piles, reaching 8.4 at day 14 in SGT and 8.9 at day 21 in SGW. Afterwards, the pH gradually decreased in both composts to reach neutrality at the end of the composting process. It has been reported by several authors that pH increases during compost formation; apparently this is due to the degradation of short chain fatty acids and the transformation of organic nitrogen to ammonium. The later descent of pH has been attributed to the bacteria aided ammonium oxidation (Michel and Reddy 1998, Sanchez *et al.* 2003).

TOC values obtained in both, SGW and SGT composts (**Fig. 4a**) decreased mainly in the first two weeks of the composting process, which is the time period corresponding to the thermophilic phase (**Fig. 1**).

Our observations support the results, previously reported, concerning to the microorganism assisted decomposition of organic matter and the decrease

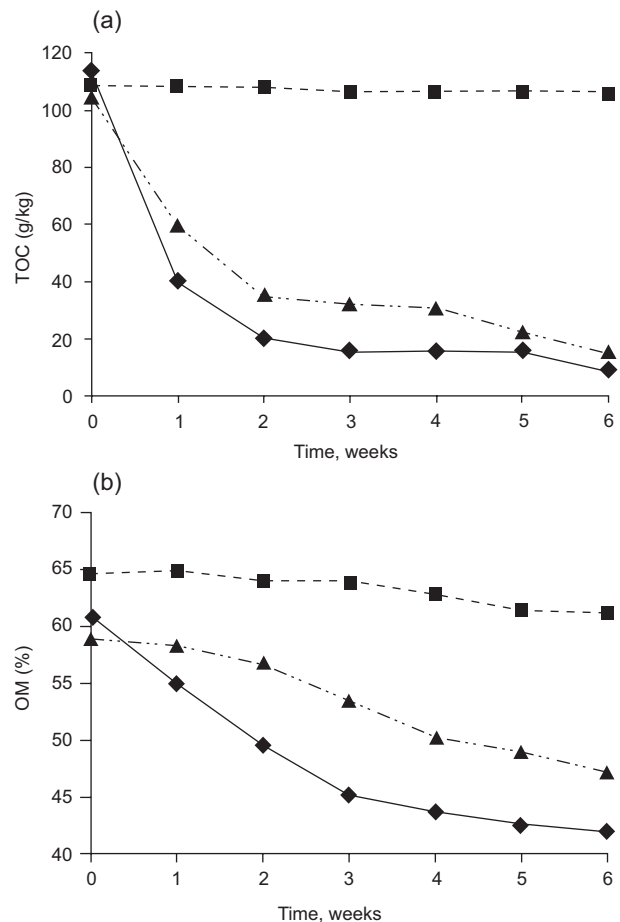


Fig. 4. Water-soluble organic carbon TOCs, g/kg (a) and total organic matter OM, % (b) assayed at different time periods during six weeks of experiment: (—◆—) SGW, (—▲—) SGT, (—■—) control pile.

in TOC levels (Adani *et al.* 1997). At the end of the process, the amount of TOC found in the two composts corresponded to 7 % and 10 % of the initial value for SGW and SGT respectively. As shown in **figure 4b**, the organic matter assay (OM) indicated a slow decrease, going down to 64 % of the initial value in SGW (36 % removal) and to 74 % in SGT (26 % removal).

The measurement results for E4/E6 are presented in **figure 5**; the initial value found in extract from sludge (control pile) was lower than those in SGW and SGT (5.2 versus 5.8 and 5.7 respectively), fact which should be ascribed to the amendment of biosolid with grass and supporting materials for compost production.

