

Isotopic fractionation of particulate organic matter and its biogeochemical implication in the littoral zone of Lake Taihu, China

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ABSTRACT

Signatures of stable isotope ratios have previously been used to trace the source and transport of particulate organic matter (POM) in freshwater and marine ecosystems. In this study, water columns were collected at 22 sites in the littoral zone of Lake Taihu in 2014 to investigate the distribution and concentration of nutrients and the stable isotope signatures of POM, and their potential interrelation. Generally, mean concentration of nitrogen forms ($\text{NH}_4^+\text{-N}$) showed substantial variation, probably because they had received large amounts of wastewater from various local industrial enterprises. Source analysis by stable carbon and nitrogen isotopic ratios showed that the main POM sources were phytoplankton. Contrasting correlations were obtained between $\delta^{15}\text{N}$ and N concentrations in effluent river mouths when compared with influent river mouths. In effluent river mouths, there was a significant positive correlation between $\delta^{15}\text{N}$ and nitrogen concentration (total nitrogen and ammonia-nitrogen), in contrast with the negative correlation in influent river mouths. According to these results, more factors should be taken into consideration when stable carbon and nitrogen isotopes of POM are used to assess the feeding relationship between consumers and prey, as well as the energy flow pathways that support the lake pelagic food webs.

Key words | Lake Taihu Basin, lakeside, stable carbon isotope, stable nitrogen isotope

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INTRODUCTION

Particulate organic matter (POM) is an important component in aquatic ecosystems considering its capacity to scavenge trace metals and hydrophobic organic matter, as well as its high oxygen demand and potential for nutrient release during degradation (Lin *et al.* 2014). Aquatic systems receive POM from multiple sources, of which allochthonous and autochthonous types serve as the two main sources (Kendall *et al.* 2001). The typical allochthonous source of POM refers to soil organic matter and leaf debris, while the main autochthonous sources are phytoplankton and macrophytes (Kendall *et al.* 2001). Stable isotope techniques have been widely used in research into aquatic systems. Through measuring stable carbon (C) and nitrogen (N) isotope ratios of POM, as well as related sources, we can understand the pollutant sources and fate of organic matter in aquatic ecosystems (Rock & Mayer 2006).

Stable C and N isotopes of POM have also been used to establish trophic baselines, so as to further assess the trophic interactions between consumers and their food, and build energy flow pathways in aquatic food webs (Gu 2009). As demonstrated in a number of studies, the stable isotope fractionation changes under different environmental conditions, or is variable among different groups of phytoplankton (Wada *et al.* 2013; Evans & Von 2013; Woodland *et al.* 2012). For example, variations in $\delta^{15}\text{N}$ in particulate matter have been observed in oligotrophic ocean and in nutrient-rich coastal water (Soares *et al.* 2015). In terms of this variation in isotopic composition, it results from the alteration of the isotopic composition of the dissolved inorganic nitrogen (DIN) compounds by microbial processes (Möbius 2013). However, it is difficult to determine the relationship between DIN concentration and $\delta^{15}\text{N}$, as contradictory results have been reported in previous studies of different

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lakes (Pastor *et al.* 2014). In the case of significant differences occurring in an individual lake, this could lead to erroneous interpretations of biogeochemical processes. Only a few studies have focused on the variations in $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ of POM in eutrophic lakes, despite the extensive analysis of POM stable isotopes used for monitoring pollution sources in marine ecosystems (Berto *et al.* 2013; Bareket *et al.* 2016).

As one of the biggest freshwater lakes in China, Lake Taihu provides multiple ecosystem services, including supplying drinking water, flood control, shipping, fishery, aquaculture, and farming. For the past few years, problems of water environmental pollution have become increasingly serious because of the rapid development of the local economy and subsequent intensive use of water resources. This kind of change might have profound impacts on C and N biogeochemical cycles in the lake, which is essential to identify the contamination sources during strategic decision making around water management. Thus, it is urgent to investigate the spatial variability of $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ of POM in surface water in Lake Taihu. Nevertheless, previous investigations focused primarily on natural waters at small scale, while fewer studies have paid attention to the distribution of C and N stable isotopes in POM in the whole lake (Ding *et al.* 2015).

