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Signals of typhoon induced hydrologic alteration in particulate organic matter from largest tropical river system of Hainan Island, South China Sea

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SUMMARY

Tropical river systems affected by climatic extremes (typhoon) are recognized as significant source of particulate organic matter (POM) delivered to their adjacent seas. Studies on POM composition in typhoon affected rivers of tropical Hainan Island are limited. The Nandu River-Estuary (NRE) is the largest river system on Hainan Island in the South China Sea, affected by frequent typhoons every year. We used elemental contents, stable isotope ratios of organic carbon and lignin phenols to characterize POM compositions in NRE during typhoon affected wet season (August, 2011) vs. normal wet season (October, 2012). Short term and heavy precipitation during typhoon in August, 2011 was evidenced with a significant hydrologic change as well as change in POM composition along the NRE. The multi-proxy results suggest that POM was degraded and their sources significantly changed along the NRE hydrograph. Results from an end member mixing model indicated that POM constituted nearly similar OM input from soil (35%) and freshwater plankton (32%) during August, 2011, in contrast POM dominated with OM from freshwater plankton (51%) during October 2012 in riverine regions of NRE. In the estuarine region, POM constituted dominant inputs from marine plankton during August, 2011 (44%) and October, 2012 (56%) as compared to other sources. Collectively, the nature of POM composition change in the vicinity of typhoon induced copious precipitation, with potential land-use intervention across the Hainan Island are key factors affecting the carbon cycling in NRE and adjacent South China Sea.

1. Introduction

River transport of particulate organic matter (POM) is an important component of marine carbon cycle, in addition, their composition is extremely sensitive to local and global environmental perturbations, and therefore it represents an important linkage with global carbon cycle. River borne POM preserved in the estuaries, deltas and continental shelves strongly influence the global biogeochemical cycles and ocean's ability towards atmospheric CO_2 sequestration (Bianchi et al., 2013). Rivers across the tropical region (30°N–30°S) importantly contributes $\sim 25 \times 10^{12}\ m^3\ yr^{-1}$ freshwater, $8.96 \times 10^{12}\ kg\ yr^{-1}$ sediment and $0.13 \times 10^{12}\ kg\ yr^{-1}$ particulate organic carbon (POC) to the global ocean. This is equivalent to ~66%, ~50% and ~70% of total annual freshwater discharge, sediment load and POC delivery from global rivers respectively (Huang et al., 2012). However, yet the global understanding of land-ocean carbon composition change and factors influencing along their dispersal pathways (rivers–estuary–sea), are hampered by complexities of natural climate change (excess/less precipitation) and anthropogenic activities (dam building, excessive agriculture, deforestation) in many tropical regions of the world.

Studies related to the POM compositions in typhoon affected tropical river–estuary systems are sparse (Liu et al., 2007a; Herbeck et al., 2011), despite the fact that typhoon affected tropical rivers are recognized as significant source of POM delivered to the adjacent seas and plays important role in global carbon cycling (Hilton et al., 2008). The varying source of POM delivered during typhoon-induced hydrologic change in tropical river–estuary systems is mainly related to the rainfall intensity (Jung et al., 2012). Besides, the human induced land-use changes (deforestation, excessive agriculture, dam building, increased industrial and domestic pollutants, etc.) largely corresponds to alteration in POM composition within the tropical river catchments, which is then delivered to near shore marine environments (Goldsmith

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et al., 2008; Hilton et al., 2008; Wu et al., 2013). Tracking above processes in typhoon affected small and large tropical rivers are evenly essential considering the fact that POM biogeochemistry has remained a bottleneck for clear understanding in many tropical regions of world.

One of such region remain understudied is the Hainan Island in South China Sea (SCS), which gets affected by frequent tropical storms (typhoon) during boreal summer. The combined effects of heavy precipitation during typhoon and prevailing anthropogenic activities (rising population, agriculture and aquaculture) have caused excessive nutrient and OM discharge from the rivers of Hainan Island and thereby affected the marine ecosystem of SCS (Liu et al., 2011; Herbeck et al., 2011; Unger et al., 2013; Wu et al., 2013). Rivers draining the high standing islands (e.g. Eel (USA); Blair et al., 2004, Santa Clara (USA); Komada et al., 2004, Strickland and Fly (Papua New Guinea); Alin et al., 2008, Rio Loco (Puerto Rico); Moyer et al., 2013 and Lanyang-Hsi (Taiwan); Kao and Liu, 2000) often delivered POM, with a greater input from the geological OM sources, especially during their flooding season. However, the less steep Hainan Rivers are hypothesized to deliver POM, with altered in composition mainly due to the typhoon related heavy precipitation and anthropogenic activities that will further influence the carbon cycling of SCS. To our knowledge, so far a complete understanding of POM composition across the river–estuary systems of Hainan Island is lacking. This study attempts to fulfill this gap by understanding the POM composition in Nandujiang (or Nandu River system; 'jiang' refers to River in Chinese), the largest river system of Hainan Island. We present the POM biogeochemistry along the Nandu River-Estuary (herein after NRE) during August, 2011 (typhoon affected wet season) and October, 2012 (normal wet seasons), by examining the results of multiple organic geochemical analyses such as molar ratio of organic carbon to total nitrogen (C/N), stable isotopic ratios of organic carbon ($\delta^{13}C_{org}$) and lignin phenols (biopolymers, synthesized by terrestrial vascular plants) composition. The main objectives of this study are to provide information on (i) spatio-temporal distribution of POM, (ii) sources of POM and (iii) factors influencing POM compositions in NRE. The combined use of lignin phenols along with C/N and $\delta^{13}C_{org}$ will help to obtain robust information about POM sources; also the information about diagenetic change can be acquired by observing specific phenol monomers (Bianchi et al., 2011; Dittmar and Lara, 2001). Collectively, the results from present investigation will provide useful background information to the fact that the river basins of tropical Hainan Island would inevitably be developed in near future owing to ongoing global population rise.

