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Technical Note

Evolution of the fatty fraction during co-composting of olive oil industry wastes with animal manure: Maturity assessment of the end product

Ridha Hachicha [a], Salma Hachicha [a], Imen Trabelsi [a], Steve Woodward [b], Tahar Mechichi [a,*]

[a] Ecole Nationale d'Ingénieurs de Sfax, Department of Biology, Route de Soukra, BP 1173, 3038 Sfax, Tunisia

[b] University of Aberdeen, School of Biological Sciences, Department of Plant and Soil Science, Cruickshank Building, St. Machar Drive, Aberdeen AB24 3UU, Scotland, UK

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ABSTRACT

Olive mill wastewater sludge, resulting from the natural evaporation of olive oil processing effluent, was co-composted with poultry manure and changes in the lipid fraction investigated. Composting was achieved after approximately 9 months, leading to a compost with high stability and maturity (C/N ratio: 11.9; cation exchange capacity (CEC): 85.9 meq 100 g^{-1} organic matter, CEC/total organic carbon: 4.2 meq g^{-1}; humic acids carbon/fulvic acids carbon: 2.2) useable directly in agriculture and having the same fertilizing capacity as farmyard manure.

Composting led to a reduction in the lipid fraction by at least 95%. Unsaturated fatty acids, particularly polyunsaturated acids, were the most degraded (reduction of 55%) leading to an increase in saturated fatty acids. This change was confirmed by the relative increase in the peroxide index from 5 to 32.5 meq O_2 kg^{-1} fats, and a decrease in the $C_{18:2}/C_{16:0}$ ratio from 0.9 to 0.3. In addition, this study demonstrated that 1.2% of the humic acids component of the compost comprised fatty acids.

1. Introduction

The olive oil industry is very important in Mediterranean countries, in terms of both the economy and tradition. Tunisia is ranked amongst the main world producers with an approximately 6.3% share of world olive oil production (FAO, 2003). The olive oil extraction industry, however, generates large quantities of waste causing serious environmental problems because of high phytotoxicity.

Depending on the extraction system (continuous or discontinuous pressure), in Tunisia olive processing generates approximately 2.6×10^5 t of solid waste annually. This so called 'olive cake' waste contains 4–9% residual oil which is often extracted by hexane in soap factories yielding the by-product 'exhausted olive oil cake' with an average production of 2×10^5 t per year (Hachicha et al., 2003). A second waste substance, olive mill wastewater (OMW), is a dark liquid characterized by high organic load [chemical oxygen demand (COD)] typically ranging from 50 to 150 g L^{-1} (McNamara et al., 2008). OMW also contains high concentrations of phytotoxic and microbially inhibitory compounds such as phenolic compounds and long-chain fatty acids that may affect the physico-chemical and biological properties of a soil if applied directly (Martin et al., 2002; Alburquerque et al., 2006; Hachicha et al., 2008a).

A number of OMW treatment methods have been employed, which may be divided into physico-chemical and biological methods. The physico-chemical treatments are generally very expensive and/or unable completely to solve the waste problem because of a need to dispose of the sludge derived from the process. The biological treatments involve either aerobic or anaerobic systems, which have certain benefits due to the potential utilization of the by-products from these processes.

In Tunisia, at present, OMW treatment is based on evaporation in open-air ponds under natural conditions yielding a sludge residue (OMW sludge), the disposal of which is problematic due to the polluting effects on soil and water. Although this solid waste is rich in readily decomposable organic matter and plant nutrients, and is often used as an organic soil amendment, the presence of excess salts and a high content of lipids and polyphenolic materials may result in negative plant growth effects if applied directly to soil (Paredes et al., 1999; Hachicha et al., 2008a). An environmentally sound and cost-effective solution to olive mill wastes treatment is required.

Composting is an efficient method of waste disposal, enabling recycling of organic matter (Carr et al., 1995; Imbeach, 1988; Greenway and Song, 2002). Several studies have investigated composting as a treatment for OMW (Paredes et al., 2000; Abid and Sayadi, 2006; Hachicha et al., 2008b) and solid cake residues (Hachicha et al., 2003, 2006, 2008b; Ait Baddi et al., 2004).

Co-composting OMW sludge with other organic residues to provide adequate chemical composition, particularly C/N, C/P and N/P ratios, could be a suitable treatment, reducing the phytotoxic

* Corresponding author. Tel.: +216 74 274 088; fax: +216 74 275 615.
E-mail addresses: tahar.mechichi@enis.rnu.tn, tahar98@yahoo.com (T. Mechichi).

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effects of the phenolic and lipid compounds present in the sludge. The resulting compost is a suitable soil amendment, providing beneficial effects in terms of soil properties, particularly in reducing the soil organic matter deficit in arid regions. Various publications report reductions in phenolic and fat content following the composting process (Sayadi et al., 2000; Gea et al., 2007; Hachicha et al., 2008a). Very little data are available, however, on the evolution of the initial lipid fraction from the OMW sludge during the composting process.

The aim of the present work, therefore, was to examine the modifications in the fatty fraction during co-composting of OMW sludge with poultry manure and to evaluate the quantity and quality of fats in the end product.

2. Materials and methods

2.1. Composting materials

Thirty five tons of olive mill wastewater sludge (OMW sludge) collected from an open-air pond was thoroughly mixed with poultry manure (PM), to give the following composition, on a fresh weight basis: 75% OMW sludge and 25% PM.

