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ORIGINAL ARTICLE

Effects of changing land use on dissolved organic matter in a subtropical river watershed, southeast China

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Abstract The composition of dissolved organic matter (DOM) is an important determinant for its biogeochemical role in the aquatic environments. Therefore, it is crucial to determine the effects of changing land use on the DOM composition in the river watershed. Water samples were collected from the outlets of 15 sub-watersheds in the subtropical Jiulong River (southeast China) for fluorescence measurements and parallel factor analysis. Two humic-like (C1 and C2) and one protein-like (C3) fluorescent components were identified. Overall, DOM in the Jiulong River watershed was dominated by humic-like materials, probably due to the fact that 69% of the watershed is covered with forest. The 15 sub-watersheds were grouped into four clusters based on the proportion of each fluorescent component in the total fluorescence, suggesting that the DOM composition could be very different among sub-watersheds. There was a strong negative correlation between C2 and C3%. C1% correlated with the water body fraction, likely associated with the aquatic production of C1. C3% correlated positively with the residential area fraction, likely indicating the influence of anthropogenic activities. These results are useful for assessing the effects of land use/land cover changes on the composition and hence biogeochemical roles of DOM in aquatic environments.

Keywords Dissolved organic matter · Fluorescence · PARAFAC · Land use/land cover · Jiulong River · China

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Introduction

Dissolved organic matter (DOM) plays an important role in the biogeochemical cycles of carbon, nitrogen and other elements in aquatic environments (Bushaw et al. 1996; Moran and Zepp 1997; White et al. 2010). It can also affect both the primary and bacterial productions in natural waters (Arrigo and Brown 1996; Amon and Benner 1996). Furthermore, the fluvial discharge of DOM to the coastal ocean, ~0.25 Gt C year^{-1}, is an important biogeochemical linkage between the land and the ocean (Hedges et al. 1997). However, the bioavailability of DOM is largely dependent on its chemical composition reflected by molecular weight (Amon and Benner 1996; Loh et al. 2004), N:C (Hunt et al. 2000) and the fraction of protein-like materials (Balcarczyk et al. 2009; Fellman et al. 2009; Hood et al. 2009), etc. The photochemical reactivity of different DOM constituents is variable as well (Spencer et al. 2009; Shank et al. 2010). Therefore, to better evaluate the biogeochemical role of DOM in both the freshwater and the receiving coastal ocean, it is important to study the DOM composition and its associated environmental factors in the river watershed.

The composition of freshwater DOM is dependent on its source and biogeochemical transformation history. It can be affected by a variety of environmental factors such as the geomorphology, hydrology, land use/land cover, aquatic light intensity and microbial activity within the watershed (Stedmon and Markager 2005a; Cory and McKnight 2005; Huang and Chen 2009; Spencer et al. 2009; Wilson and Xenopoulos 2009; Yamashita et al. 2010; Williams et al. 2010; Hong et al. 2011). In particular, the DOM composition can be quite different among the watersheds with different land use/land cover patterns (Stedmon and Markager 2005a; Huang and Chen 2009).

For example, industrial, golf course and residential DOM end-members have higher fluorescence quantum yield, absorption spectral slope and photo/biological lability, but lower C:N than forest and wetland end-members (Huang and Chen 2009).

The combination of excitation-emission matrix fluorescence spectroscopy and parallel factor analysis (EEMs-PARAFAC) is a powerful tool in the DOM study (Stedmon et al. 2003). It is very useful to identify different fluorescent components and to trace their source and dynamics in aquatic environments (Stedmon and Markager 2005a; Kowalczuk et al. 2009; Balcarczyk et al. 2009; Fellman et al. 2009; Williams et al. 2010). However, to the best of our knowledge, the correlation analysis between PARAFAC components and the fractions of different land use/land cover types has not been examined except two recent studies that are focused, respectively, on glacial coverage and agricultural land use (Hood et al. 2009; Williams et al. 2010). Thus, there is still limited information on the different roles of the land use/land cover types in affecting the DOM composition revealed by EEMs-PARAFAC, although this is important for estimating the effects of land use/land cover changes on the DOM composition.