2. Materials and methods

2.1. Study area description

The Nandujiang originates from Bawang hills in the central Hainan Island and meander north-east–north direction about 320 km before draining into the Qiongzhou Strait in SCS (Fig. 1). The entire catchment of NRE (7022 km^2) occupy 21% of total geographical area of Hainan Island and its estuarine region is located in densely populated Haikou district (population density; 910 km^{-2}). The NRE bifurcates into three channels (Haidianxi, Henggou and Beiganliu) in the estuarine region, amongst Beiganliu act as major freshwater discharge outlet to Qiongzhou Strait. Climatologically, the average annual precipitation on Hainan Island is about 1774 mm, which oscillates between 1200 mm and 2200 mm. Distinct wet (May–October) and dry (November–April) seasons are prevalent over the Island, with 80% of total annual precipitation occur during wet season. Tropical cyclones cross over the Island during July–October, causing copious precipitation (highest during August), which equals to ~30% to the total annual precipitation over Hainan Island (Wu et al., 2007). Average freshwater discharge and sediment load for a period from 1957 to 2008 in NRE are 5.7×10^9 m^3 yr^{-1} and 0.4×10^9 kg yr^{-1} respectively, which equals to ~20% and 10% of total freshwater discharge (31×10^9 m^3 yr^{-1}) and sediment load (4×10^9 kg yr^{-1}) respectively, from all the rivers of Hainan Island (Yang et al., 2013; Zhang et al., 2013). The Songtao reservoir exists in the upper reaches of NRE, with an annual water storage capacity of 3.3×10^9 m^3 that equals to ~60% of the total river runoff.

The land-use pattern over NRE varies from upland forest cover to lowland urbanized areas, which is due to a massive deforestation that caused significant reduction in forest cover from 169×10^3 km^2 to 5.8×10^3 km^2 between 1933 and 2008 (Zhang and Zhu, 2012). In the estuarine water, diurnal tidal currents (amplitude: 1.1 m) oscillate in a north-east–north direction across the Qiongzhou Strait (Wang and Ou, 1986). Also, year-round water mass with strong riverine character and high suspended sediment transport from north-east region of SCS and enter into the Beibu Gulf through Qiongzhou Strait off NRE (Su and Weng, 1994; Tang et al., 2003).

2.2. Sample collection

Water samples were collected from eighteen locations in NRE, during August, 2011 and October, 2012 (Fig. 1). A bucket was lowered from the bridge to collect water in the riverine region, whereas sampling in the estuarine waters were done by using a 5 L Niskin water sampler lowered from a mechanized boat. Estuarine stations were chosen along a salinity gradient from 0 to 35 psu (psu: practical salinity unit) during the neap low tide period (tidal height: 1.3–2.2 m). The stations were selected according to the observed spatial distribution of salinity in NRE and to cover a wide range of salinities. A series of typhoons cross over the Island during this study period (Fig. 1). Typhoons during 2011 were strong and crossed closely over the NRE, whereas typhoons in 2012 affected Leizhou Peninsula and marine regions off Hainan Island (Fig. 1). The typhoon Nock-ten (developed in coastal Philippine) with strongest wind speed (40 knots h^{-1}), crossed over the Hainan Island on July 29, 2011 and caused heavy rainfall (~220 mm within 12 h). This was the closest typhoon event occurred during August, 2011 campaign of this study, which was carried out after 3 days and during peak discharge (567 ± 45 m^3 s^{-1}) condition of NRE. The hydrographic parameters (temperature, salinity and pH) were measured directly with a multi-parameter probe (WTW MultiLine F/sets3) and the precision for temperature, salinity and pH measurement was 0.01 °C, 0.01 psu and 0.001 pH units respectively. Suspended particulate matter (SPM) was collected by filtering known volume of water through pre-weighed 47 mm GF/F filter papers (pre-combusted at 450 °C for 5 h) and later the filters were used for the analysis of POC, PN (refers to organic carbon and total nitrogen content of SPM respectively) and $\delta^{13}C_{org}$ (refers to stable carbon isotopic ratio of POC). About 10–15 L of water was separately filtered through 142 mm GF/F filters by using a pressure (N_2) filtration system, and then the filter was dried at 45 °C and used for lignin phenol analysis.

2.3. Bulk chemical analyses

The contents of POC and PN (relative precision ±5%) were determined using an Elemental Analyzer (Model: Vario EL III; Elementar Co.). The weight percentages of POC were analyzed after removing the carbonate fraction in vapour phase acidification (i.e. exposing the filters with concentrated hydrochloric acid (HCl) in desiccators for 24 h and then oven-dried at 45 °C). Weight percentages of PN