In this study, water columns were collected at 22 sites in Lake Taihu in 2014, a lake famous for its hypereutrophic status. The main purposes of the present study were to (1) investigate the concentration and distribution of nutrients in the littoral zone of the whole of Lake Taihu, (2) analyze the spatial variability of $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ of POM and identify its source, and (3) further discuss the relationship between $\delta^{15}\text{N}$, $\delta^{13}\text{C}$ and N in different representative areas.

MATERIAL AND METHODS

Lake Taihu, located in the lower reaches of the Yangtze River, is one of the five famous and largest freshwater lakes in China. The lake is important for flood control, water supply, cultivation, irrigation, navigation, and tourism. Moreover, with an extensive area of about $3.69 \times 10^4 \text{ km}^2$, the lake supports more than 60 million people (about 1,500 people/ km^2 on average) who are engaged in agriculture and industry (Niu *et al.* 2015). The levee project around Lake Taihu is one of the 10 key projects for comprehensive management in the Taihu basin. Completed in 2005, this hydraulic construction is an effective measure to control diffuse pollution into the lake. Lake Taihu has been an important natural laboratory for understanding the eutrophication process. The lake usually receives its inflow from the

southwest and discharges to the northeast because of the catchment's topographic features (Huang & Zhu 1996). In this study, a total of 22 surface water samples were collected from the littoral zone of Lake Taihu in August 2014. As seen in Figure 1, sites S01–S13 were located in the southwest of the littoral zone (IRM: influent river mouth), and sites S14–S22 were located in the northeast of the littoral zone (ERM: effluent river mouth).

Water samples were stored in acid-cleaned polyethylene bottles. After water samples were taken back to the laboratory, a portion of each sample was filtered through $0.45 \mu\text{m}$ cellulose-acetate filter paper for chemical analysis (Wang *et al.* 2007). Another portion of each sample was filtered through Whatman GF/F fiberglass filters (47 mm) and stored at 4°C until analysis. The filter papers were further processed for the analysis of C and N content and their isotopic ratio of POM. Several water quality parameters including pH, temperature (T), and dissolved oxygen (DO) were determined in the field using a handheld multi parameter meter (YSI, USA). Total phosphorus (TP), total nitrogen (TN) and ammonia nitrogen ($\text{NH}_4^+\text{-N}$) were measured using the Chinese standard methodology for lake water surveys (Wang *et al.* 2007). These methods are similar to the American standard methods for those parameters (Wang *et al.* 2007). Few variations (coefficient of variation $<15\%$) were observed among the water index values (TP, TN and $\text{NH}_4^+\text{-N}$) of the three replicates for all samples. The suspended POM samples were dried at 60°C and acidified with HCl vapor to remove any carbonate if present in the samples. The C and N contents and their isotope ratios in the POM samples were determined by using an isotope ratio mass spectrophotometer equipped with an elemental analyzer (Fisons NA1500-Finnigan MAT 252; Jha & Masao 2013).

RESULTS

Physico-chemical parameters of water quality

Physico-chemical parameters, including T, pH, DO, TP, $\text{NH}_4^+\text{-N}$ and TN, of the water column were summarized in Table 1. The T, pH and TN values of the water samples showed low variation, indicating a homogeneous spatial variation of these indexes in the column. The mean concentrations of DO, $\text{NH}_4^+\text{-N}$, and TP in IRM were observed to be 1.18, 1.73 and 1.44 times greater, respectively, compared with their mean values in ERM. The results indicated a significant spatial variation in concentrations of chemical parameters in the water column.