OMW sludge was characterized by high mineral elements content and high lipid fraction composed mainly by oleic acid (72% dw) and palmitic acid (16.7% dw). PM was rich in nitrogen, with a low C/N ratio: 12. The physico-chemical properties of the raw materials are presented in Table 1.

2.2. Composting procedure

OMW sludge was mixed with PM and the mixture was humidified with further OMW during the thermophilic phase and with water during the maturation period. The volume of effluent, used during the humidification operation was approximately 1.5 L kg^{-1} composted solid materials.

The composting process was held over 9 months. During the biological transformation, moisture was maintained around 50–60% by adding OMW each time the mixture was homogenized and aerated. Generally, the compost pile was turned mechanically twice each week during the thermophilic phase, and once a week throughout the maturation period.

Sampling at random points during pile turning ensured representative results. The subsamples were pooled and mixed into a single sample. These representative samples were homogenized and subdivided into three subsamples, one of which was frozen prior to determination of inorganic nitrogen. A second subsample was oven dried (105 °C for 24 h) to determine moisture content, and the third subsample was air-dried, then ground to less than 0.5 mm prior to analysis. All analyses were performed in duplicate.

Temperatures were measured using a mercury thermometer on a daily basis at different positions in the core of the pile, and the average of all measurements was recorded.

2.3. Analytical methods

Water content was determined by weight loss (drying 100 g compost or waste samples to constant weight at 105 °C). Organic matter (OM) content (20 g dry sample) was assessed by determining loss-on-ignition at 430 °C for 24 h (Navarro et al., 1993). Electrical conductivity (EC) and pH were determined in a 1:10 (w/v) water soluble extract. Total organic carbon (TOC) and total nitrogen (N_T) were measured by automatic microanalysis (Navarro et al., 1991), whereas, NH_4^+ was analysed by steam distillation using MgO. Cation exchange capacity (CEC) was determined using the ammonium acetate method (APHA, 1995).

Fat content was determined by extracting 25 g of sample in a Soxhlet with 200 mL hexane after which the dried organic fraction was weighed. The fatty acids were subjected to trans-esterification after alkaline methanolysis. Fatty acid methyl esters were analysed by gas chromatography (ATI UNICAM 610) and C_{18}/C_{16} ratio was calculated using external standards. The peroxide index was determined using the titrimetric method of Union Internationale de Chimie Pure Appliquée (1979). The analysis of the humic acids (HA) and fulvic acids (FA) content was performed according to Ciavatta et al. (1990): 2 g of compost were treated with 100 mL of a 0.1 N $NaOH/Na_4P_2O_7$ solution. After centrifugation, the supernatant was filtered through a 0.45 μm millipore filter. The HA and FA were fractionated by acidification of 25 mL of the extract with 50% H_2SO_4 (pH < 2), separating the HA (precipitated) from the FA (in solution). Phosphorus was measured colorimetrically at 430 nm as a molybdovanadate phosphoric acid (Kitson and Mellon, 1944). Potassium was determined by flame photometry whereas calcium, magnesium, iron, copper and chromium were measured by atomic absorption spectrometry. Polyphenols were estimated using a modification of the Folin method for quantification (Maestro Durán et al., 1991).

2.4. Statistical analysis

The means and standard deviations of analysis/determination results were calculated using the Statistica software package (version 5.1, StatSoft Inc., Tulsa, OK, USA). All analyses were performed in duplicate.

3. Results and discussion

3.1. Composting process development

Temperatures in the co-composting mixture increased rapidly, reaching 60–70 °C within approximately 60 d (Fig. 1). This high temperature lasted for at least 4 months. These conditions (temperature and time) are sufficient to guarantee compost sanitation in terms of the international requirements (Frazer and Lau, 2000; Hachicha et al., 2008c). After this period, the temperature in the mixture gradually dropped to approximately 30 °C at the end of the biological process.

The long thermophilic period and the high temperature recorded in the co-composting mixture resulting from OMW watering may be attributed to the high decomposable OM content, including fats, in the composting mixture. The high chemical energy content of fats in the compost could be responsible for a long thermophilic period in such composting processes (Gea et al., 2007). According to Lin (2008), maintaining thermophilic conditions assists in decomposing oil-containing wastes. In general olive mill wastes lead to long thermophilic composting processes.

Table 1
Physico-chemical properties of the initial raw materials (dw basis). Data presented are average values with standard deviations.

Samples	pH	Dry matter (%)	Ash (%)	TOC (%)	Total N (%)	C/N ratio	Fats (%)	Polyphenols (%)
OMW sludge	6.1–6.5	38.7 ± 2.1	52.7 ± 1.5	44.7 ± 1.4	1.1 ± 0.1	40.7 ± 1.3	18.4 ± 0.2	1.0 ± 0.1
Poultry manure	4.1–4.4	32.2 ± 1.7	60.2 ± 2.7	22.1 ± 1.3	1.8 ± 0.3	12.4 ± 1.2	3.3 ± 0.2	n.d.
OM Wastewater	4.9–5.1	63.9 ± 9.0	36.3 ± 0.5	33.3 ± 3.5	0.6 ± 0.1	56.4 ± 7.4	7.2 ± 0.6	3.1 ± 0.3

n.d.: not determined; TOC: total organic carbon, dw: dry weight.