



Characterization of isolated fractions of dissolved organic matter derived from municipal solid waste compost

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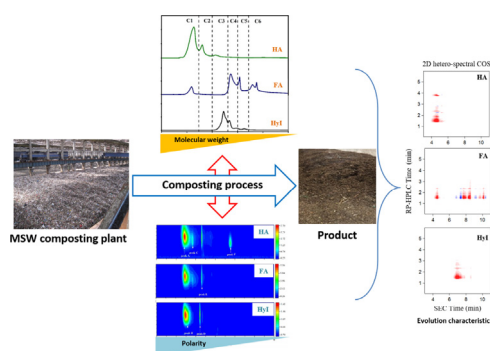
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HIGHLIGHTS

- The MW and polarity property of HA, FA, and Hyl fractions were studied.
- 2D-COS showed different sequential orders of compost-derived DOM fractions.
- For the first time, the evolution of Hyl fraction in composting was studied.
- Correlations between MW and polarity of DOM fractions were studied.

GRAPHICAL ABSTRACT



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ABSTRACT

Understanding the heterogeneous evolution characteristics of dissolved organic matter fractions derived from compost is crucial to exploring the composting biodegradation process and the possible applications of compost products. Herein, two-dimensional correlation spectroscopy integrated with reversed-phase high performance liquid chromatography and size exclusion chromatography were utilized to obtain the molecular weight (MW) and polarity evolution characteristics of humic acid (HA), fulvic acid (FA), and the hydrophilic (Hyl) fractions during composting. The high-MW humic substances and building blocks in the HA fraction degraded faster during composting than polymers, proteins, and organic colloids. Similarly, the low MW acid FA fractions transformed faster than the low weight neutral fractions, followed by building blocks, and finally polymers, proteins, and organic colloids. The evolutions of Hyl fractions during composting occurred first for building blocks, followed by low MW acids, and finally low weight neutrals. With the progress of composting, the hydrophobic properties of the HA and FA fractions were enhanced. The degradation/humification process of the hydrophilic and transphilic components was faster than that of the hydrophobic component. Compared with the FA and Hyl fractions, the HA fraction exhibited a higher MW and increased hydrophobicity.

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1. Introduction

Composting is an environmentally friendly and economically viable biological approach for the treatment of municipal solid waste (MSW) (Wei et al., 2014; Zhao et al., 2017). Organic waste is transformed into

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stable and complex macromolecules during the composting process, and these macromolecules are useful as a nutrient source and for soil amendment in order to reduce groundwater contamination, air pollution, and greenhouse gas emissions (Lashermes et al., 2012; Zhang et al., 2016). An understanding of the characteristics of dissolved organic matter (DOM) evolution during composting is of importance, since most microbial metabolism during MSW composting occurs in solid-liquid interface. Indeed, DOM is considered a good indicator of the stability and maturity of composts (Shao et al., 2009; He et al., 2011; Zhang et al., 2016). In addition, it is viewed as a binding center for organic pollutants, heavy metals, bioactive elements, affects the species, distribution, and environmental fate of toxic matter (He et al., 2015; Zhao et al., 2017). Therefore, studies on the composition and evolution of DOM are necessary to improve composting efficiency as well as to, assess the stability and environmental risks of compost products.

Compost-derived DOM is a heterogeneous mixture substance, with its properties varying significantly during the composting stages (He et al., 2015; Zhao et al., 2016). Previous studies have focused on the characteristics of compost-derived humic acid (HA) or fulvic acid (FA) fractions. (Amir et al., 2008; Vieyra et al., 2009; Zhao et al., 2016; Yuan et al., 2017). However, a less discussed fraction, the hydrophilic (Hyl) fraction, constitutes a substantial proportion of the compost-derived DOM content; therefore, further research is needed to fully elucidate the properties of the Hyl fraction (Ma et al., 2001; He et al., 2006). The physicochemical properties of the HA, FA, and Hyl fractions, including molecular weight (MW) and polarity, appear to affect the transformation, degradation, and metal complexation ability of DOM. It has been suggested that low MW DOM fractions are considered to be reactive, while large MW fractions are refractory (Monreal et al., 2010). Further, the large MW DOM fractions are regarded to have greater bioactivity compared to the low MW counterparts (Amon and Benne, 1994; Kaiser and Benner, 2009). Additionally, the hydrophobic DOM fraction has been shown to be less degradable than the hydrophilic fraction and to play an important role in the binding of heavy metals (He et al., 2015; Yoo et al., 2016). Finally, it has been reported that metal ions have stronger binding affinities toward the hydrophilic DOM fraction than to the hydrophobic fraction (Pernet-Coudrier et al., 2011; Louis et al., 2014; Yoo et al., 2016).