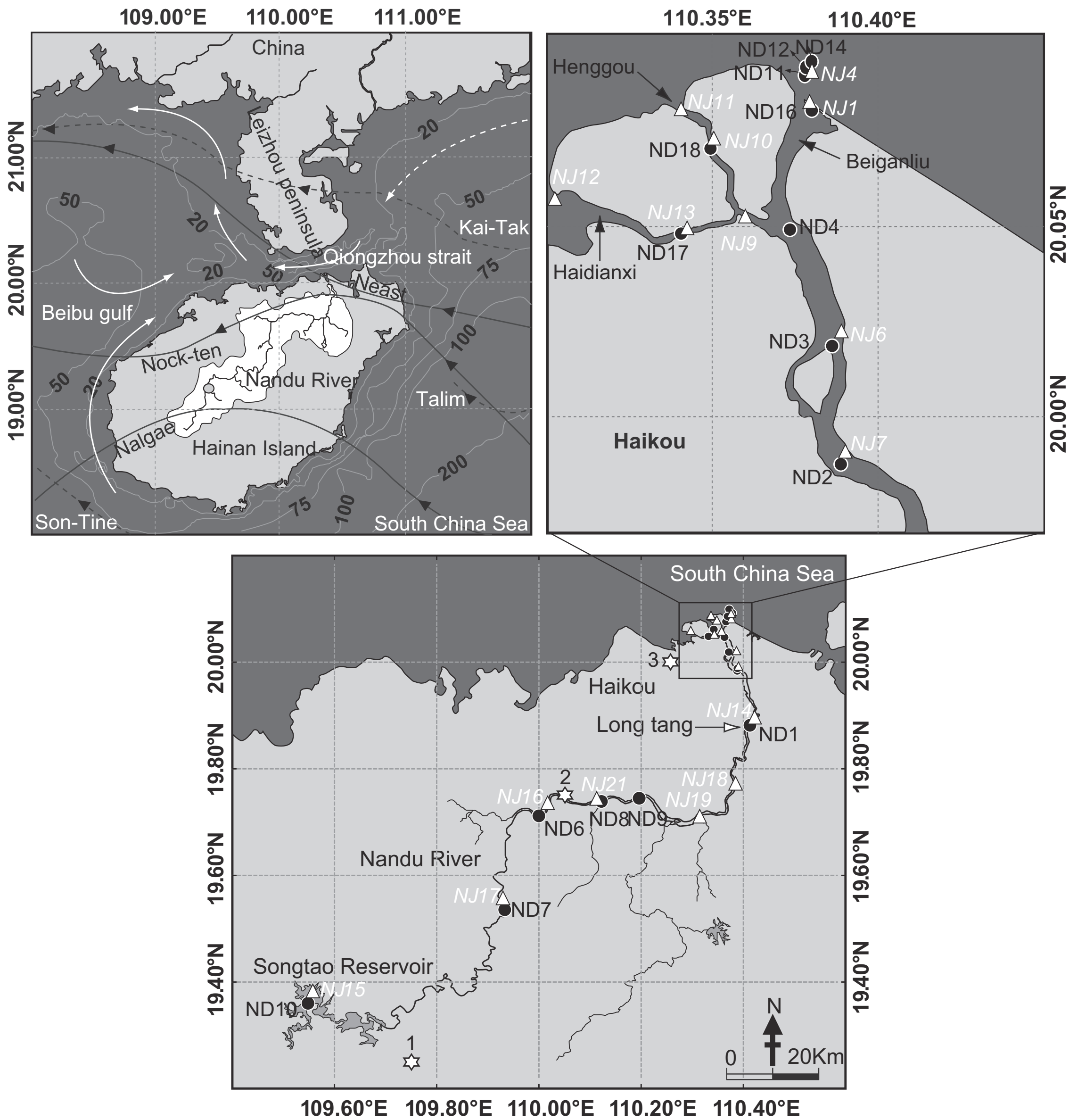

Fig. 1. Upper panel: Hainan Island and surrounding landmass across the South China Sea. Highlighted is the Nandujiang basin draining into the Qiongzhou strait. Arrow headed black lines indicated, traveling routes of different typhoons across the Hainan Island during year 2011 (solid) and 2012 (dashed). White arrows (solid) indicated general cyclonic circulation pattern and mixed coastal water mass (dashed) including water from large rivers such as Pearl River in the north as modified from Su and Weng (1994). Lower: Sampling location on Nandujiang and Songtao reservoir during August, 2011 (circles) and October, 2012 (triangles) with an enlarged view of estuarine region (upper right). Asterisk marks (1, 2 and 3) are the available rain gauge stations on Nandujiang basin where from daily as well as long term rainfall data obtained and used in this study.

were analyzed similarly, but without acidification. Stable carbon isotopic compositions of SPM were determined using a Flash EA 1112 Elemental Analyzer interfaced with continuous flow Isotope Ratio Mass Spectrometer (Model: DELTA plus/XP). Determinations of $N(^{13}C)/N(^{12}C)$ abbreviated herein as ($^{13}C/^{12}C$) and are expressed relative to conventional standards i.e. Vienna-Pee Dee Belemnite (V-PDB) for carbon (Coplen, 2011), as relative isotope-ratio difference, δ notation, defined as:

$$\delta X = [(R_{sample} - R_{standard})/R_{standard}] \times 10^3 \quad (1)$$

where $X = {}^{13}C$, $R = {}^{13}C/{}^{12}C$ and δ is expressed in parts per mil with symbol ‰ (Coplen, 2011). Analytical precision for $\delta^{13}C_{org}$, determined by replicate analysis of the same sample, was ±0.1‰.

2.4. Lignin phenol analysis

Lignin phenols were extracted from SPM matrices using microwave assisted cupric oxide (CuO) digestion technique (Goñi and Montgomery, 2000). The filter containing SPM (depending on POC content so as to have ~5 mg POC) were placed in acid-washed Teflon vessel, then added with copper (II) oxide powder, ferrous ammonium sulphate ($Fe(NH_4)_2(SO_4)_2$) and 8 M NaOH. The vessels were nitrogen-purged and placed in a Microwave Accelerated Reaction System (MARS5; CEM) at 150 °C for 90 min at pressure ranging between 55 and 70 PSI. Upon completion, the solutions were cooled to room temperature and a known amount of Ethylvanillin was added to it, which was checked later for analytical recovery. Recovery rate of Ethylvanillin was 95 ± 2% during this study. The phenolic monomers were extracted into 99:1 (volume ratio) of ethyl acetate/petroleum ether, dried and analyzed as trimethylsilyl derivatives of N,O-bis(trimethylsilyl)tri fluoroacetamide (BSTFA) and trimethylchlorosilane (TMCS) (99:1) by a Agilent 6890N gas chromatography (DB-1 column, FID) as detailed in Yu et al. (2011). The mean relative precisions for different CuO oxidation products were calculated by repetitive measurement of a specific sample and were found to be $\leqslant$10% in this study.

