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Q1 Molecular characterization of effluent organic matter in 2 secondary effluent and reclaimed water: Comparison to 3 natural organic matter in source water

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A B S T R A C T

Municipal wastewater reclamation is becoming of increasing importance in the world to 18
 solve the problem of water scarcity. A better understanding of the molecular composition of 19
 effluent organic matter (EfOM) in the treated effluents of municipal wastewater treatment 20
 plants (WWTPs) is crucial for ensuring the safety of water reuse. In this study, the molecular 21
 composition of EfOM in the secondary effluent of a WWTP in Beijing and the reclaimed 22
 water further treated with a coagulation–sedimentation–ozonation process were charac- 23
 terized using a non-target Fourier transform ion cyclotron resonance mass spectrometry 24
 (FT-ICR MS) method and compared to that of natural organic matter (NOM) in the local 25
 source water from a reservoir. It was found that the molecular composition of EfOM in the 26
 secondary effluent and reclaimed water was dominated by CHOS formulas, while NOM in 27
 the source water was dominated by CHO formulas. The CHO formulas of the three samples 28
 had similar origins. Anthropogenic surfactants were responsible for the CHOS formulas in 29
 EfOM of the secondary effluent and were not well removed by the coagulation-sedimentation- 30
 ozonation treatment process adopted. 31

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47 Introduction

48 Municipal wastewater reclamation is becoming of increasing
 49 importance in the world to solve the ever-growing water scarcity
 50 problem (Asano et al., 2007). In comparison with tap water,
 51 however, the treated effluents and reclaimed water contain
 52 much higher concentrations of organic matter, and what these
 53 organic matters are remains a big question (Fatta-Kassinos et al.,
 54 2011). The organic matter in the treated effluents (EfOM) is
 55 considered to be a heterogeneous mixture of complex organic
 56 matter, including natural organic matter (NOM) originating from

drinking water, soluble microbial products (SMPs) from acti- 57
 vated sludge, and anthropogenic compounds, including phar- 58
 maceuticals and endocrine disruptors with industrial and 59
 residential origins (Barker and Stuckey, 1999; Shon et al., 60
 2006a). It may also negatively affect the performance of the 61
 reclamation treatment process. For example, EfOM may con- 62
 tribute significantly to membrane fouling (Xu et al., 2010; Zheng 63
 et al., 2014) and impact the advanced oxidation process as 64
 it directly reacts with ozone and hydroxyl radicals (Gonzalesa 65
 et al., 2011; Audenaert et al., 2013). Furthermore, EfOM 66
 was proved to be an important precursor for disinfection 67

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by-products (DBPs) during wastewater chlorination (Krasner et al., 2009; Gerrity et al., 2015). To control these EfOM related problems, a better understanding of EfOM properties is required.

In practice, EfOM is usually measured in terms of surrogate parameters, such as chemical oxygen demand (COD), biochemical oxygen demand (BOD), and total or dissolved organic carbon (TOC or DOC) (Michael-Kordatou et al., 2015). These parameters only provide a general quantification of EfOM, and cannot provide the composition and structural information of EfOM. Various analytical methods have been used to characterize the composition and structure of EfOM, such as elemental analysis (EA), Fourier-transform infrared (FT-IR) spectroscopy, ultraviolet/visible (UV/vis) spectroscopy, fluorescence excitation/emission matrix (EEM) spectroscopy, and nuclear magnetic resonance (NMR) (Dignac et al., 2001; Ma et al., 2001; Shon et al., 2006b; Zheng et al., 2014; Michael-Kordatou et al., 2015). However, due to the extremely complex nature of EfOM, these methods are not able to provide detailed composition information for EfOM at the molecular level. Recently, Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR MS) has been employed in characterization of NOM from different water environments (Stenson et al., 2003; Koch et al., 2005; Hertkorn et al., 2008; Kim et al., 2006; Sleighter and Hatcher, 2008). The ultrahigh resolution and mass accuracy of FT-ICR MS combined with electrospray ionization (ESI) allows the determination of unambiguous and exact molecular formulas (Stenson et al., 2003; Kim et al., 2006; Hertkorn et al., 2008). Recently, Gonsior et al. (2014) demonstrated the versatility of FT-ICR MS as a technique for characterizing the molecular composition of EfOM. However, our knowledge of the molecular composition of EfOM, especially EfOM in reclaimed water, is still very incomplete.

