Bioresource Technology 135 (2013) 616–621

Contents lists available at SciVerse ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Spectroscopic analysis and biodegradation potential study of dissolved organic matters in sewage sludge treated with high-pressure homogenization

Yuxuan Zhang [a], Panyue Zhang [a,*], Jianbin Guo [b,*], Weifang Ma [a], Lingpeng Xiao [a]

[a] Beijing Key Lab for Source Control Technology of Water Pollution, Beijing Forestry University, Beijing 100083, China
[b] School of Soil and Water Conservation, Beijing Forestry University, Beijing 100083, China

HIGHLIGHTS

- Sludge HPH treatment affected compositional and structural characteristics of DOM.
- Main components of DOM after HPH treatment were proteins, polysaccharides and liquids.
- Protein-like substances was the dominant fluorescent organic matters in DOM.
- Substances with aromatic structure were released after HPH treatment.
- DOM characteristics were responsible for DOM biodegradation potential.

ARTICLE INFO

Article history:
Available online 25 September 2012

Keywords:
Sludge disintegration
Fourier-transform infrared
Fluorescence excitation-emission matrix
Ultraviolet
Biodegradation property

ABSTRACT

The effect of high-pressure homogenization (HPH) treatment on characteristics of dissolved organic matters (DOM) in sewage sludge was investigated. Soluble chemical oxygen demand (SCOD), dissolved organic carbon (DOC), protein and polysaccharide concentration in sludge supernatant significantly increased after HPH treatment. Fourier-transform infrared (FTIR) spectra showed that the main components in the DOM for raw sludge were protein and polysaccharide, while for the treated sludge were protein, polysaccharide and lipid. The spectra of fluorescence excitation-emission matrix (EEM) for DOM showed two protein-like peaks at the excitation/emission wavelengths (Ex/Em) of 225/330–340 nm and 275/310–335 nm. A single broad shoulder representing substances with aromatic structure in range 245–270 nm was found in ultraviolet (UV) spectra of the DOM for the treated sludge. Homogenization pressure and homogenization cycle number affected the content of aromatic carbon per unit DOC. The maximum BOD_5/SCOD ratio of 0.48 was achieved at 60 MPa with a single homogenization cycle.

1. Introduction

Recently, high sewage sludge production from conventional activated sludge process has become a common environmental and cost problem for wastewater treatment plants, so there has been increasing researches focused on sludge reduction and utilization. Sludge disintegration is an efficient pretreatment to improve the reutilization potential of sludge as a resource. Within various sludge disintegration technologies, high-pressure homogenization (HPH) treatment as a mechanical pretreatment method is quite effective in sewage sludge disintegration, which can disrupt floc structure and sludge cell walls, and release extracellular polymeric substances (EPS) and intracellular materials into liquid phase (Paquin, 1999; Wuytack et al., 2002). HPH pretreatment coupled with anaerobic digestion improving biogas production has been reported as a sewage sludge treatment technology (Onyeche et al., 2003; Rai and Rao, 2009; Zhang et al., 2012).

Dissolved organic matters (DOM) are well known to play an important role in anaerobic digestion after sludge disintegration. A large amount of DOM is released from sludge solids into sludge supernatant, which includes different kinds of organic compounds, such as carbohydrates, proteins, organic acids, and some biologically resistant compounds. Compositional and structural characteristics of DOM are particular useful for studying transformation and fate of many sludge components in the following anaerobic digestion processes. Therefore, more studies are in great need to understand the DOM characteristics after HPH sludge disintegration.

Different groups of DOM can affect their bioavailability, toxicity and ultimate fate (Garnier et al., 2005; Hur et al., 2006). Various

* Corresponding authors. Address: College of Environmental Science and Engineering, Beijing Forestry University, Qinghua East Road 35, Haidian District, Beijing 100083, China. Tel.: +86 15001255497; fax: +86 10 62336900 (P. Zhang).
E-mail addresses: zzyyxx0618@163.com (Y. Zhang), panyue_zhang@bjfu.edu.cn (P. Zhang), jianbinguo@bjfu.edu.cn (J. Guo), mpeggy@163.com (W. Ma), xlp1677@gmail.com (L. Xiao).

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http://dx.doi.org/10.1016/j.biortech.2012.09.034

methods have been used to provide definitive compositional and structural information about DOM characteristics at the molecular level. Among these analytical methods, spectroscopic techniques such as Fourier-transform infrared (FTIR), fluorescence excitation-emission matrix (EEM) and ultraviolet (UV) spectroscopies, appear to be the most widely used methods because of their experimental simplicity and nondestructive property (Her et al., 2003; Kalbitz et al., 2000; Kim and Yu, 2005; Li et al., 2005). Although numerous data of spectroscopic analyses on DOM characteristics have been reported (Her et al., 2003; Hur et al., 2006; Santos et al., 2009; Wang et al., 2009), no work has been devoted to the DOM from sewage sludge disintegrated with HPH treatment.

The objective of this study was to investigate the effect of HPH treatment on the compositional and structural characteristics of DOM. The chemical characteristics of sewage sludge were analyzed to evaluate the sludge disintegration using HPH treatment. Spectroscopic analyses such as FTIR, fluorescence EEM and UV spectroscopies were used to study the effect of the HPH treatment on compositional and structural characteristics of the sludge DOM. Furthermore, the DOM biodegradation property was assessed by BOD_5/SCOD ratio.