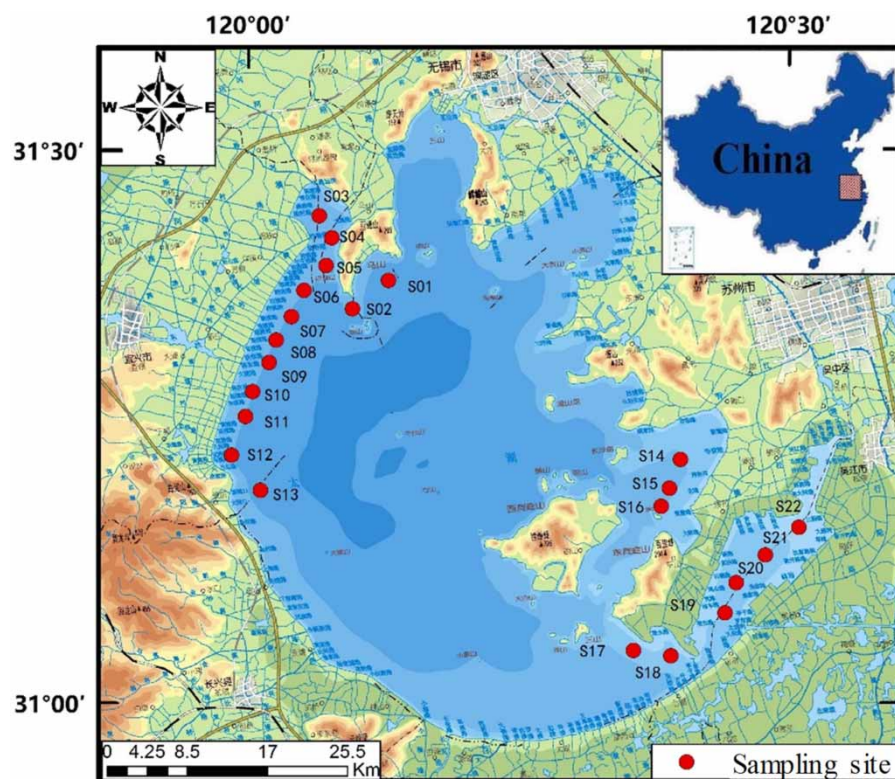


Figure 1 | Study area and sampling sites in Lake Taihu.

Table 1 | Physico-chemical parameters of water quality in Lake Taihu

Water type	Descriptive parameters	T °C	pH	DO (mg/L)	TN (mg/L)	NH ₄ ⁺ -N (mg/L)	TP (mg/L)
IRM	Sampling number	15	15	15	15	15	15
	Mean	31.80	7.64	6.11	3.06	2.33	0.26
	SD	1.28	0.67	2.56	1.78	0.93	0.13
ERM	Sampling number	7	7	7	7	7	7
	Mean	29.77	7.17	5.17	3.81	1.35	0.18
	SD	1.90	0.43	1.79	1.62	0.43	0.13

The isotopic composition and C:N ratio of POM is shown in Figure 2. The range of C and N isotopic ratios and the molar C:N ratio of suspended POM of samples are shown as follows: $\delta^{13}\text{C}$, -33.92 to -21.39‰ , $\delta^{15}\text{N}$, 1.34 to 10.46‰ and C:N ratios, 3.08 to 7.97 .

Correlation of $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ with environmental variables

To understand the relationships between stable isotopes and environmental variables correlation analysis was performed, with the correlation coefficients between variables shown in Table 2. The value of $\delta^{15}\text{N}$ was positively correlated with DO and pH ($r^2 = 0.577$, $p < 0.01$ and $r^2 = 0.449$, $p < 0.05$,

respectively), while $\delta^{15}\text{N}$ was negatively correlated with TN and NH₄⁺-N ($r^2 = -0.766$, $p < 0.01$ and $r^2 = -0.521$, $p < 0.01$, respectively). The value of $\delta^{13}\text{C}$ was only significantly correlated with pH ($r^2 = 0.440$, $p < 0.05$).