The lignin phenols (i.e. sum of syringyls (S = syringaldehyde (Sl) + acetosyringone (Sn) + syringic acid (Sd): particulars of angiosperm plant), vanillyls (V = vanillin (Vl) + acetovanillon (Vn) + vanillic acid (Vd): present in all terrestrial plants) and cinnamyls (C = p-coumaric acid (PCD) + ferulic acid (FAD): corresponds to non-woody tissues) are expressed either Λ8 (normalized with POC) and/or Σ8 (normalized with SPM concentrations) throughout this study (Hedges and Mann, 1979). A series of non-lignin products: *p*-Hydroxyphenols (*p*-hydroxybenzaldehyde (Pl) + *p*-hydroxyacetophenone (Pn) + *p*-hydroxybenzoic acid (Pd)) and 3,5-dihydroxybenzoic acid (DHBA) are also extracted through CuO oxidation procedure. Amongst, Pn is eventually derived from lignin, whereas Pl and Pd can also be derived from protein rich materials, so Pn/P ratio was used as an indicator for non-plant materials present in the SPM (Otto and Simpson, 2006). Similarly, the DHBA can either be generated from algae and/or from soil, therefore ratio of DHBA/V was used as an indicator of degradation state of plant OM in soil (Houel et al., 2006). The digenetic nature of plant tissues within the SPM was judged based on ratios $(Ad/Al)_V$ (i.e. Vd/Vl) and $(Ad/Al)_S$ (i.e. Sd/Sl) often used in literatures (e.g. Hedges and Mann, 1979). The S/V and C/V ratios are indicators of the abundance of angiosperm vs. gymnosperm and/or woody vs. non-woody tissues respectively (Hedges and Mann, 1979). During the degradation of plant OM, C phenols degrade faster than S and V due to the labile nature of former phenol; however the degradation pattern is highly ambiguous in the processes such as sorption and/or soil OM incorporation (da Cunha et al., 2001). Therefore to avoid misinterpretations lead by the use of S/V and C/V, the S, V and C were further normalized with Λ8 to produce lignin phenol vegetation index (LPVI), which firmly can indicate towards the actual digenetic character of lignin (Tareq et al., 2004). The LPVI was calculated as follows;

$$\mathrm{LPVI} = [\{S(S+1)/(V+1)+1\} \times \{C(C+1)/(V+1)+1\}] \qquad (2)$$

2.5. Statistical analyses

One-way analysis of variance (ANOVA) with repeated measures was carried out after Komogorov–Smirnov normality and homogeneity tests to understand the significance of spatial (river vs. estuary) and temporal (August, 2011 vs. October, 2012) variability in biogeochemical data from NRE. Pearson's two-tailed correlation coefficients (r) and regression coefficients (R^2) were used to determine the significant associations among variables. Overall data was subjected to Principal component analysis (PCA) to investigate the forcing of POM composition in NRE. Normalization of data was preferred before the PCA analysis and principal components with Eigen values >1 is only discussed, as they supposedly have strong influence on variances in a data matrix (Pradhan et al., 2009). In PCA, Λ8 was considered as a unique lignin phenol parameter, POC normalized yields of DHBA, Pl, Pn and Pd were used as indicators of non-lignin products, whereas $\delta^{13}C_{org}$, POC, PN and N/C were used to infer the POM quality, with supplementary parameters such as salinity, pH and SPM. All statistical analyses were performed using SPSS 10.0 and results were plotted using Grapher 4.0.

2.6. Mixing model

We determined the relative contribution of major OM pools exchanged along the NRE using Stable Isotope Analysis in R (SIAR), which is one of the more modern Bayesian mixing model (Parnell et al., 2010) and freely available in open source R-package (http://cran.r-project.org/). One of the striking feature of SIAR over the existing models (e.g. IsoSource, Least-square sense) is that, it deal with the variability in sources as well as mixtures in a more straightforward way by using uncertainties (e.g. mean and standard deviation) and estimate the proportional contributions from potential sources. SIAR has the advantage of including residual error term in the form of standard deviations and use Markov Chain Monte Carlo (MCMC) method, which produces simulations of potential values of proportional source contributions towards a fitting algorithm (Jackson et al., 2009; Parnell et al., 2010). It also includes the covariance structure that provides information about the functional ability of the model and discriminates between the sources, via diagnostic matrix plot of proportional source contributions. The matrix plot produces actual correlation coefficients of posterior distributions that represent the correlation between sources in such a way that, if two sources are very close to each other, then most feasible solutions could involve one or other sources but not both at the same time (Parnell et al., 2010). Also in the posterior distribution, these sources will show a negative correlation with lower correlation co-efficient, which indicate towards the ability of the model to isolate contributions from each source. The SIAR has been previously used in geochemical studies for the estimation of the proportional contributions from different OM sources in river–estuary and coastal systems elsewhere (Dubois et al., 2012; Sarma et al., 2014; Krishna et al., 2015).

In this study, the proportional contributions from different OM sources were obtained by using $\delta^{13}C_{org}$ and N/C and the results are discussed based on the median values of box-plots with 25%, 75% and 95% credibility internals. The use of Λ8 in our model was unfavorable, since OM sources such as plankton yield zero lignin upon CuO oxidation (Λ8 = 0; Rezende et al., 2010), which may affect the overall model output. The end member limits of potential sources were adopted from published literatures are discussed and reported later in the text.

