



Estimation of different source contributions to sediment organic matter in an agricultural-forested watershed using end member mixing analyses based on stable isotope ratios and fluorescence spectroscopy

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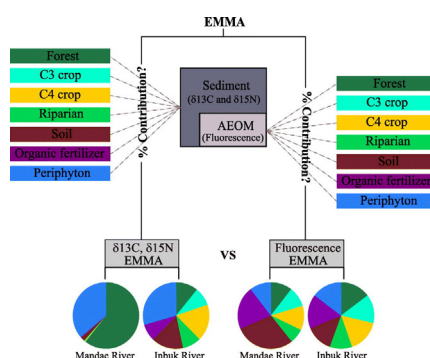
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HIGHLIGHTS

- Different sources samples were characterized using fluorescence and stable isotopes.
- Relative source contributions of SeOM were estimated by EMMA.
- The results of EMMA were compared with isotope versus fluorescence parameters.
- Inability of AEOM of sediments to represent original bulk POM was highlighted.

GRAPHICAL ABSTRACT



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ABSTRACT

The two popular source tracing tools of stable isotope ratios ($\delta^{13}\text{C}$ and $\delta^{15}\text{N}$) and fluorescence spectroscopy were used to estimate the relative source contributions to sediment organic matter (SeOM) at five different river sites in an agricultural-forested watershed (Soyang Lake watershed), and their capabilities for the source assignment were compared. Bulk sediments were used for the stable isotopes, while alkaline extractable organic matter (AEOM) from sediments was used to obtain fluorescent indices for SeOM. Several source discrimination indices were fully compiled for a range of the SeOM sources distributed in the catchments of the watershed, which included soils, forest leaves, crop (C3 and C4) and riparian plants, periphyton, and organic fertilizers. The relative source contributions to the river sediment samples were estimated via end member mixing analysis (EMMA) based on several selected discrimination indices. The EMMA based on the isotopes demonstrated that all sediments were characterized by a medium to a high contribution of periphyton ranging from ~30% to 70% except for one site heavily affected by forest and agricultural fields with relatively high contributions of terrestrial materials. The EMMA based on fluorescence parameters, however, did not show similar results with low contributions from forest leaf and periphyton. The characteristics of the studied watershed were more consistent with the source contributions determined by the isotope ratios. The discrepancy in the EMMA capability for source

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assignments between the two analytical tools can be explained by the limited analytical window of fluorescence spectroscopy for non-fluorescent dissolved organic matter (FDOM) and the inability of AEOM to represent original bulk particulate organic matter (POM).

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1. Introduction

Sediments operate as sources and sinks of nutrients and pollutants in aquatic ecosystems. They store a large amount of organic carbon, acting as a dominant site for organic matter (OM) breakdown and nutrient regeneration (Burone et al., 2003; Ruddy, 1997). They are considered as an archive of past environmental conditions and biogeochemical processes in the surrounding areas (Torres et al., 2012). Sources of OM in aquatic sediments are multiple and diverse. The two major classified sources are (i) allochthonous sources such as materials from land (e.g., plants or soils) and/or upper catchment ecosystems, including also sources from anthropogenic activities (e.g., organic fertilizer, effluents from wastewater treatment facilities) and (ii) autochthonous such as OM derived from biota (e.g., algae, bacteria, plankton, and macrophytes) (Derrien et al., 2015). Identifying the origins of the OM in sediments provides a deep understanding of the dynamics of sediment organic matter (SeOM) and its role as a source of energy and nutrients in aquatic systems, as well as the distributions of contaminants and eutrophication processes (Dunn et al., 2008). It can also facilitate and improve the decision making regarding water management if SeOM can be related to a potential threat to drinking water and/or aquatic ecosystems (Derrien et al., 2017b).

Widely utilized tools to identify the OM origins, are stable carbon and nitrogen isotopes and fluorescence spectroscopy (Coble, 2007; Lambert et al., 2011; Xiao and Liu, 2010; Yang and Hur, 2014). Stable isotope ratios ($\delta^{13}\text{C}$ and $\delta^{15}\text{N}$) have been considered the most effective method of tracking both the sources and the transformation processes of organic matter (Toming et al., 2013). Physical, chemical or biological processes in natural environments can lead to changes in the isotopic composition due to a difference in atomic mass between ^{12}C and ^{13}C . For instance, according to the types of plants (e.g., C3, C4 or crassulacean acid metabolism (CAM)) and/or their specific photosynthetic paths, the isotopic ratios may subject to change. C3 presents the values between -33 to -24% , while the values range from -16 to -10% for C4 and between -20 and -10% for CAM. Several studies demonstrated the strong capability of the carbon isotope ratios to distinguish between allochthonous and autochthonous origins (Amiotte-Suchet et al., 2007; Benner et al., 1997; Lambert et al., 2011; Lehmann et al., 2002; Meyers, 1994; Toming et al., 2013). The use of nitrogen stable isotopes for source discrimination is limited due to very complex geochemical cycling of nitrogen and the involvement of many species in the nitrogen pool (inorganic and organic forms) (Bianchi and Canuel, 2011). However, the combined use of nitrogen and carbon stable isotopes enables effective source tracing of particulate OM (POM) (Barros et al., 2010; Gao et al., 2012; Graham et al., 2001; Ogrinc et al., 2005).

