



Structural transformation study of water-extractable organic matter during the industrial composting of cattle manure

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ABSTRACT

Various spectroscopic approaches were used to characterize the evolution of water-extractable organic matter (WEOM) during cattle manure composting. Using the results of spectroscopic characterization as bases, multivariate statistical analysis was performed to investigate the correlation among the different parameters related to organic matter transformation. The results show that the composting process degraded lignin, aliphatics, alcohols, and protein-like materials, and increased the amount of water-extractable aromatic compounds and humic-like materials, as well as the humification degree of cattle manure. The degradation process primarily occurred during the first few days of composting, whereas the formation of fulvic- and humic-like substances took place during the later stages. The spectroscopic parameters obtained from the WEOM analysis were divided into two groups: one describes the state of decomposition, and the other reflects the stabilization and maturation of the compost.

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1. Introduction

Recycling organic wastes from agricultural sources by composting is widely applied in China. Particular attention must be paid to the changes that the material undergoes during composting to obtain high quality compost. Various parameters, such as C/N ratios, water-extractable organic matter (WEOM), electrical conductivity, and cation exchange capacity, have been applied to monitor organic matter transformation and evaluate the stability of composted materials [1–3]. Composting is the biochemical transformation of waste organic matter by microorganisms, whose metabolism occurs in the water-soluble phase. Thus, a change in WEOM is a good indication of organic transformation and compost stability [4]. In addition, WEOM can interact with a number of inorganic and organic pollutants because of the presence of oxygen-containing and aromatic functional groups in its constituents, which play significant biochemical and geochemical roles in land systems [5]. Therefore, the structural characterization of WEOM extracted from composted materials is essential for the successful application of composting.

To date, WEOM analysis has mainly focused on conventional parameters, such as dissolved organic carbon (DOC) concentration and

the ratio of DOC to dissolved organic nitrogen [4,6–8]. Little work has been done to identify the compositions and structural characteristics of WEOM extracted from cattle manure during composting.

Spectrometric measurements can provide significant information on the conventional chemical parameters of compost maturity. Indices derived from UV–visible (UV–vis), Fourier transform infrared (FTIR), and fluorescence spectra have proven useful in investigating the structural characteristics of organic matter [8,9]. As a rapid and economical method, UV spectrometric analysis at certain wavelengths, such as 280 nm, obtains data that are positively correlated with aromatic carbon content and molecular weight, and provides information on the molecular structure of WEOM [10]. By contrast, FTIR can provide clues about the functional groups of complex samples [11]. As a non-destructive tool, fluorescence spectroscopy can be used to quantify the humification degree of organic materials [12]. However, using only one method to investigate organic matter composition is insufficient. The integration of various techniques is the best way to characterize organic matter. The use of several techniques to characterize the same sample can generate a large amount of data. Multivariate statistical analysis can be used to handle and analyze these data because it allows for the extraction of more information from data obtained through spectroscopic techniques [13].

The aims of the current work are (1) to investigate the structural transformation characteristics of WEOM extracted from cattle manure in the course of composting by various spectrometric analytical techniques, and (2) use multivariate statistical analysis to examine the data obtained through the spectroscopic methods.

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2. Materials and methods

2.1. Source materials and composting process

Experimental samples were obtained from Zhangjiakou Jinnong Biotech Co., Ltd., Zhangjiakou, China. The company was founded in 4 April, 2008, and its mature compost output is approximately $1 \times 10^5 \text{ t y}^{-1}$. The composted materials were mainly comprised of cattle manures in the company, and the fermentation process was conducted in grooves. The entire composting process lasted approximately 41 days, during which time the composted materials were forked over several times to maintain aeration. We did not control the composting operation or attempt to influence the course of the composting process. During the composting process, triplicate composite samples were collected at different points from the top to the bottom of the piles after 0, 7, 14, 26, and 41 days.

2.2. WEOM extraction and analysis of water-extractable organic carbon (WEOC)

Compost samples were freeze-dried at -54°C , ground, and sieved. Particles smaller than 2 mm in diameter were chosen for analyses. Each sample (20 g) was extracted using 200 mL deionized water (1:10 ratio). Extracts were centrifuged at 12,000 rpm for 15 min and then filtered through a $0.45 \mu\text{m}$ membrane filter. The WEOC concentration in the filtrates was measured using an Analytik Jena Multi N/C 2100 TOC analyzer (Analytik Jena, Jena, Germany). A portion of the filtrates was freeze-dried at -54°C , and the rest were stored in the dark at 4°C in acid-washed, oven-dried amber glass flasks until further analyses.

2.3. FTIR spectra

FTIR spectroscopy was performed using a Nicolet Nexus 670 FTIR spectrophotometer (Nicolet, Madison, USA). KBr pellets were obtained by pressing a mixture of 1 mg WEOM solid sample and 100 mg KBr (spectroscopic grade) under $10,000 \text{ kg cm}^{-2}$ for 2 min. The spectra were obtained in the range of $4000\text{--}400 \text{ cm}^{-1}$ at 2 cm^{-1} resolution, and 64 scans were performed on each acquisition.