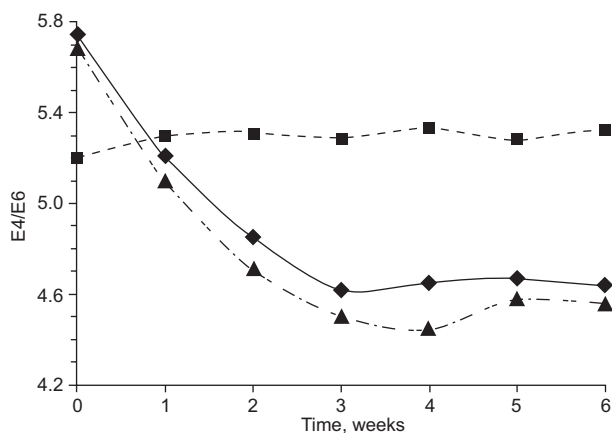


Fig. 5. Profiles of E4/E6 (the ratio between absorbances measured at 465 nm and 665 nm in HA extract) changes in three piles during six weeks of composting: (—◆—) SGW, (---▲---) SGT, (---■---) control pile.

During the experiment, E4/E6 ratio steadily decreased in the extracts from both piles reaching the value of 4.6 in SGW at three weeks and 4.4 in SGT at four weeks. The decrease of E4/E6 ratio is related to the formation and aggregation of humic substances with the shift toward higher molecular mass, in agreement with the proposed models of the formation of humic acids (Sanchez *et al.* 2003).

Total acidity and phenolic groups of the humic acids are shown in **figure 6**. In both SGW and SGT compost, there is an increase in functional groups as the composting process proceeds.

In the case of total acidity, the value changes from 3.41 to 4.85 meq/g in the SGW compost and from 3.28 to 4.45 meq/g in SGT, reaching typical values for this parameter for soil samples (Stevenson 1994). The increment for the phenolic group in composts SGW and SGT was from 1.2 to 2.09 meq/g and from 1.19 to 1.88 meq/g respectively.

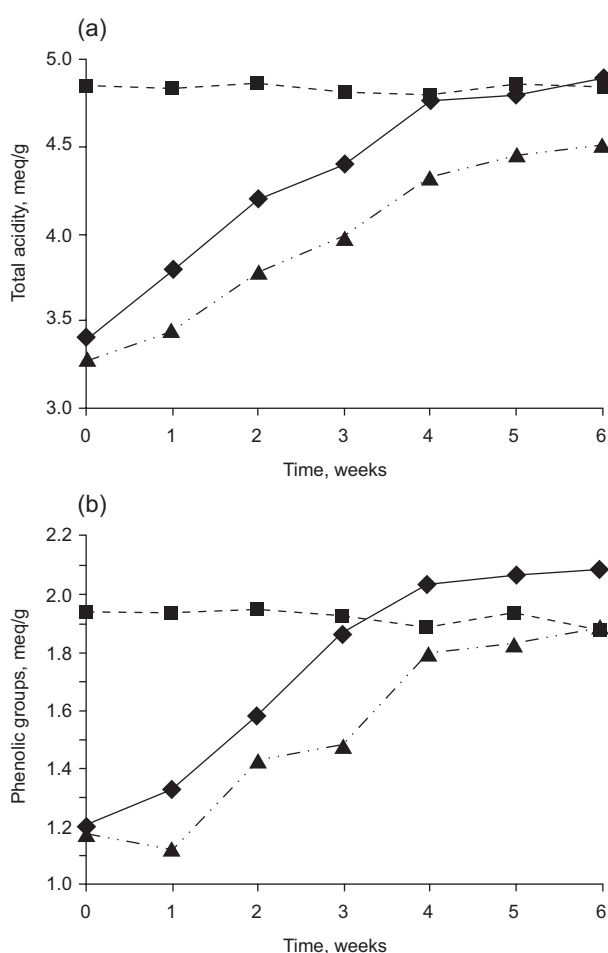


Fig. 6. Total acidity, meq/g (a) and phenolic groups, meq/g (b) evaluated in three piles over six weeks period: (—◆—) SGW, (---▲---) SGT, (---■---) control pile.

In order to get a further insight on the humification process during composting, the compost extracts, obtained at different time periods, were analyzed by size exclusion chromatography. Typical chromatograms of extracts from SGW and SGT piles obtained at two, six and thirteen weeks are presented in **figure 7a** and **7b**.

