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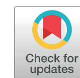
Tracking variations of fluorescent dissolved organic matter during wastewater treatment by accumulative fluorescence emission spectroscopy combined with principal component, second derivative and canonical correlation analyses



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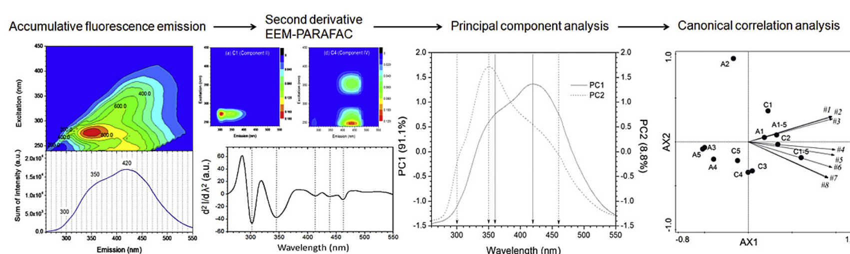
HIGHLIGHTS

- Define AFE spectroscopy compared with synchronous spectroscopy to characterize DOM fractions.
- Employ AFE spectroscopy coupled with PCA to reveal DOM variations.
- Develop second derivative AFE approach compared with EEM-PARAFAC to track components and contents of DOM.

GRAPHICAL ABSTRACT



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ABSTRACT

Accumulative fluorescence emission (AFE) spectroscopy combined with principal component analysis (PCA), second derivative and canonical correlation analysis (CCA) was firstly developed into an available tool to track variations in dissolved organic matter (DOM) fractions and contents during wastewater treatment. Samples were collected from a wastewater treatment plant with a traditional anaerobic/anoxic/oxic (A2O) process. The AFE spectroscopy deduced from the sum of intensities along the excitation wavelengths of fluorescence excitation emission matrix (EEM), could distinctly track tyrosine-like, tryptophan-like, fulvic-like substances. The AFE spectroscopy with the PCA not only disaggregated DOM fractions into the tyrosine-like, tryptophan-like, microbial humic-like, fulvic-like and humic-like substances, but discriminated DOM fractions from the physical sedimentation, anaerobic/anoxic and oxic processes. Absolute areas of fluorescence components obtained by the second derivative AFF spectra had positive liner correlations with Fmax of the relevant components modeling from EEM-PARAFAC, especially the tryptophan-like ($R^2 = 0.95$, $p < 0.01$) and tyrosine-like ($R^2 = 0.83$, $p < 0.01$) substances. The CCA of the sites presented that the potential factors contained the tryptophan-like and tyrosine-like substances. This indirectly proved that the tryptophan-like and tyrosine-like substances were the dominant components of fluorescent DOM, which were further removed in A2O than the other fluorescent components. The CCA of the fluorescent components exhibited that the potential factors included the sites #1 to #6, which were located in the original wastewater, sand setting, primary sedimentation, anaerobic,

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anoxic, facultative units. This elaborated that the fluorescent components were mainly degraded in the physical sedimentation, anaerobic and anoxic processes.

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1. Introduction

Dissolved organic matter (DOM) in wastewater mainly consists of proteins, polysaccharides and humic substances, which are biologically originated compounds (Xia et al., 2009; Maqbool and Hur, 2016; Wagner et al., 2016). The heterogeneous mixture of DOM constantly represents a challenge to all wastewater treatment performances, whose component and concentration dominate membrane fouling, coagulation efficiencies, microbial activities, disinfection by-products, oxidant requirements, contaminant mobility and transport (Henderson et al., 2009; Ishii and Boyer, 2012; Wang et al., 2012; Zhou and Meng, 2016). Potential methods are demanded to track variations of DOM fractions and contents in the wastewater treatment plant (WWTP), which are conducive to promptly modifying the operation parameters (Dahm et al., 2013; Yu et al., 2013, 2014; Yang et al., 2015).

Fluorescence excitation emission matrix (EEM) spectroscopy has been regularly applied to characterize DOM in the WWTP, which may indicate protein-like, fulvic-like and humic-like substances by a peak-method (Hudson et al., 2007). Multivariate data analyses, such as regional integration, parallel factor analysis (PARAFAC) and self-organizing map, have been widely used to quantitatively interpret EEM (Chen et al., 2003; Yu et al., 2014; Maqbool et al., 2016). The PARAFAC has been thoroughly employed to disaggregate the complex EEM data into different fluorescent components for both quantitative and qualitative evaluation (Stedmon and Bro, 2008; Osburn et al., 2012; He et al., 2014; Wei et al., 2016). Furthermore synchronous fluorescence spectroscopy coupled with second derivative has been carried out to assess removal efficiencies of DOM fractions from the wastewater in the WWTP (Yu et al., 2013).