There are various approaches to assessing the heterogeneous properties of DOM fractions. Optical spectroscopy is the most frequently used tool to track the change of in DOM composition during the composting process, including techniques such as spectrophotometric absorbance recorded at specific wavelengths and fluorescence measured in excitation/emission or synchronous scan modes (Vieyra et al., 2009; He et al., 2011; Wei et al., 2014; Zhao et al., 2017). However, absorbance or fluorescence spectra do not reveal the polarity and MW character of DOM components in sufficient detail (He et al., 2011; Yan et al., 2012). Reversed-phase high performance liquid chromatography (RP-HPLC) or size exclusion chromatography (SEC) with tandem detectors have been used to unravel the polarity and MW character of DOM fractions (Li et al., 2013; He et al., 2015; Jokubauskaite et al., 2015). Because of the extreme diversity of DOM constituents, the individual isolate RP-HPLC or SEC chromatographic features often overlap (Yu et al., 2011; Yan et al., 2012). Therefore, using a single technique to assess the highly heterogeneous properties of DOM is insufficient, for which the integration of various techniques is needed to comprehensively characterize the composition and structure of individual DOM fractions (He et al., 2015). Two-dimensional correlation spectroscopy (2D-COS) can resolve issues regarding peak overlap in traditional chromatography by extending the spectra toward a second dimension, enabling the accurate identification of the sequential order of spectral changes (Noda and Ozaki, 2004; Lee and Hur, 2017). In addition, 2D hetero-spectral correlation spectroscopy is often used to investigate the correlation between bands in two different types of chromatography (Yu et al., 2011).

To the best of our knowledge, no studies have been previously conducted to explore the heterogeneous evolution of compost-derived HA,

FA, and Hyl fractions using a combination of 2D-COS with RP-HPLC and SEC (referred to as 2D-CORP-HPLC and 2D-COSEC). Compared with the conventional RP-HPLC and SEC techniques, 2D-CORP-HPLC and 2D-COSEC offer advantages in identifying subtle changes or differences that might occur within a small molecular size range of individual DOM fractions. The objectives of this study were (1) to track changes in MW and polarity within compost-derived HA, FA, Hyl fractions through abovementioned spectroscopic techniques and; (2) to assess the heterogeneous evolution behavior in the three fractions during composting using 2D-COS.

2. Materials and methods

2.1. Composting process and sampling

The composting process was discussed in our previous reports (He et al., 2011, 2014). Briefly, the composting raw materials consisted of MSW collected from a composting plant in Beijing, China, cleared of any visible metal, glass, plastic and stone. The residual MSW were piled for composting for 51 days, wherein, the thermophile stage, during which the piles were turned every 2 days, lasted 21 days, with humidity maintained at 50–65% through the addition of composting leachates. The maturity stage, during which the piles were forked every 7 days, lasted for 23 days. Compost samples (0.5 kg) were collected at 0, 7, 14, 21, and 51 days, in triplicate, from three different depths, and mixed together to ensure a representative sample of the entire pile.

2.2. Extraction and fractionation procedure

Extraction of the HA, FA, and Hyl fractions from the MSW was achieved through alkaline extraction and acidic precipitation. Purification was performed by repeated alkaline extraction and acidic precipitation, water washing and dialysis (Ma et al., 2001; He et al., 2006). Compost samples (30 g) were added into 300 mL of 0.1 M NaOH + Na₄P₂O₇ (solid to water ratio of 1:10, w/v) and shaken for 24 h at room temperature. The extracts were centrifuged at 10,000 rpm for 15 min, and the supernatant was filtered through 0.45 µm membrane filter. The filtrate was acidified to pH 1 with 6 M HCl to precipitate HA. The precipitate (HA) was re-dissolved with 0.1 M NaOH, centrifuged at 10,000 rpm for 15 min and rinsed with Milli-Q water. The supernatant (FA and Hyl) was adjusted to pH 2 and pumped through a cleaned XAD-8 resin column. The portion that passed through the resin column was the Hyl fraction and the retained portion was FA fraction. The absorption resin was eluted by backwashing with 0.1 M NaOH (flow 12 mL min⁻¹). The FA and Hyl fractions were respectively desalted by passing through a hydrogen type saturated cation exchange resin. The HA, FA, and Hyl fractions were freeze-dried and stored in a desiccator containing silica gels.

2.3. UV-Vis and fluorescence spectroscopy

The dissolved organic carbon content was measured using an Analytik Jena Multi N/C 2100 TOC analyzer. Prior to UV-Vis and fluorescence spectral analysis, all samples (HA, FA and Hyl) were diluted to 10 mg L⁻¹. UV-Vis spectroscopy was performed on a UNICO model UV-4802 double beam spectrophotometer in the wavelength range 200–550 nm. Specific ultraviolet absorbance at 254 nm (SUV₂₅₄) was defined as the UV absorbance at 254 nm divided by dissolved organic matter. The E₂₅₀/E₃₆₅ (absorbance at 250 nm and 365 nm, respectively), and E₂₆₅/E₄₆₅ (absorbance at 265 nm and 465 nm, respectively) ratios were calculated. In addition, the slopes of the 275–295 nm (S_{275–295}) and 350–400 nm (S_{350–400}) regions were calculated according to Helms et al. (2008). The spectral slope ratio S_R was defined as the ratio of S_{275–295} to S_{350–400}.

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