The Jiulong River is a typical subtropical river in the southeast China. It is the second largest river in Fujian Province and comprises many sub-watersheds with varying land use/land cover patterns. River discharge is the most important source of DOM for the Jiulong River estuary (Guo et al. 2007, 2011). However, the watershed processes that control the exported DOM composition and reactivity have not been well addressed. This study aimed to: (1) identify the fluorescent components in 15 typical sub-watersheds in the Jiulong River watershed using EEMs-PARAFAC; (2) reveal the similarity and difference in the DOM composition among the sub-watersheds; and (3) examine the relationships between the DOM composition and the land use/land cover patterns.

Materials and methods

Study area

The Jiulong River is a subtropical river located in Fujian Province, southeast China, with a drainage area of 14,741 km^2. The mean annual temperature is 19.9–21.1°C, and the mean annual precipitation is 1,400–1,800 mm (Chen et al. 2008). There are three major tributaries in the watershed, namely North Stream, West Stream and South Stream (Fig. 1). The North Stream is the main tributary with a length of 274 km. The mean annual runoff of North Stream and West Stream together is 123×10^8 m^3, with ~74% being discharged in the wet season from April to September. The population density of the watershed is >200 persons per km^2, and hence, there are intensive human activities such as sewage discharge and agricultural activity.

Field sampling

The drainage areas of North Stream and West Stream are 9,803 and 3,964 km^2, together accounting for 93.4% of the total area of the Jiulong River watershed. Fifteen typical sub-watersheds in the North Stream and West Stream were sampled in the wet season during August 11th–14th, 2009, after a major precipitation event from August 7th–10th, 2009, while the small South Stream was not included in this study (Fig. 1; Table 1). The 15 sub-watersheds account for ~52% of the drainage area of the Jiulong River and are either flowing through the city (like the Xiao tributary through the Longyan City), with intense agricultural activities in the watersheds (like the four sub-watersheds in the West Stream), or within the pristine mountainous area (like the Linbang tributary). Fifteen surface grab water samples were collected from the outlets of all the sub-watersheds, and hence, the influences of the environmental factors in the whole sub-watersheds on the DOM composition could be assessed. The samples were filtered through acid-rinsed Millipore polycarbonate filters with nominal

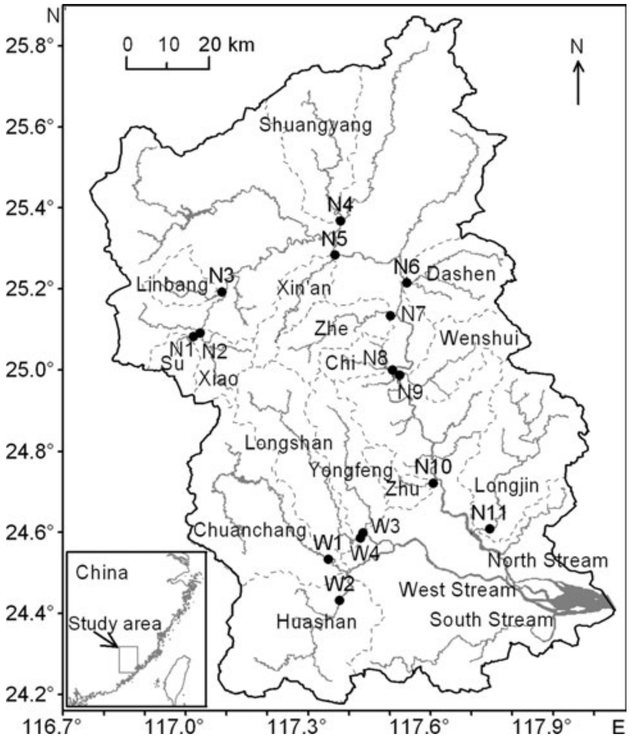

Fig. 1 Study area and sampling stations, with the name and the boundary (the *dashed line*) of the 15 sub-watersheds in the Jiulong River watershed

Table 1 The sample number, the proportions of C(1–3) in the total fluorescence and percentages of the 6 land use/land cover types in the 15 studied sub-watersheds of the Jiulong River