Fluorescence spectroscopy has also been widely used to trace OM sources especially using water- or alkali-extractable OM (WEOM or AEOM) from soils and sediments (Coble, 2007; Derrien et al., 2017a; He et al., 2016b; Osburn et al., 2012; Santín et al., 2009). A fraction of the OM, named FDOM, can emit fluorescence after absorbing UV–Visible light. The investigation of the spectroscopic characteristics allows to distinguish different fluorescent components, and helps to identify the types of sources of samples due to a large variability of the characteristics affected by their origins. Many optical indices can be derived from fluorescence spectroscopy, and their capabilities for source identification have been tested in many aquatic environments such as wastewater, rivers, groundwater, lakes, rainwater, and oceans (Derrien et al., 2017b; Fichot et al., 2013; Hur et al., 2006; Inamdar et al., 2011). Meanwhile, fluorescence excitation emission matrices combined with

parallel factor analysis (EEM-PARAFAC) could provide alternative indices for OM sources by using the relative abundances or the relative ratios of different independent fluorescent components, which are decomposed from the EEM datasheet of bulk samples (Stedmon and Bro, 2008; Stedmon et al., 2003).

Although the spectroscopic methods have provided reliable tools to trace the source of organic matter in many previous literatures, they also revealed limited analytical window, which might lower their efficiency, especially, in a complex and mixed environment (Derrien et al., 2017b; Goncalves-Araujo, 2016; Schindler Wildhaber et al., 2012; Yang and Hur, 2014). The Soyang Lake watershed is the largest reservoir system of South Korea. It is located in the upstream region of the Han River (Fig. 1), which serves as the main source of drinking water for about 23 million people of South Korea (Lee et al., 2016). The watershed has a total area of 2700 km² with the altitude range from 80 to 1700 m (Jung et al., 2015; Tenhunen et al., 2011). Approximately 85% of the catchment is covered with forest, 7% is used intensely as arable land (including crops of radishes, cabbages, ginseng, corn, potatoes, and paddy fields) and another 7% as residential area (Choi et al., 2010; Lee et al., 2013). The steep slopes of the watershed facilitate an extremely high transport of POM from various potential sources especially during summer Monsoon season (e.g., annual precipitation of 1370 mm, with 70% of it occurring from late June to September (Arnhold et al., 2014; Tenhunen et al., 2011)). Therefore, sediments in this watershed can be viewed as an interesting location to explore the source discrimination capabilities of various indices.

The overall goal of the study was to compare fluorescence and stable carbon and nitrogen isotopes for assessing their relative contributions to river sediments with the carbon sources within a drainage-basin. For this, several source tracing indices were compiled for many potential SeOM sources, which include soils, forest, crop (C3 and C4), riparian plants, periphyton, and organic fertilizers. To the best knowledge of the authors, this is the first study applying both techniques on similar sourced samples. The relative contributions of different OM sources to the river SeOM samples in Soyang Lake watershed were estimated via end member mixing analysis based on the compiled indices.

2. Materials and methods

2.1. Study area and sampling

Sampling for OM sources and sediments was in May 2015. The source materials were chosen considering that the upstream catchments are mostly forested and agricultural lands. Forest leaves, C3 and C4 crop plants, riparian plants, soil, and organic fertilizer were collected as the OM allochthonous sources from forested areas, river banks and/or agricultural fields. Periphyton (e.g., autochthonous source) was collected from the river beds of the Inbuk and Buk streams since the plankton could be a major source of POM in storm events (Hur et al., 2014). In total, 36 source samples, representing 7 different sources, were collected in the field (Table S1). Five surface sediment samples were collected using a grab sampler (Ekman dredge) in 4 different major tributaries of the Soyang Lake watershed: Mandae River (MD), Inbuk River (IBa and IBb), Buk River (B) and Soyang River (S) (Fig. 1). At the laboratory, the source and sediment samples were stored at -20 °C. They were freeze-dried and grinded for further analyses. Branches and leaves were removed from soils before grinding.

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