In general, the elution profiles registered at the wavelength of 500 nm indicate two molecular mass (MM) fractions, in agreement with the results obtained previously in the analysis of municipal waste compost (Wrobel *et al.* 2003). The compounds with MM > 30 kDa eluted in a form of a broad peak with retention times lower than 14 min; while the fraction of compounds with MM < 30 kDa formed a second broad peak with retention time higher than 14 min. For all samples analyzed, the total area under the chromatograms obtained for SGW was always higher, indicating that a more efficient humification process is taking place, in respect to

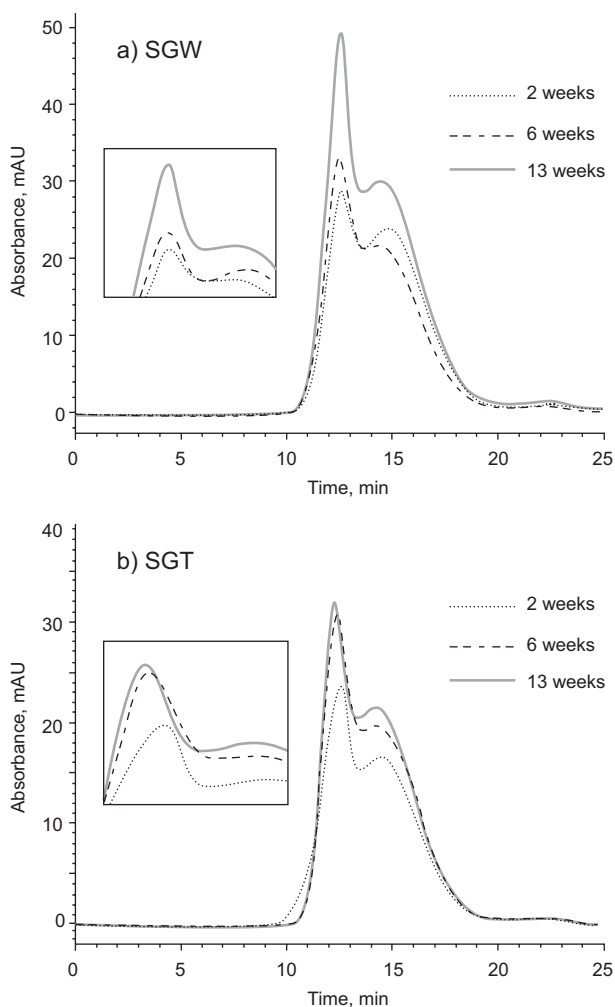


Fig. 7. Typical size exclusion chromatograms (detection at 500 nm) obtained for humic fractions extracted at different time periods of composting: (a) control pile (b) SGW and (c) SGT piles. (two weeks (.....); six weeks (-----); 13 weeks (=====))

SGT. It can also be observed that the increase of chromatographic peaks was more pronounced with the increasing time of composting for SGW (**Fig. 7a**) as compared to SGT (**Fig. 7b**). At the same time, the amount of humic substance extracted from the control pile was negligible and did not increase during the experiment. These facts and also previously presented results support the necessity of a biosolid amendment in the compost production, and indicate that wooden sticks provide better composting conditions with respect to tezontle.

On the other hand, in **figures 7a** and **7b** a shift of the chromatographic signals toward lower retention times can be also observed, which points to the increase of HA molecular mass during composting. For a more clear presentation of this effect, in

figures 8a and **8b** the changes of MM values versus composting time is plotted for SGW and SGT respectively. Since the chromatographic peaks were broad, tentative MM values at the peak maxima were taken for this purpose.

At first it can be noted that more drastic changes of molecular mass occurred in the fraction of MM > 30 kDa (**Fig. 8a**) as compared to MM < 30 kDa (**Fig. 8b**) during the composting process both in, SGW and in SGT. Furthermore, as it is shown in **figure 8a**, for two weeks a similar increase of high MM occurred in the two compost piles (about 37% with respect to the initial value); at longer periods of time a steady, slow increase of MM occurred in SGW, while in SGT