Sub-watershed	Sample no.	C1%	C2%	C3%	Arable land (%)	Forestland (%)	Grassland (%)	Water body (%)	Residential area (%)	Bare land (%)
North stream										
Su	N1	45.9	32.8	21.2	21.90	56.57	0.08	1.18	20.27	0.00
Xiao	N2	45.2	26.2	28.6	14.23	78.23	0.47	1.17	5.60	0.31
Linbang	N3	45.8	33.9	20.3	14.54	82.36	0.17	0.84	2.09	0.00
Shuangyang	N4	47.9	38.3	13.8	7.61	88.77	1.18	1.01	1.27	0.15
Xin’an	N5	48.6	36.2	15.2	7.68	89.21	0.71	1.44	0.64	0.32
Dashen	N6	47.5	40.5	12.0	19.12	78.19	0.11	1.16	1.22	0.19
Zhe	N7	46.2	40.0	13.8	16.31	78.05	0.14	1.16	4.25	0.09
Chi	N8	46.9	41.2	11.9	13.16	85.36	0.73	0.69	0.07	0.01
Wenshui	N9	49.1	34.0	16.9	31.60	64.50	0.10	1.62	1.03	1.16
Zhu	N10	49.0	38.2	12.8	15.23	79.81	0.14	1.17	2.60	1.05
Longjin	N11	47.0	29.7	23.3	33.00	57.59	0.26	3.51	5.28	0.36
West stream										
Chuanchang	W1	48.8	34.2	17.0	18.47	75.84	0.74	1.48	2.64	0.84
Huashan	W2	50.1	33.0	16.9	41.36	52.36	0.55	1.45	3.82	0.47
Yongfeng	W3	48.2	34.8	17.0	19.33	75.83	0.81	1.27	1.63	1.13
Longshan	W4	48.9	34.1	16.9	18.17	77.36	0.24	1.50	2.15	0.58
South stream (no sample)										

pore size of 0.22 μm. The filtrates were stored in cold (4°C) and in the dark for EEMs measurements.

EEMs measurements

EEMs were measured using a Cary Eclipse fluorescence spectrophotometer in signal-to-noise mode, immediately after the samples were transported to the laboratory. The emission spectra were scanned every 2 nm at wavelengths of 230–600 nm, with the excitation wavelengths of 220–450 nm (at 5 nm interval). The fluorescence spectra were corrected for instrument-specific biases using the correction files provided by the manufacturer. Samples with high absorptions were diluted to prevent inner-filter effects. The EEMs of samples were calibrated by the Raman peak of water (Lawaetz and Stedmon 2009) and subtracted from a Raman normalized Milli-Q water EEMs scanned on the same day.

PARAFAC and cluster analysis

The EEMs were modeled by PARAFAC, using MATLAB 7.5 with the DOMFluor toolbox (Stedmon and Bro 2008). Eleven samples collected during the same investigation from the major tributaries or the middle or upper stream of sub-watersheds were also included in PARAFAC. The fluorescence data with excitation wavelengths <250 nm or emission wavelengths <300 nm were not used to exclude noise signals (Stedmon and Markager 2005a). Different fluorescent components were identified by PARAFAC, and the number of components was determined by split-half validation (Stedmon et al. 2003; Stedmon and Bro 2008). The fluorescent components were characterized by their excitation-emission fluorescence spectra (Fig. 2). Each component emitted fluorescence over a series of excitation-emission wavelengths, and hence, the maximum fluorescence $F_{\max}$ was used to evaluate its fluorescence intensity in each sample (Stedmon and Markager 2005a; Kowalczuk et al. 2009; Guo et al. 2011). The DOM composition was represented by the relative contribution of each component to $F_{\max}$ sum of all components (i.e., C1, C2 and C3%, Table 1; Fellman et al. 2009; Kowalczuk et al. 2009). C(1–3)% were arc sin transformed to meet the normality assumption of correlation analysis and cluster analysis.

To group the 15 sub-watersheds based on their DOM composition (i.e., C1, C2 and C3%), hierarchical cluster analysis was carried out using SPSS 13.0. This study used the squared Euclidean distance for measuring the similarity among clusters and the Ward’s method as the agglomeration technique.