2.4. UV-vis spectra

For UV-vis and excitation-emission matrix (EEM) spectroscopy, the WEOC concentrations of all WEOM samples were adjusted to 7 mg L^{-1} , in accordance with the result of the pre-experiment. UV-vis absorption spectra were recorded with a Shimadzu UV-1700 PC spectrophotometer (Shimadzu, Kyoto, Japan) at a wavelength range of 200–400 nm. The reference beam blank was Milli-Q purified water. The specific ultraviolet absorbance at 280 nm (SUVA_{280}) was calculated as the absorbance divided by the WEOC concentration, and the E_{253}/E_{203} ratio was calculated as the ratio of the absorbance at 253 and 203 nm.

2.5. EEM spectra

EEM spectra were obtained using a Perkin-Elmer model LS-50B fluorescence spectrophotometer (Perkin Elmer, NJ, USA). The slit widths for the excitation and emission monochromators were set at 5 nm. The EEM spectra were obtained at a scan speed of 1200 nm min^{-1} over 200–440 nm and 280–520 nm ranges of excitation and emission wavelengths, and the excitation wavelength increment was set at 5 nm. The scattering was regulated using interpolation in the areas affected by the first- and second-order Rayleigh and Raman scattering [14], after which the fluorescence regional integration (FRI) technique was adopted to analyze the EEM spectra [15].

2.6. Multivariate statistical analysis

Principal component analysis and hierarchical cluster analysis were conducted using SPSS version 16.0 (SPSS International, Chicago, USA) to analyze the correlation among the different parameters from various spectrometric techniques. Hierarchical cluster analysis was performed on all the parameters by the between-groups linkage cluster method, which uses the squared Euclidean distance as a similarity measure.

3. Results and discussion

3.1. FTIR spectra

As shown in Fig. 1a, the FTIR spectra of WEOM at different composting durations exhibited similar peak locations. The spectra were characterized by (i) an intense broad band at approximately 3405 cm^{-1} , mainly caused by the O—H stretching vibrations of the hydroxyl groups of alcohols, phenols, and organic acids; (ii) a weak band around 2923 cm^{-1} , assigned to the C—H stretching vibrations of aliphatic structures; (iii) a weak, sharp band at about 2027 cm^{-1} , caused by the C=C stretching vibrations of the C=C=C groups; (iv) a strong absorbance band at around 1645 cm^{-1} , ascribed to the C=C stretching vibrations of aromatic rings; (v) a sharp band at

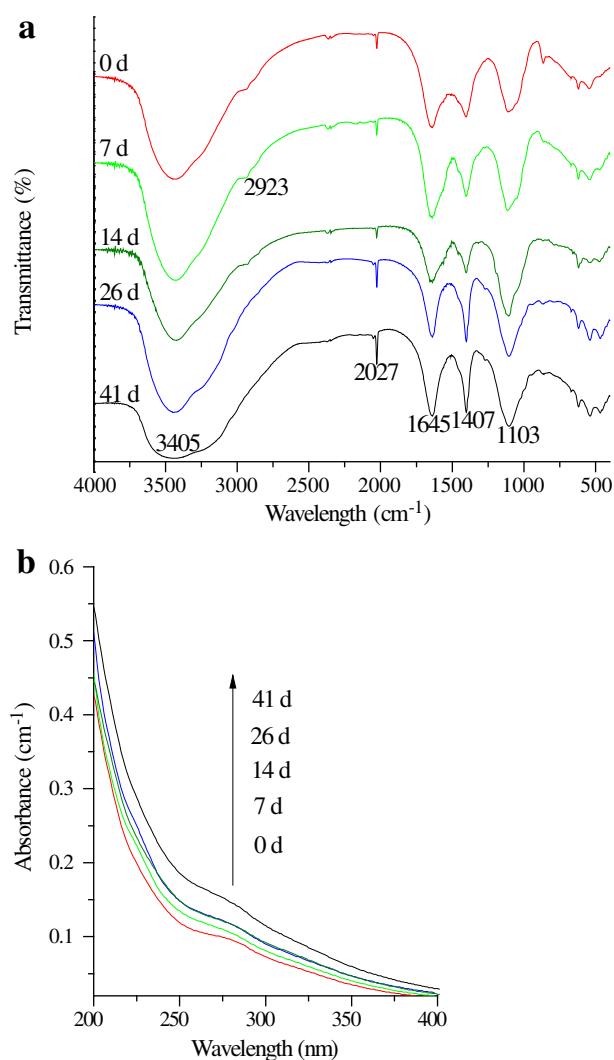


Fig. 1. Evolution of the FTIR spectra (a) and UV-vis spectra (b) of WEOM during cattle manure composting.

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