To better understand the relationship between stable isotopes and NH₄⁺-N, all sites were separated into two groups according to location. The obvious relationship among variables is shown in Figure 3. Contrasting correlations of $\delta^{15}\text{N}$ and nitrogen concentration (TN, NH₄⁺-N) were obtained between effluent river mouths when compared with influent river mouths. In effluent river mouths, there was a significant positive correlation between $\delta^{15}\text{N}$ and nitrogen concentration (TN, NH₄⁺-N), while there was a negative correlation in the influent river mouths.

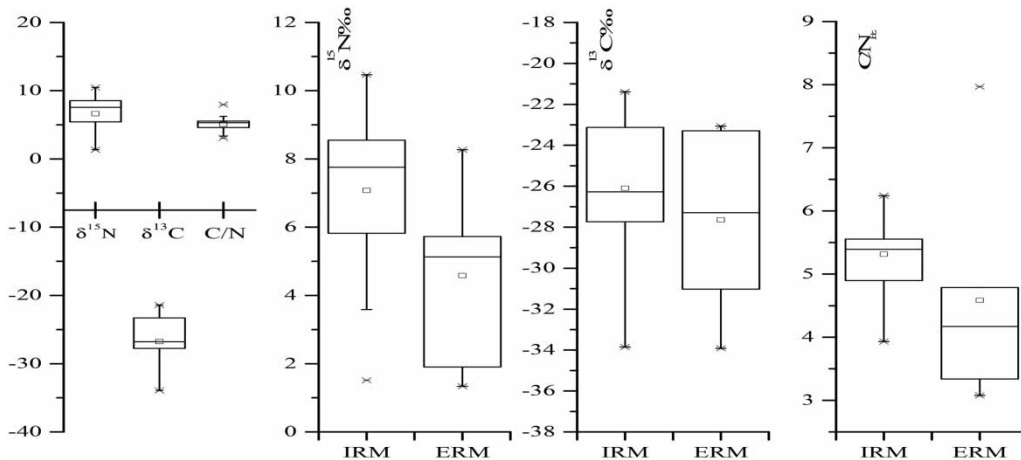


Figure 2 | Isotopic composition and carbon: nitrogen (C:N) ratio of particulate organic matter in Lake Taihu. IRM: influent river mouth; ERM: effluent river mouth.

Table 2 | Correlation of $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ with environmental variables ($n = 22$)

Element	DO	pH	TN	$\text{NH}_4^+\text{-N}$	TP
$\delta^{15}\text{N}$	0.577**	0.449*	-0.766**	-0.521*	-0.316
$\delta^{13}\text{C}$	0.360	0.440*	-0.036	-0.226	0.249

*Significance at $p < 0.05$, **Significance at $p < 0.01$.

DISCUSSION

Variation in water quality

By 2000, with only 0.4% of the land surface and 3% of the population, this basin accounted for 10% of the total gross domestic product (GDP) of China (Li *et al.* 2014). Taihu

Basin has become the most industrialized and urbanized area in China (Li *et al.* 2014). During the last several decades, the population and economy have grown rapidly in this region, resulting in a large amount of untreated domestic sewage and industrial wastewater being discharged into the lake body via river runoff. Among the developing countries, most of the rivers in the urban areas are at the end points of effluent discharge. Related reports have shown that 70% of contaminants in Lake Taihu originate from the surrounding tributaries (Wu *et al.* 2007). Therefore, the highest concentrations of TN and TP were observed in the southwest of the littoral zone, where many manufacturing factories have been built and wastewater from domestic and industrial plants is discharged. In addition, the mean concentration of TN in the water column showed less variation in IRM than in ERM,