3. Results

3.1. Hydrology and hydrographic characteristics of NRE

Due to the impact of typhoon (Nock-ten), average water discharge ($567 \pm 45\ m^3\ s^{-1}$) and rainfall (14 ± 6 mm day^{-1}) in NRE was extremely higher during August, 2011, as compared to the water discharge ($66 \pm 9\ m^3\ s^{-1}$) and rainfall ($\leqslant$1 mm day^{-1}) during October, 2012 (Fig. 2). Accelerated water discharge conditions were also noticed during September–October, 2011 and May–August, 2012 due to the impact of typhoons during those periods. The range of water temperature during August, 2011 (20.0–31.7 °C mean: 29.2 ± 2.8 °C) was much higher than that observed during October, 2012 (27.3–29.8 °C mean: 28.6 ± 0.5 °C). Variations in water temperature across NRE, during both the campaigns were not statistically significant (Table 1). The pH showed nearly identical ranges during August, 2011 (7.1–8.7) as well as in October, 2012 (6.8–8.1) campaigns and the spatial variation of pH was statistically significant ($p \leqslant 0.01$; ANOVA). During both the campaigns, pH values decreased from Songtao to Long tang (freshwater end point) and thereby increased towards estuarine region by mixing with alkaline/saline water. The average pH of estuarine water was lower during August, 2011 as compared to October, 2012 campaign (Table 1). The salinity ranges in the estuarine waters were 0–27.4 psu (8.3 ± 9.9 psu) during August, 2011 and 3.4–31.4 psu (22.0 ± 9.9 psu) during October, 2012, with a significant variation during both the campaigns as well as from river towards estuary ($p \leqslant 0.01$; ANOVA). Lower ranges of salinity indicated, higher

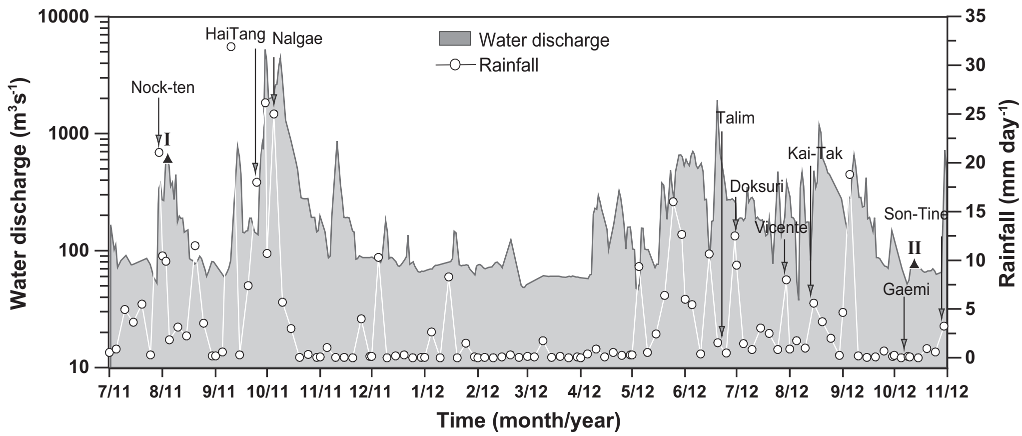

Fig. 2. Average daily freshwater discharge (scaled along left side of abscissa) of Nandujiang at Long tang terminal site. Average daily rainfall (scaled along right side of abscissa) is computed from the average values of rainfall at three key rain gauge stations (marked in Fig. 1) covering the entire Nandujiang basin. *Source:* Utah Climate Center (for 3; Haikou; CHM00059758) and http://cdc.cma.gov.cn (for 1 and 2). Roman numerals I and II denote the sampling dates of the present study. Each study was conducted over a 3–6 days period centreing the plotted date. Sampling during August, 2011 was characterized by high discharge due to typhoon Nock-ten crossed over Haina.

freshwater input to the estuarine region during August, 2011 (Fig. 3; Table 1). The range of SPM (6.4–79.3 mg/L) during August, 2011 was significantly higher than that observed during October, 2012 (1.8–62.8 mg/L). Roughly, the SPM concentrations were increased from Songtao to Long tang and then decreased towards estuarine region, without any significance (Fig. 3; Table 1).

3.2. Elemental and isotopic characteristics

The contents of POC observed at Songtao were extremely higher (13.5%; August, 2011 and 38.4%; October, 2012) as compared to the other stations in NRE. These values standout as outliers during statistical analysis, therefore average values reported here are excluding the POC values of Songtao. The average POC contents during August, 2011 (2.5 ± 0.6%) and October, 2012 (2.1 ± 1.7%) in NRE were nearly similar. Spatial variation of POC across NRE was statistically significant ($p \leqslant 0.01$; ANOVA; Fig. 3; Table 1) and the range of POC during August, 2011 (1.8–3.9%) was much lower than that during October, 2012 (0.6–6.7%). First-order POC export from NRE was calculated by multiplying POC content at Long tang (ND1 and NJ14) with average freshwater discharge during sampling period. POC export was found to be much higher (63 ± 17 t day^{-1}) during August, 2011 than October, 2012 (4 ± 1 t day^{-1}) campaign.

POC showed significant negative correlation with salinity during October, 2012 ($R^2 = 0.78$, $p \leqslant 0.05$; Fig. 4) as compared to that in August, 2011. Similarly the relationship between POC and SPM ($R^2 = 0.65$, $p \leqslant 0.05$) was significant during October, 2012 as compared to that in August, 2011. An extremely higher POC was associated with lower SPM at stations ND10 and NJ15 (Songtao), therefore was not included for the regression line. There was no marked POC difference found among estuarine channels (Haidianxi, Henggou and Beiganliu) during August, 2011. However, average POC contents of Haidianxi (0.9 ± 0.4%) and Henggou (0.9 ± 0.3%) were extremely lower as compared to that in Beiganliu (2.1 ± 0.4%) and other stations of NRE during October, 2012 (Table 1). There is an overall decrease in C/N (mol/mol) ratio from upstream to the freshwater end-point during August, 2011 as well as October, 2012 in NRE. The range of C/N (excluding Songtao) during August, 2011 (5.3–9.4) was nearly similar to C/N during October, 2012 (3.8–8.8). However the average value of C/N was slightly higher during August, 2011 (7.2 ± 1.5) than October, 2012 (5.7 ± 1.4) but without any significant variation along the NRE hydrograph (Fig. 3; Table 1). The $\delta^{13}C_{org}$ of NRE were ranged from −19‰ to −27‰ (mean: −24.1 ± 2.2‰) during August, 2011 and from −20.3‰ to −30.0‰ (mean: −24.7 ± 3.1‰) during October, 2012, without any significant difference during both the campaigns (Table 1). Spatial variation of $\delta^{13}C_{org}$ was statistically significant ($p \leqslant 0.05$; ANOVA), however $\delta^{13}C_{org}$ varied positively with salinity during August, 2011 ($R^2 = 0.92$, $p \leqslant 0.05$) as well as in October, 2012 ($R^2 = 0.89$, $p \leqslant 0.05$) campaign (Figs. 3 and 4). Across the SPM gradient, $\delta^{13}C_{org}$ values were largely scattered during both the campaigns on NRE (Fig. 4). The average $\delta^{13}C_{org}$ along Haidianxi (−22.1 ± 0.6‰) and Henggou (−20.8 ± 0.5‰) were extremely higher as compared to that in Beiganliu (−25.0 ± 0.8‰) and other stations during October, 2012 (Table 1).