The main objective of this study was to characterize and compare the detailed molecular compositions of EfOM in the secondary effluent from a municipal wastewater treatment plant (WWTP) and the reclaimed water treated with a coagulation-sedimentation-ozonation process. In addition, the molecular compositions of the two samples were compared to the molecular composition of NOM in a drinking water reservoir in Beijing to evaluate the differences between EfOM and NOM. The results of this study are helpful for the management of reclaimed water quality.

1. Material and methods

1.1. Sample collection and pretreatment

Secondary effluent and reclaimed water samples were collected from a WWTP in Beijing, China. The treatment capacity of this WWTP is 200,000 m³/day. The wastewater is treated by the Carrousel oxidation ditch process. Part of the secondary effluent is further treated at an advanced reclaimed water treatment facility. The advanced treatment processes consist of ozonation, coagulation-flocculation and sedimentation, rapid sand filtration and UV disinfection. The secondary effluent water is ozonated by mixing ozone and water from opposite directions in a large tank, and the process is repeated in a second tank. Each tank is 2.4 m in width and 7 m long, with a water depth of 5 m. The optimal concentration of

ozone is 0.5 mg/mg DOC and the contact time is 10 min. For coagulation-flocculation and sedimentation, a given volume of stock polyaluminum chloride solution (with 15 mg/L liquid form containing 10% of Al₂O₃) was added to the ozonation effluent water. After slowly stirring for 16 min, the mixed liquid flows into a sedimentation tank and the supernatant flows into a rapid sand filter. Rapid sand filtration consists of 12 parallel single-medium filter units (6.0 m × 5.0 m). Typically, sand (particle size 0.9 mm–1.3 mm) is used as the filtering material in the filters. The filtration rate and period between backwashes were 15 cm/min and 24 hr, respectively. The final treatment unit of advanced treatment processes was UV disinfection with UV dosage 400 J/m². The advanced reclaimed water treatment facility has a treatment capacity of 60,000 m³/day. The source water sample was collected from the Miyun Reservoir, which is the largest reservoir in north China and is the main drinking water source for Beijing City. Samples were collected in pre-cleaned glass bottles and delivered under cooled conditions within 4 hr to the laboratory. Once arrived, the samples were filtered through 0.45 μm Supor filter membranes (Pall, USA) and extracted using solid phase extraction (SPE) immediately.

Procedures for water sample pretreatment were carried out according to previous studies (Zhang et al., 2012a, 2012b). Briefly, 500 mL secondary effluent water, 500 mL reclaimed water, and 1000 mL source water samples were acidified with hydrochloric acid (p.a. grade, Merck, Germany) to pH 2 and pumped through Sep-pak C18 solid phase extraction cartridges (1 g, 6 mL, Waters, USA) at a flow rate of 5 mL/min. The SPE cartridges were activated and conditioned with methanol and acidified ultrapure water. For complete removal of inorganic ions, the cartridge was rinsed with 10 mL acidified ultrapure water before elution. Immediately after extraction, the cartridges were eluted with 20 mL LC-MS grade methanol (Merck, Germany). The eluted samples were freeze-dried and stored at -18 °C in the dark. The DOC recovery percentage of SPE was 65%.

1.2. FT-ICR MS analysis

The ultra-high resolution mass spectrometry analyses were performed using a Bruker Apex Ultra 9.4T FT-ICR MS interfaced with negative ion mode electrospray ionization (ESI-). The sample extract was dissolved in methanol and injected into the electrospray source at 180 μL/hr using a syringe pump. The operating conditions for negative ion formation consisted of a 4.0 kV emitter voltage, 4.5 kV capillary column introduce voltage, and -320 V capillary column end voltage. The mass range was set at *m/z* 150–800. The data size was set to 4 M words, and 256 scans were acquired for each spectrum.

1.3. Mass calibration and data analysis.

The FT-ICR mass spectra were externally calibrated for a mass range of 150–800 using a sodium formate aqueous solution, and internally recalibrated with a known homologous series (Zhang et al., 2012a, 2012b). After internal calibration, mass accuracy of <1 ppm was achieved. The *m/z* values between 200 and 600 Da with relative abundance greater than 6 times the standard deviation of the baseline noise value were exported

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