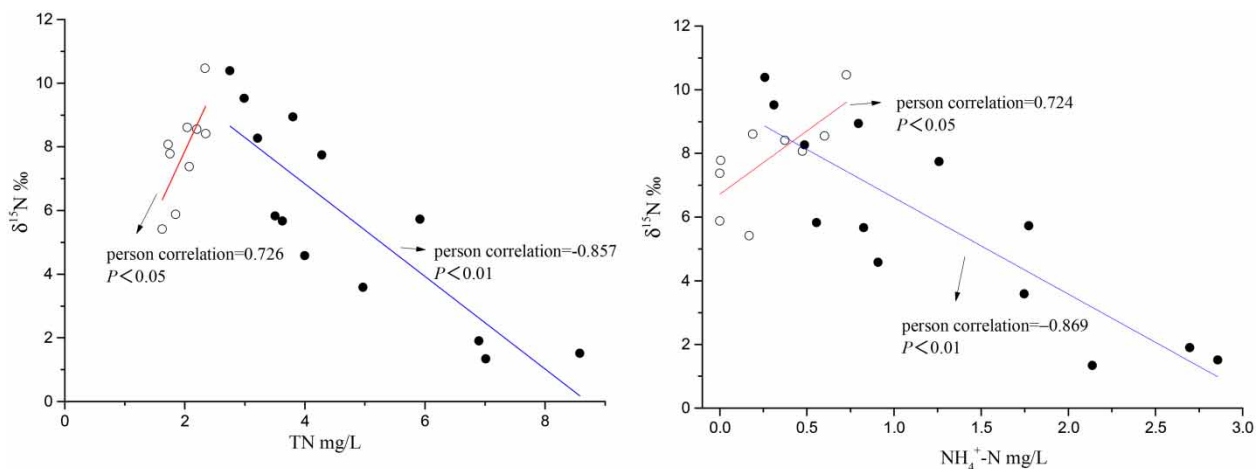


Figure 3 | Correlation coefficients between $\delta^{15}\text{N}$ and nitrogen concentrations. Note: 'open circles' are sampling stations located in effluent river mouths (S14–S22), 'solid circles' are sampling stations located in influent river mouths (S01–S13), 'red line' is linear regression curve in effluent river mouths, 'blue line' is linear regression curve in influent river mouths. Please refer to the online version of this paper to see this figure in colour: <http://dx.doi.org/10.2166/wst.2017.439>.

while the mean concentration of nitrogen forms ($\text{NH}_4^+\text{-N}$) showed an obvious variation because of nitrification.

C and N isotopes in POM and their biogeochemical implications

The differences in composition of autochthonous POM input from the river, and that from the local terrestrial vegetation, could affect the C cycling in floodplains (Lin *et al.* 2014). Signatures of stable isotope ratios have previously been used to trace the source and transport of particulate organic C in tropical rivers and in temperate river reaches (Rock & Mayer 2006). Furthermore, a combination of measurements of $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and C:N ratio has been recently used to assess the degradation of POM and indicate the importance of different sources of POM in an estuary (Watanabe & Omura 2008).

In the study, we distinguished the ratio of allochthonous and autochthonous organic matter by examining the differences in C and N composition in POM. It is generally believed that when the C:N ratio is under 10, organic matter in the sediment is derived mainly from allochthonous organic matter; when the C:N ratio exceeds 10, organic matter in the sediment is mainly autochthonous, and when the C:N ratio is approximately 10, allochthonous and autochthonous organic matters have reached an approximate equilibrium (Kendall *et al.* 2001). As shown in Figure 2, the C:N ratios of POM from the inner lakeside zone of Lake Taihu were 3.08–6.24, and the mean ratio was 5.00, thereby revealing that organic matter in the POM is mainly autochthonous.

The isotopic compositions of POM reflect the relative proportions of allochthonous terrestrial organic matter, and autochthonous particles. However, the interpretation for the isotopic ratio of bulk samples such as POM is complicated. This is mainly because of the existence of multiple sources with overlapping isotopic range in a watershed area, and the isotopic signatures might have been changed because of organic matter degradation and transportation (Jha & Masao 2013). Hence, a single stable isotope value is not expected to be a very useful indicator of source. To improve the accuracy of the analysis, double indicators for POM component analysis were used in this study. The following review of the literature provides data on the $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and C:N values of major potential sources of POM to streams. As an aid to the reader, the average values and normal ranges of values (in parentheses) described below are summarized in Table 3. Comparison of $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and C:N values for POM (Figure 4), results showed that the main POM sources are phytoplankton.