3.3. Lignin phenols and ratios

The average concentrations of Λ8 and Σ8 in NRE were higher during August, 2011 (Λ8: 0.8 ± 0.6 mg/100 mg POC; Σ8: 2.3 ± 1.4 mg/10 g SPM) as compared to that in October, 2012 (Λ8: 0.6 ± 0.4 mg/100 mg POC; Σ8: 1.7 ± 2.6 mg/10 g SPM). The stations ND10 and NJ15 (Songtao) showed extremely lower Λ8 and Σ8. Concentrations of Λ8 and Σ8 significantly decreased from river towards estuary during both the campaigns in NRE (Fig. 3; Table 1). The inter-relationship of Σ8 with salinity was negative during both campaigns, whereas relationship between Λ8 and salinity was negative only during August, 2011, without any specific trend during October, 2012. Such condition is also associated with scattered variation of Λ8 along Haidianxi and Henggou during October, 2012 (Fig. 4). There was no significant relationship found among Λ8, Σ8 and SPM concentrations during both the campaigns (Fig. 4).

The range of C/V (0.16–0.54) and S/V (0.65–1.21) during August, 2011 was nearly similar to the range of C/V (0.25–0.68) and S/V (0.69–1.30) during October, 2012. The ranges of $(Ad/Al)_V$ was nearly similar during August, 2011 (0.3–1.8 mean: 1.0 ± 0.7) and October, 2012 (0.4–1.6 mean: 0.8 ± 0.4), with higher values observed in Haidianxi and Henggou during August, 2011. Similarly, the ranges of DHBA/V varied from 0.1 to 0.3 during both campaigns (Table 1). The Pn/P showed significant spatial and temporal variation along the NRE (Table 1). Values of Pn/P were higher during August, 2011 as compared to October, 2012 in all stations of NRE (Fig. 3). Distribution of LPVI showed a significant spatial variation from river towards estuary during both the campaigns (Fig. 3). Average LPVI values at Haidianxi (149 ± 21) and Henggou (211 ± 88) were lower during October, 2012 as compared to that in August, 2011 (Table 1). Significant interrelationship between

Table 1

Hydrography, bulk elemental, isotopic and biomarker parameters observed at different sampling locations on NRE during August, 2011 and October, 2012. *p* values resulted from ANOVA highlights the significance levels at 95% ($p \leqslant 0.05$) and 99.9% ($p \leqslant 0.01$) in this study.