Land use/land cover classification

Landsat Thematic Mapper (TM) satellite imagery of May 7th, 2007, in the wet season with 30-m resolution was used to create land cover classifications in the Jiulong River

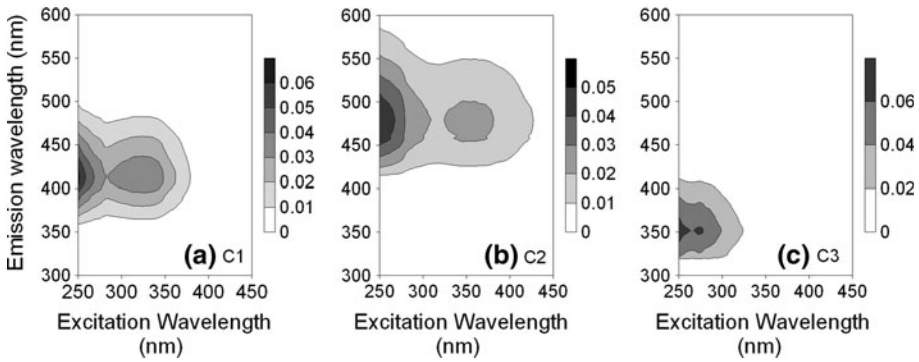

Fig. 2 Excitation-emission matrix spectra of the three fluorescent components identified using PARAFAC

Watershed. Land categories were generated using a combination of manual on-screen digitizing and unsupervised classification based on cluster analysis. Landsat TM data were first separated into 30 classes, which were then merged into six classes: arable land, forestland, grassland, bare land, water body and residential area (Table 1, Huang et al. 2010, 2011). Overall, the watershed land cover in 2007 is 69.4% forestland, 18.4% arable land, 4.7% residential area, 3.5% grassland, 3.1% water body and 1.0% bare land (Huang et al. 2011). The percentages of the 6 land use/land cover types were arc sin transformed to meet the normality assumption of correlation analysis.

Results and discussion

Fluorescent components

PARAFAC can be used to identify different and independently variable fluorescent components, which are characterized by their excitation-emission spectra (Stedmon et al. 2003; Stedmon and Bro 2008). Three fluorescent components were identified using PARAFAC in our study (Fig. 2): two humic-like (C1 and C2) and one protein-like (C3). C1 had two excitation maxima at ≤250 and 325 nm and one emission maximum at 414 nm. It was similar to a combination of the traditionally defined peaks A and M (Coble 1996) and the humic-like component in previous studies (C4 in Stedmon et al. 2003; C5 and C6 in Stedmon and Markager 2005a). The excitation/emission maxima (≤250, 345/478 nm) of C2 resembled a combination of the humic-like peaks A and C reported in Coble (1996) and hence was also a humic-like component (similar to C3 in Stedmon et al. 2003; C2 in Fellman et al. 2009; C3 in Guo et al. 2011). C3 had excitation/emission maxima at ≤250, 275/350 nm, which was identical to that of tryptophan (Coble 1996). Therefore, it was a tryptophan-like component (similar to C7 in Stedmon and Markager 2005a; C8 in Cory and McKnight 2005; C8 in Fellman et al. 2009).

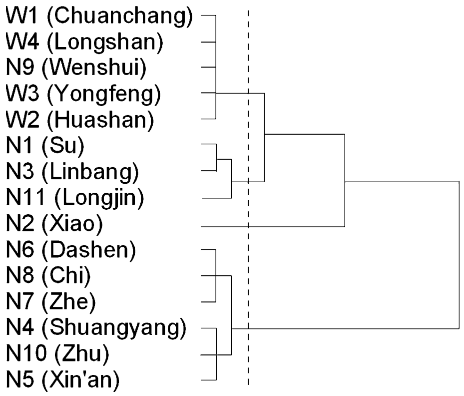

Fig. 3 Dendrogram from cluster analysis of the 15 sub-watersheds based on the DOM composition (i.e., arc sin (C(1–3)%)

Overall, DOM in the Jiulong River watershed was dominated by humic-like materials, as indicated by the low percent of the protein-like C3 (12–29%, with a mean of 17 ± 5%, Table 1). C1 had the highest contribution to the total fluorescence (45–50%, with a mean of 48 ± 1%), followed by C2 (26–41%, with a mean of 35 ± 4%). This was probably due to the fact that 69.4% of the watershed was covered with forest (Huang et al. 2011). Stedmon and Markager (2005a) and Fellman et al. (2009) find that DOM in the soils and the stream in the forest watersheds is dominated by humic-like components.