Table 3 | Typical average compositional values of major particulate organic matter (POM) sources

POM source	Type	$\delta^{13}\text{C}$	$\delta^{15}\text{N}$	C:N ratio
Plankton		–42 to –24	–15 to 20	5 to 8
Macrophytes		–28 to –18	–15 to 20	10 to 30
Soil organic matter	C3	–32 to –22	2 to 5	8 to 15
	C4	–16 to –9		
Terrestrial plants	C3	–32 to –22	3 to 7	> 15
	C4	–16 to –9		

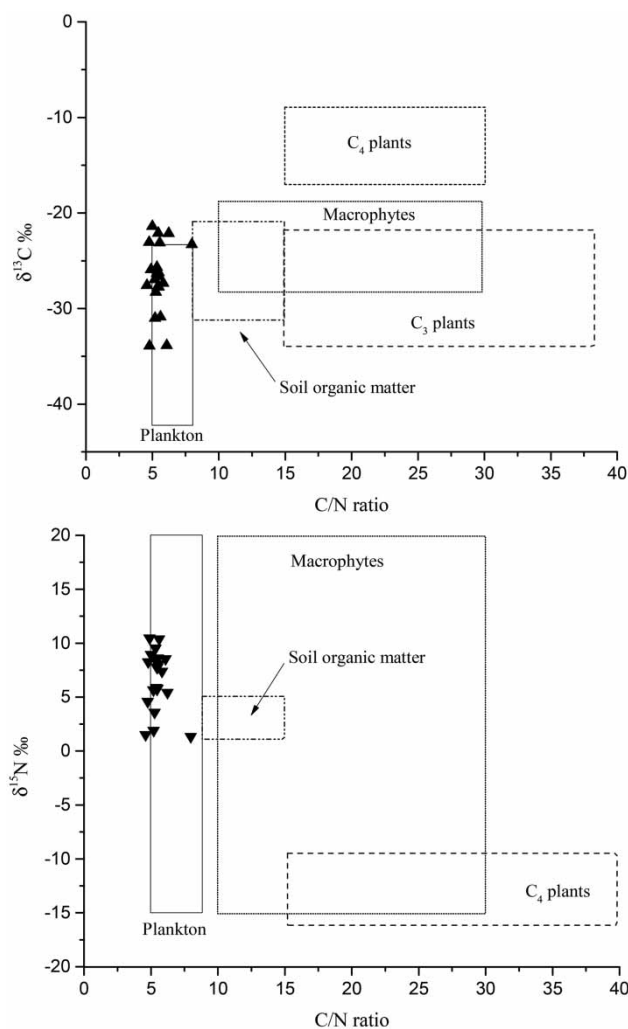


Figure 4 | Comparison of $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and C:N values for particulate organic matter (POM) in Lake Taihu. Note: 'solid triangles' are the sample values.

Variation of C and N isotopes in POM

Lakes surrounded by watersheds with strong human influences might receive higher contributions from agriculture

and domestic discharges than lakes in remote areas (Gu 2009). Therefore, lakes with different geographical locations and morphometries might be provided with different sources and isotopic composition of particulates, as well as dissolved matter that can profoundly influence the ^{15}N signature of POM. In our study, the main POM sources are phytoplankton, and N isotope fractionation by phytoplankton follows the Rayleigh model under batch culture conditions (Granger *et al.* 2004). In nitrate- and ammonium-rich environments, fractionation during the growth process of algae would exert a major control on particulate $\delta^{15}\text{N}$ in aquatic ecosystems. In the Lake Taihu Basin, the IRM region showed the highest concentration of $\text{NH}_4^+\text{-N}$, which suggests that it receives large amounts of wastewater from the basin. During transit in the lake, lighter isotopes tend to react faster than heavier ones, which causes enrichment of light isotopes in the substrate and depletion in the reactant. This phenomenon could explain why $\delta^{15}\text{N}$ was negatively correlated with TN and $\text{NH}_4^+\text{-N}$ ($r^2 = -0.857$, $p < 0.01$ and $r^2 = -0.869$, $p < 0.01$, respectively) in IRM.