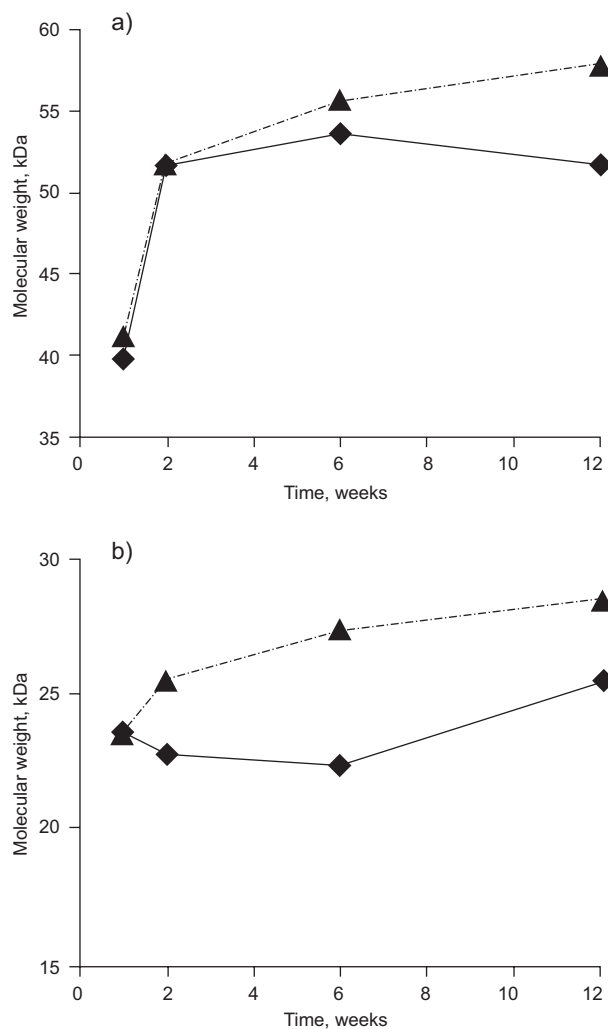


Fig. 8. Changes of the molecular mass of humic extracts during the composting process: (—◆—) SGW, (---▲---) SGT. Tentative MM value corresponding to the mass at the maximum of broad chromatographic peak is presented in each case.—(a) humic substances with MM > 30 kDa; (b) humic substances with MM < 30 kDa

there was a tendency toward lowering MM in this fraction of humic substances.

More pronounced differences between the two composts can be observed for the fraction of compounds with MM < 30 kDa (**Fig. 8b**). In particular, the increase of molecular mass was apparently delayed in SGW as compared to SGT. Furthermore, the profiles of MM evolution in the two HA fractions corresponding to SGW suggest that the aggregation of small HA molecules would contribute to the progress of humification, in agreement with data previously reported (Jouraiphy *et al.* 2005, Amir *et al.* 2006).

CONCLUSIONS

An evaluation of the composting process using municipal sewage sludge from the treatment plant located in San Miguel de Allende, city located at central Mexico was done in this work. Obtained results revealed the necessity of adding sludge amendments for increasing bulking properties; in particular, it can be affirmed that in respect to including tezontle (SGT), a better process performance was obtained using wooden sticks wastes (SGW) as a bulking agent. In the thermophilic phase, higher temperatures were reached in SGW with respect to SGT (about 10 °C difference) and elevated temperatures were maintained for longer time periods (about one week difference). Even though efficient reduction of fecal coliforms was achieved in both systems, a shorter time was needed for SGW to accomplish USEPA class A compost requirements (two weeks in SGW versus three weeks in SGT). The results of spectrophotometric assays typically used in the compost characterization, confirmed the decomposition of organic matter and generation of humic substances, as it is shown by the gradual decrease of TOC, OM and E4/E6 ratio over the time of composting, as well as an increase of total acidity, phenolic and carboxyl groups. The fractionation of humic extracts by size exclusion chromatography indicated the increase of their total amount and also the increase of HA molecular mass with the time of composting process. The results obtained for SGW seems to confirm that the aggregation of smaller molecules contributes to the humification process. Overall, this work demonstrates the feasibility of municipal sewage sludge compost production for soil fertilization use, and it provided new data on the evolution of the molecular mass in HA fraction during composting.

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