Compared with broad fluorescence emission spectroscopy obtained in conventional fluorescence measurement, accumulative fluorescence emission (AFE) spectroscopy obtained from the EEM spectroscopy can provide a narrow and sharp spectrum. The AFE spectroscopy is formed by the sums of the intensities along the excitation wavelengths in the EEM spectroscopy, which has been applied to monitor the evolution of extra virgin olive oils (Hernández-Sánchez et al., 2017). However its selectivity is restricted by extensive spectral overlaps. Derivatives can amplify narrow band and avoid broad band, with which the AFE spectroscopy can reduce the extensive spectroscopic overlaps and reject matrix interferences.

The aim of this study is to (i) deduce the AFE spectroscopy from the EEM spectroscopy of DOM from wastewater in the WWTP, (ii) track the fluorescent components and investigate their variations in different performance units using principal component analysis (PCA), (iii) calculate abundances of the fluorescent components by the second derivative AFE spectroscopy and verify their feasibilities with the corresponding components from the EEM-PARAFAC, and (iv) seek latent factors of the wastewater treatment and DOM degradation using canonical correlation analysis (CCA).

2. Materials and methods

2.1. Sample collection

This study was conducted in a large-scale WWTP located in Beijing, China, whose treatment capacity is approximately 1million m^3 d^{-1}. A traditional anaerobic/anoxic/oxic (A2O) process is employed in the WWTP for simultaneous removal of nitrogen, phosphorus and carbon (Yu et al. 214). Wastewater samples were collected from the corresponding effluent outflows of the different units, i.e. the original wastewater (#1), the sand setting (#2), the primary sedimentation (#3), the anaerobic (#4), the anoxic (#5), the facultative (#6), the oxic (#7) and the secondary sedimentation (#8). In a given performance unit, quadruplicate wastewater samples were collected with a Wildco Kemmerer 1.2 L sampler, and simultaneously transferred into an EE BOD bottle (Yu et al., 2014). These samples passed through glass fiber filters (Whatman GF/F, 0.45 μm, pre-combusted at 450 °Cfor 4 h). The filtrates were poured into precombusted glass amber bottles respectively, and darkly kept at 4 °C until analyzed.

2.2. Fluorescence spectra measurement

Fluorescence spectra were acquired using a Hitachi fluorescence spectrophotometer (F-7000) equipped with a 150 W Xenon lamp. The slit widths were fixed at 5 nm for both excitation and emission wavelengths, and PMT Voltage to 700 V. The spectrophotometer was calibrated against the Raman signal of the instrument and standardized in quinine sulfate units (ppb QSU) (Osburn et al., 2012). The synchronous spectroscopy was measured by scanning simultaneously both excitation (ex), varied from 260 to 550 nm, and emission (em) wavelengths at 5 nm intervals, while keeping a constant wavelength difference $\Delta\lambda = \lambda_{em}\text{-}\lambda_{em} = 18$, 30 or 55 nm (Kalbitz et al., 1999; Hur et al., 2009; Yu et al., 2013). To obtain the EEM spectroscopy, excitation and emission wavelengths ranged from 240 nm to 450 nm (5 nm increments), and from 260 nm to 550 nm (5 nm increments), respectively. Two following steps were employed to adjust the EEM data. Firstly the excitation and emission data were corrected for instrument-specific response to reduce innerfiltering effects (Stedmon and Bro, 2008). Secondly the spectra were subtracted from the spectra of a blank solution (Milli-Qwater) to delete water Raman scatter peaks (Murphy et al., 2011).

2.3. AFE spectroscopy definition

Fluorescence intensities are presented in the EEM spectroscopy as a function of excitation and emission wavelengths, where the signal intensity at a given pixel matches a pair of excitation and emission wavelengths. The sums of the intensities along the wavelengths axes can be calculated, which are be used to identify the relevant bands. The sums of the intensities along the excitation wavelengths may form an emission spectrum (Hernández-Sánchez et al., 2017), which is defined as an AFE spectroscopy.

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