Nitrogen isotopic fractionation associated with N uptake and assimilation by phytoplankton is one of the most important fractionation processes in the biogeochemical cycle of N in the lake (Waser *et al.* 1998). The N assimilation process is responsible for the isotopic differences between the nitrate substrate and the biomass N. Algae preferentially assimilate light ($\delta^{14}\text{N}$) NH_4^+ , and then the residual NH_4^+ should become isotopically heavier. In Lake Taihu, the presence of the heavier isotope from aquaculture is also an important source of $\text{NH}_4^+\text{-N}$, which might help to explain the existence of different relationships of $\delta^{15}\text{N}$ and N concentrations between influent and effluent river mouths.

Different kinds of algae can also lead to different isotopic signatures. Vuorio *et al.* (2006) separated a variety of algae from natural water bodies, followed by analysis of their N isotopic signatures and found that N from different algal species varied greatly; the N isotopic signature for N-fixing algae *Anabaena* spp. and *Aphanizomenon* spp. ranged from 0.5‰ to 2.0‰. In the present study, POM was mainly from phytoplankton whose N composition was affected by a series of factors, including species composition, growth characteristics, and the isotope characteristics of the DIN sources used (Hadas *et al.* 2009). For example, under anaerobic conditions, nitrification could transform the $\text{NH}_4^+\text{-N}$ with high levels of light isotope into nitrite and nitrate. Thereby, the use of different forms of N by phytoplankton could directly result in N composition change. In

addition, the factors influencing phytoplankton growth, such as light, temperature, nutrient concentrations and the main N cycle process in water (nitrification, denitrification, N fixation or others) could also affect the N composition of algae (Hadas *et al.* 2009). In general, when DIN is sufficient in the lake or the phytoplankton grows slowly, the isotopic signature of N from DIN and phytoplankton varies greatly, while in the case of deficient DIN or fast growing phytoplankton, the difference between the isotopic composition of DIN and phytoplankton N is smaller (Syväranta *et al.* 2008).

There are other factors that can result in changes in isotope composition, such as different sources of pollution, water environment changes caused by hydrodynamic processes and changing environmental conditions. There are still gaps in information on the relationship between isotope composition and N sources in our work. It should be noted that, in the present study, the correlation between isotopic composition and N sources was either positive or negative depending on location in the same eutrophic lake. This reminds us that the influence of external factors should be taken into consideration when N isotopes are used as a baseline to evaluate predator–prey relationships and geochemical cycles.

CONCLUSIONS

In this paper, we describe a detailed study on spatial variations in the $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ of various POMs in the littoral zone of Lake Taihu, a lake famous for its hyper-eutrophic status. Moreover, the relationship between $\delta^{15}\text{N}$ and nutrients was discussed. Generally, the mean concentration of TN in the water column showed low variation between IRM and ERM, while the mean concentration of nitrogen forms ($\text{NH}_4^+\text{-N}$) showed an obvious variation because of nitrification, which is the biological conversion of organic or inorganic N compounds from a reduced to a more oxidized state. Source analysis by stable C and N isotopic ratios showed that the main POM sources were from phytoplankton. Furthermore, there was a significant positive correlation between $\delta^{15}\text{N}$ and nitrogen concentrations (TN, $\text{NH}_4^+\text{-N}$) in effluent river mouths, while the correlation tended to be negative in influent river mouths. Furthermore, there was a negative correlation between $\delta^{15}\text{N}$ and nitrate concentration because of biogeochemical processes. Although we have not provided direct evidence for the isotope fractionation changes, we consider that our work should be considered a warning: care must be taken in the

use of stable C and N isotopes of POM to assess the feeding relationship between consumers and prey, as well as the energy flow pathways that support the lake pelagic food webs.

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