2. Methods

2.1. Sewage sludge sample and HPH treatment

Sewage sludge was collected from a municipal wastewater treatment plant located in Beijing, China. The sewage sludge was thickened to a total solid content (TS) of 14.78 g/L by gravity settling for 24 h as the experimental sludge sample. The sludge sample was stored at 4 °C before use. The HPH apparatus was a GJJ-0.03/100 high-pressure homogenizer (Puzong Inc., China). The raw sludge samples were firstly treated with a single homogenization cycle at six homogenization pressures (*P*): 20 MPa, 30 MPa, 40 MPa, 60 MPa and 80 MPa. In order to discuss the effect of homogenization cycle number (*N*), one to three homogenization cycles were chosen at a homogenization pressure of 60 MPa.

2.2. Analytical methods

For raw and treated sewage sludge samples, total solid content (TS), volatile solid content (VS), total chemical oxygen demand (TCOD), soluble chemical oxygen demand (SCOD), and volatile fatty acids (VFA) were determined according to APHA standard methods (Eaton et al., 2005). The pH was measured by means of WTW pH-meter 537. Dissolved organic carbon (DOC) determination was carried out via a high temperature combustion analyzer (multi N/C 3100 TOC, Analytik Jena AG, Germany). Biochemical oxygen demand after 5 days (BOD_5) was detected using OxiTop respirometer systems by 5-day BOD test. Protein concentration was determined by the coomassie brilliant blue G-250 method at an absorbance of 595 nm. Polysaccharide concentration was measured using the anthrone method at an absorbance of 630 nm.

2.3. Spectroscopic analysis of DOM

After HPH treatment, both raw and treated sewage samples were centrifuged at 8000 rpm for 10 min. Centrifuged supernatant was filtrated through a 0.45 μm filter membrane. Part of the filtrate was freeze-dried to obtain the DOM powder for FTIR analysis. The rest of filtrate was diluted 50 times with Milli-Q water for fluorescence EEM and UV analyses.

The DOM power mixing with KBr was used for FTIR analysis. FTIR spectra were performed on a FTIR spectrometer (Spectrum 100D, PerkinElmer, USA) in the 4000–400 1/cm range scans, resolution ±1 1/cm.

The spectra of fluorescence EEM were measured using a fluorescence spectrophotometer (F-7000, HITACHI, Japan). The spectra of fluorescence EEM were collected by scanning and recording emission spectra ranging from 200 to 500 nm at 5 nm steps, with 5 nm increments of excitation wavelengths range of 200–400 nm. The slits for excitation and emission were set to 5 nm and the scan speed was 1200 nm/min. The software origin 8.0 was employed to process the EEM data.

The UV spectra were measured with an EVOLUTION 3000 UV–visible spectrophotometer (Thermo Fisher Scientific Inc, USA). The spectra were obtained over the wavelength range of 190–400 nm.

3. Results and discussion

3.1. Effect of HPH treatment on chemical characteristics of sewage sludge

The chemical characteristics of sewage sludge before and after HPH are displayed in Table 1. After HPH treatment, pH showed a slight reduction, TS, VS and TCOD almost had no changes, indicating that there hardly were chemical reactions during HPH process. However, SCOD, DOC, protein and polysaccharide concentrations presented a significant increase, which indicated that HPH treatment was beneficial to the release of organic matters such as proteins and polysaccharides from sludge solids into sludge supernatant due to the disruption of floc structure and sludge cell walls.

Table 1 also shows that the effect of homogenization pressure and homogenization cycle number on various chemical characteristics of sewage sludge. The pH decreased with the increase of homogenization pressure or homogenization cycle number, which might result from the release of acidic materials during HPH treatment, for example, the VFA in sludge supernatant increased by about 300 mg/L (as CH_3COOH) after HPH treatment at 80 MPa with a single homogenization cycle. TS, VS and TCOD were not affected by the change of HPH operating conditions. SCOD, DOC, protein and polysaccharide concentrations increased about three times with the homogenization pressure increasing from 20 MPa to 80 MPa, and increased by 30–40% with three homogenization cycles instead of a single homogenization cycle, indicating that the increase in homogenization pressure or homogenization number cycle was beneficial to the release of organic matters from sludge solids into sludge supernatant.

3.2. Spectroscopic analyses of DOM

3.2.1. FTIR spectra

The FTIR analysis was performed to determine components of DOM. All spectra are gathered in Fig. S1 (Supporting Information (SI)). FTIR spectra of DOM from raw and treated sludge samples showed similar vibrational bands, indicating that similar components existed in DOM of sludge samples before and after HPH treatment. Major spectral bands of DOM in raw sludge were assigned as follows: 3400 1/cm attributed to intermolecular O–H stretching, 1645 1/cm due to amide C═O stretching in protein components, 1547 1/cm suggested by amide N–H stretching in protein components, 1120 1/cm from carbohydrate C–C or C–O stretching in polysaccharide components (Chen et al., 2002; Minor and Stephens, 2008; Rai and Rao, 2009; Reddy et al., 2007; Santos et al., 2009). The results indicated that the main components of DOM in raw sludge were protein and polysaccharide substances.

Besides above spectral bands, a distinctive band in FTIR spectra of DOM after HPH treatment appeared at 2928 1/cm, which was