Station	Latitude (°N)	Longitude (°E)	Region	Hydrographic properties				Bulk properties			Biomarker concentrations and indices								
				T (°C)	pH	S (psu)	SPM (mg/L)	POC (%)	C/N (mol/mol)	$\delta^{13}C_{org}$/‰	Λ8 (mg/100 mg POC)	Σ8 (mg/10 g SPM)	C/V	S/V	(Ad/Al)v	Pn/P	DHBA/V	P/(V + S)	LPVI
Period (August, 2011)																			
ND10	19.36	109.55	Songtao	30.7	7.7	0.0	6.4	13.57	36.0	−27.1	0.20	2.73	0.16	0.74	0.33	0.07	0.32	2.28	72
ND7	19.53	109.98	River	28.1	7.6	0.0	28.7	2.89	9.40	−25.2	1.98	5.74	0.45	1.06	0.72	0.17	0.08	0.36	432
ND6	19.73	110.00	River	27.6	7.4	0.0	54.0	3.21	7.65	−24.8	0.96	3.07	0.41	1.05	0.82	0.16	0.09	0.33	368
ND8	19.74	110.12	River	29.3	6.8	0.0	53.7	3.95	9.30	−24.7	0.89	3.52	0.54	1.14	0.74	0.13	0.12	0.42	590
ND9	19.74	110.20	River	29.9	6.9	0.0	79.3	3.45	7.86	−24.2	0.90	3.31	0.49	1.18	0.72	0.14	0.12	0.40	544
ND1	19.89	110.42	River	20.0	6.8	0.0	41.0	2.31	6.65	−26.0	0.90	2.07	0.40	0.99	0.84	0.09	0.13	0.83	327
ND2	19.99	110.39	Estuary	29.4	7.1	0.0	68.1	1.95	5.51	−25.8	0.92	1.79	0.30	0.93	0.72	0.12	0.15	0.66	210
ND3	20.02	110.39	Estuary	29.1	7.6	0.0	45.0	2.11	6.29	−25.4	1.24	2.61	0.42	1.21	0.61	0.10	0.16	0.48	451
ND4	20.05	110.38	Estuary	30.3	7.4	0.8	63.0	2.11	6.80	−24.7	0.84	1.78	0.39	1.11	0.68	0.09	0.16	0.70	373
ND17	20.05	110.34	Haidianxi	31.7	8.1	27.4	53.2	2.79	–	−19.0	0.12	0.34	0.46	0.93	1.81	0.06	0.21	3.05	380
ND18	20.07	110.35	Henggou	31.7	8.1	16.2	26.3	2.79	7.72	−22.8	0.09	0.25	0.34	0.65	2.97	0.04	0.20	4.40	156
ND16	20.08	110.38	Beiganliu	31.2	7.3	2.3	31.0	2.39	6.71	−25.2	0.70	1.66	0.26	0.81	1.49	0.11	0.10	0.82	143
ND11	20.09	110.38	Beiganliu	29.2	8.0	20.0	62.0	2.42	9.24	−19.7	0.26	0.63	0.34	1.03	0.69	0.10	0.11	1.31	276
ND12	20.09	110.38	Beiganliu	30.4	7.9	10.7	47.0	2.03	5.44	−22.3	0.58	1.18	0.30	0.90	1.15	0.07	0.09	1.40	200
ND14	20.09	110.38	Beiganliu	30.7	7.6	5.4	47.0	1.84	5.30	−24.6	1.75	3.22	0.35	1.02	1.41	0.08	0.09	0.84	285
Period (October, 2012)																			
NJ15	19.38	109.56	Songtao	28.7	7.6	0.0	1.8	38.4	201	−27.4	0.07	2.64	0.68	0.76	0.77	0.04	0.24	7.03	500
NJ17	19.53	109.98	River	27.3	7.5	0.0	5.9	6.75	8.78	−25.9	1.58	10.65	0.35	0.87	1.01	0.08	0.08	0.74	235
NJ16	19.73	110.00	River	28.6	7.3	0.0	11.6	5.19	3.79	−30.3	0.72	3.73	0.36	0.92	0.98	0.03	0.10	2.49	258
NJ21	19.74	110.12	River	29.3	7.1	0.0	13.7	4.14	7.70	−27.1	0.49	2.05	0.57	1.23	0.53	0.10	0.08	0.70	703
NJ19	19.71	110.32	River	28.4	7.6	0.0	50.3	1.36	5.06	−25.9	0.63	0.85	0.55	1.15	1.47	0.06	0.18	1.37	618
NJ18	19.77	110.39	River	28.2	7.2	0.0	55.2	2.35	4.81	−27.2	0.40	0.93	0.54	1.21	1.64	0.05	0.20	2.44	633
NJ14	19.89	110.42	River	28.7	7.1	0.0	62.8	1.41	6.40	−27.0	1.30	1.84	0.47	1.30	0.65	0.07	0.18	0.88	576
NJ7	19.99	110.39	Estuary	29.8	7.6	3.4	24.4	1.89	4.31	−28.2	0.64	1.21	0.29	0.90	0.53	0.07	0.09	1.06	189
NJ6	20.02	110.39	Estuary	29.2	7.9	10.7	17.8	1.76	5.69	−26.5	0.42	0.73	0.36	0.75	0.62	0.03	0.16	2.83	209
NJ13	20.05	110.34	Haidianxi	28.7	8.4	29.7	34.0	1.21	6.96	−22.5	0.55	0.66	0.28	0.70	0.91	0.04	0.11	2.96	134
NJ12	20.06	110.30	Haidianxi	28.8	8.7	30.6	33.3	0.63	5.06	−21.6	0.41	0.26	0.33	0.69	0.48	0.05	0.19	1.83	163
NJ9	20.05	110.36	Henggou	28.7	8.1	24.5	31.3	1.23	4.70	−21.2	0.31	0.38	0.35	1.10	0.51	0.05	0.20	1.95	312
NJ10	20.07	110.35	Henggou	28.4	8.1	30.7	46.2	0.85	4.96	−20.3	0.63	0.53	0.25	0.88	0.44	0.03	0.08	1.77	155
NJ11	20.08	110.34	Henggou	28.3	8.2	31.0	51.1	0.64	4.67	−21.1	0.74	0.45	0.27	0.85	0.64	0.07	0.09	0.78	166
NJ1	20.08	110.38	Beiganliu	28.2	7.9	12.6	12.3	2.46	6.74	−25.6	0.23	0.58	0.52	1.06	0.57	0.03	0.20	2.95	518
NJ4	20.09	110.38	Beiganliu	28.6	8.0	15.9	17.4	1.84	6.40	−24.4	0.16	0.29	0.44	0.88	0.52	0.04	0.33	3.22	329
ANOVA		*p* value		0.06	0.00	0.00	0.32	0.01	0.11	0.01	0.02	0.00	0.15	0.11	0.46	0.02	0.50	0.07	0.06
(space)		*F*		3.9	14.5	13.4	1.1	8.3	2.7	7.6	6.2	13.5	2.3	2.7	0.6	6.8	0.5	3.8	4.1
ANOVA		*p* value		0.71	0.10	0.05	0.02	0.47	0.18	0.28	0.46	0.96	1.00	0.47	0.15	0.00	0.93	0.09	0.59
(time)		*F*		0.1	3.0	4.3	6.7	0.5	1.9	1.2	0.6	0.0	0.0	0.5	2.2	18.6	0.0	3.1	0.3