Cluster analysis results

Generally, the 15 sub-watersheds were grouped into four clusters based on the DOM composition (Fig. 3). The mean fraction of each component for each cluster is shown in Fig. 4. Cluster 1 included all the 4 sub-watersheds in the West Stream (Chuanchang, Longshan, Yongfeng and Huashan) and one tributary in the North Stream (Wenshui). This suggested some similarities in environmental conditions among the sub-watersheds in the West Stream (such as geological settings and intense agricultural activities). DOM in cluster 1 was characterized by the highest C1%

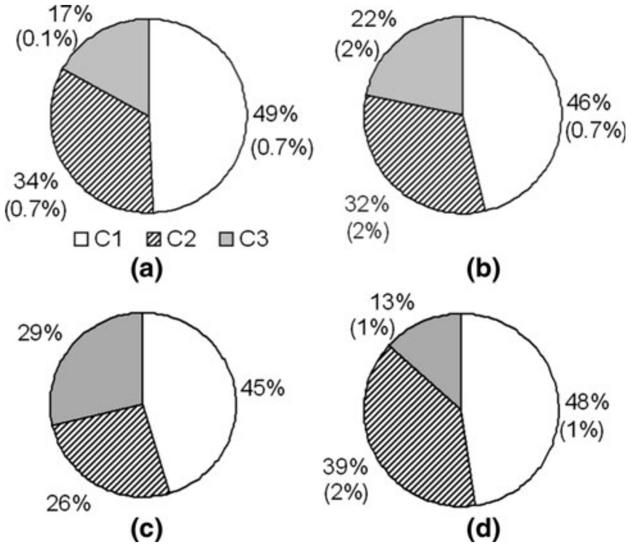

Fig. 4 The DOM composition for sub-watersheds in cluster 1–4 (**a**–**d**), the mean fraction of each component in each cluster was shown, along with the standard error in the *brackets*

and a relatively low percent of the protein-like C3 (Fig. 4a).

Cluster 2 included three sub-watersheds (Su, Linbang and Longjin) and was characterized by relatively high C3%. The sampling site was located in the mountainous area with clean water for the Linbang tributary but closely to residential areas for the Su and Longjin sub-watersheds. Therefore, the elevated tryptophan-like DOM level could be derived from either aquatic production or sewage discharge.

Cluster 3 included only one tributary (Xiao), with the stream mouth located in the city area. DOM in this tributary had the lowest C2% and the highest C3%, likely associated with the high content of protein-like materials in the sewage discharge as reported in Hudson et al. (2007) and Guo et al. (2010).

Cluster 4 included the other 6 sub-watersheds, all of which were located in the middle part of the North Stream. DOM in this cluster had the lowest C3% and the highest C2% and hence was mainly humic-like. This was likely due to the higher percent of forestland in these sub-watersheds (mean: 83.2 ± 5.2%) than others (mean: 69.0 ± 11.2%).

In summary, the 15 sub-watersheds were grouped into four clusters, with different DOM compositions. This also indicated that the biogeochemical reactivity of DOM might vary among the four clusters. For example, previous studies find that the bioavailability of aquatic DOM is positively correlated with the fraction of protein-like components (e.g., Balcarczyk et al. 2009; Fellman et al. 2009; Hood et al. 2009). Therefore, it is crucial to reveal further the environmental factors affecting the DOM composition.

The correlation between DOM composition and the land use/land cover

Since the percentages of C(1–3) add up to unity, correlation analysis was carried out for arc sin (C1–3)%. The results showed a moderate but insignificant correlation between arc sin (C1%) and arc sin (C3%) ($r = -0.48$, $p = 0.07$) and a strong correlation between arc sin (C2%) and arc sin (C3%) ($r = -0.95$, $p < 0.001$). For similar reasons as (C1–3)%, correlation analysis was carried out for arc sin transformed percentages of the 6 land use/land cover types (Table 2). Since the historical land transition in the Jiulong River watershed is dominated by that between the natural (including forestland, grassland, bare land and water body) and the agriculture (Huang et al. 2010), arc sin (forestland%) correlated strongly and negatively with arc sin (arable land%) ($r = -0.92$, Table 2). These results were taken into account when discussing the correlations between (C1–3)% and land use/land cover types.

Arc sin (C1%) generally correlated with arc sin (water body%) (Fig. 5a). Peak M in C1 is initially proposed to be of marine origin by Coble (1996) and can be produced by marine phytoplankton (Romera-Castillo et al. 2010). This component is also common in freshwater (Stedmon et al. 2003; Stedmon and Markager 2005a), and its correlation with the water body fraction in this study suggested that it might receive addition from aquatic production in freshwater as well.

The arc sin transformed fraction of protein-like C3 generally correlated positively with that of residential area (Fig. 5b). Based on previous studies, sewage discharge is characterized by abundant protein-like DOM (Baker 2001; Hudson et al. 2007; Guo et al. 2010), and the abundant anthropogenic nutrient inputs might stimulate the algal growth (Wong and Wong 2004; Jennerjahn et al. 2009; Nixon and Fulweiler 2011), which is another source for protein-like DOM (Stedmon and Markager 2005b; Hong et al. 2011). In addition, Huang et al. (2011) find positive correlations between nutrient levels, biological oxygen demand and residential area (%) in Jiulong River. In Fig. 5, the maximum value of arc sin residential area (%) was identified as outliers using Grubbs test and perhaps the effect on C3% saturated with extreme residential area fraction.

Therefore, our results showed the correlation between the DOM composition and the percentages of different land use/land cover types, indicating the impacts of changing land use/land cover on the DOM composition. The increasing rate of residential area in the Jiulong River watershed has accelerated from 13 $\mathrm{km}^2\ \mathrm{year}^{-1}$ (0.09% year^{-1}) during 1996–2002 to 61 $\mathrm{km}^2\ \mathrm{year}^{-1}$ (0.41% year^{-1}) during 2002–2007 (Huang et al. 2010). With the socioeconomic development and the increasing

Table 2 Correlation analysis results of the arc sin transformed percentages of the 6 land use/land cover types

	Arable land	Forestland	Grassland	Water body[a]	Residential area[a]
Forestland	−0.92***				
Grassland	−0.34	0.45*			
Water body[a]	0.50*	−0.45	−0.13		
Residential area[a]	0.38	−0.53*	−0.28	0.15	
Bare land	0.28	−0.19	0.01	0.63**	−0.07

*** $p \leq 0.01$; ** $p \leq 0.05$; * $p \leq 0.1$

[a] The maximum values of arc sin (water body%) and arc sin (residential area%) were identified as outliers using Grubbs test and were excluded in correlation analysis

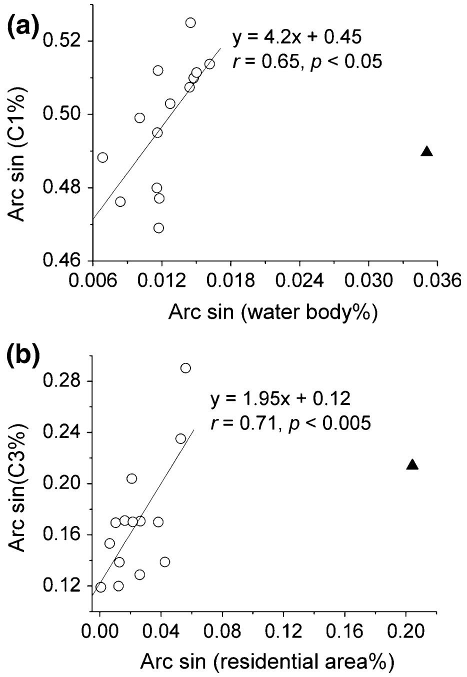

Fig. 5 The correlation between the DOM composition and land use/land cover (the maximum values of arc sin (water body%) and arc sin (residential area%) as indicated by *filled triangle* was identified as outliers using Grubbs test and were excluded in the correlation analysis)

human activity, the impacts of land use/land cover change on the freshwater DOM composition may be increasingly notable. The change in the DOM composition is also expected to affect its bioavailability and hence biogeochemical roles in both the freshwater and the receiving coastal ocean.

Conclusions

The biogeochemical roles of DOM in the aquatic environments are dependent largely on its composition. However, the latter could vary greatly within the river watershed, like those in the four clusters of the 15 subwatersheds in the subtropical Jiulong River watershed. The DOM composition correlated with the fractions of different land use/land cover types such as water body and residential area in our study. Therefore, land use/land cover changes may lead to changes in the composition and hence biogeochemical roles of DOM in the river watershed.

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