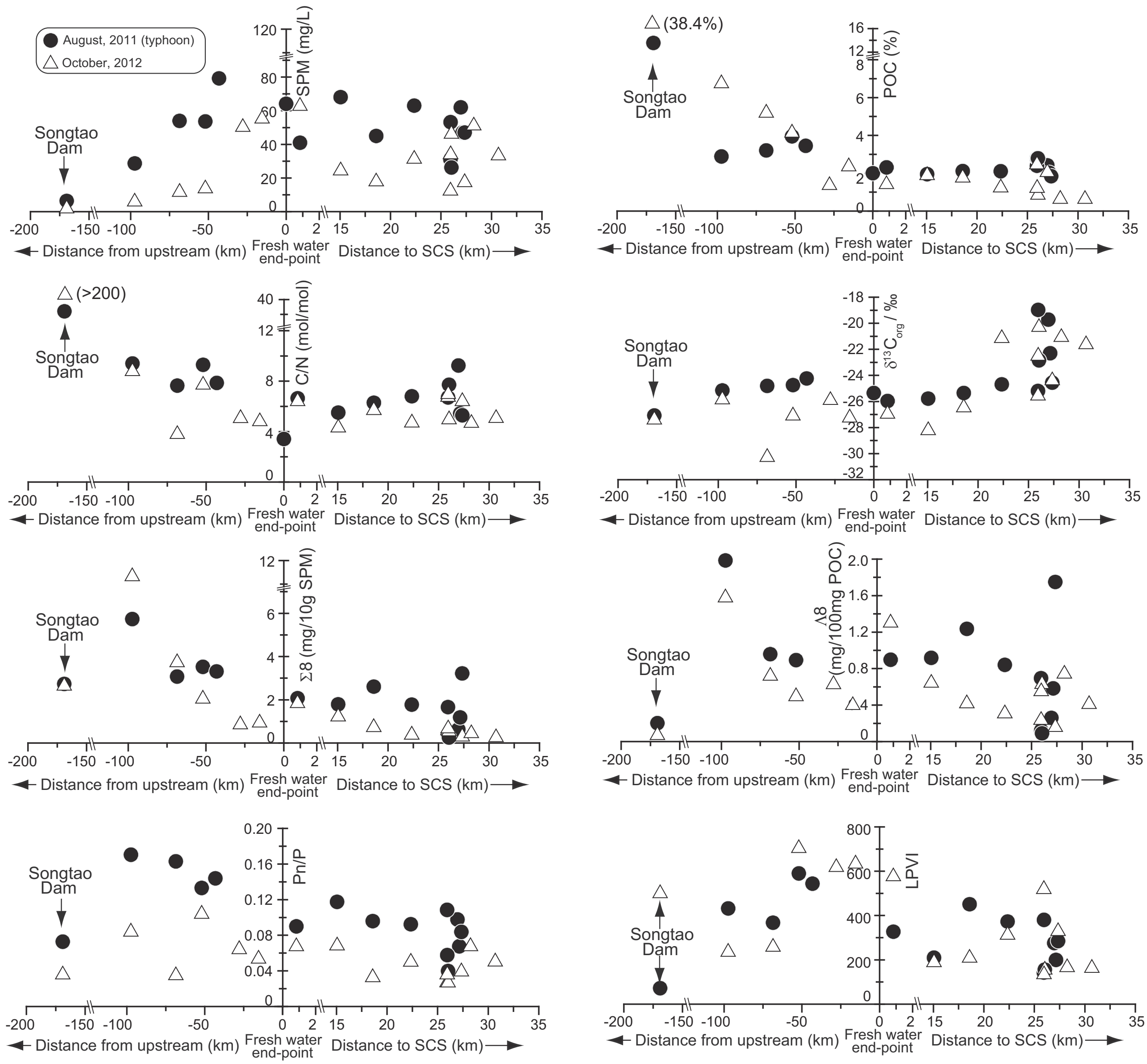

Fig. 3. Distribution bulk element, isotopic and lignin phenol characteristics in SPM collected during August, 2011 (circles) and October, 2012 (triangle) from Nandujiang. Limits of parameters are plotted in the *Y*-axis, difference in scale in *X*-axis are represented as riverine stations with distance from the upstream to the freshwater end point (Long tang) plotted in left and estuarine stations after Long tang plotted as their distance to the South China Sea in the right. Station names up to the Long tang point are marked in the first plot.

Λ8 and P/(V + S) was noticed during both the campaigns, whereas the relationship between Λ8 and DHBA/V was scattered (Fig. 5). Moreover, the abundance of lignin derived phenols (S + V + C) was higher during August, 2011 (55 ± 17%) as compared to the October, 2012 campaign (40 ± 14%) in NRE (Supplementary Table A).

3.4. Provenance of POM in NRE

The POM provenances for NRE were judged based on the interrelationship among N/C, $\delta^{13}C_{org}$ and Λ8 (Fig. 6). The C/N refers to PN, whereas N/C refers to POC, therefore, N/C ratio is more consistent than C/N ratio to be used in combination with $\delta^{13}C_{org}$, since both the parameters deal with POC and give statistical robust information (Perdue and Koprivnjak, 2007). In this study, the OM pools were limited to autochthonous (plankton) and allochthonous materials (plant and soil) transported across the NRE. The lowest $\delta^{13}C_{org}$ (−30.3‰) found in the riverine region of NRE during October, 2012 is in the range of $\delta^{13}C_{org}$ of freshwater algae (−33.2‰; Kao and Liu, 2000) and also close to the $\delta^{13}C_{org}$ of riverine phytoplankton reported for tropical region (e.g. −33.0‰; Hamilton et al., 1992; −26.6‰; Yu et al., 2010 and −31.2‰; Krishna et al., 2015), but much lower than typical marine plankton of SCS (−21.0‰ to −22.0‰; Liu et al., 2007b), tropical Sepetiba Bay, Brasil (−20‰; Rezende et al., 2010) and nearby Arabian Sea (−20.2‰; Fontugne and Duplessy, 1986). Therefore mean values −30.8 ± 3.1‰ and −20.8 ± 0.8‰ were chosen as end members for freshwater plankton and marine plankton respectively, with Λ8 values as zero (Goñi et al., 2003; Rezende et al., 2010; Pradhan et al., 2014a). The $\delta^{13}C_{org}$ values for end members such as plants (−29.0 ± 1.1‰) and soil (−23.1 ± 2.8‰) were adopted from literatures that report $\delta^{13}C_{org}$ of plant and soil end members in Wenchanghe, Hainan Island (Bao et al., 2013), nearby Pearl river

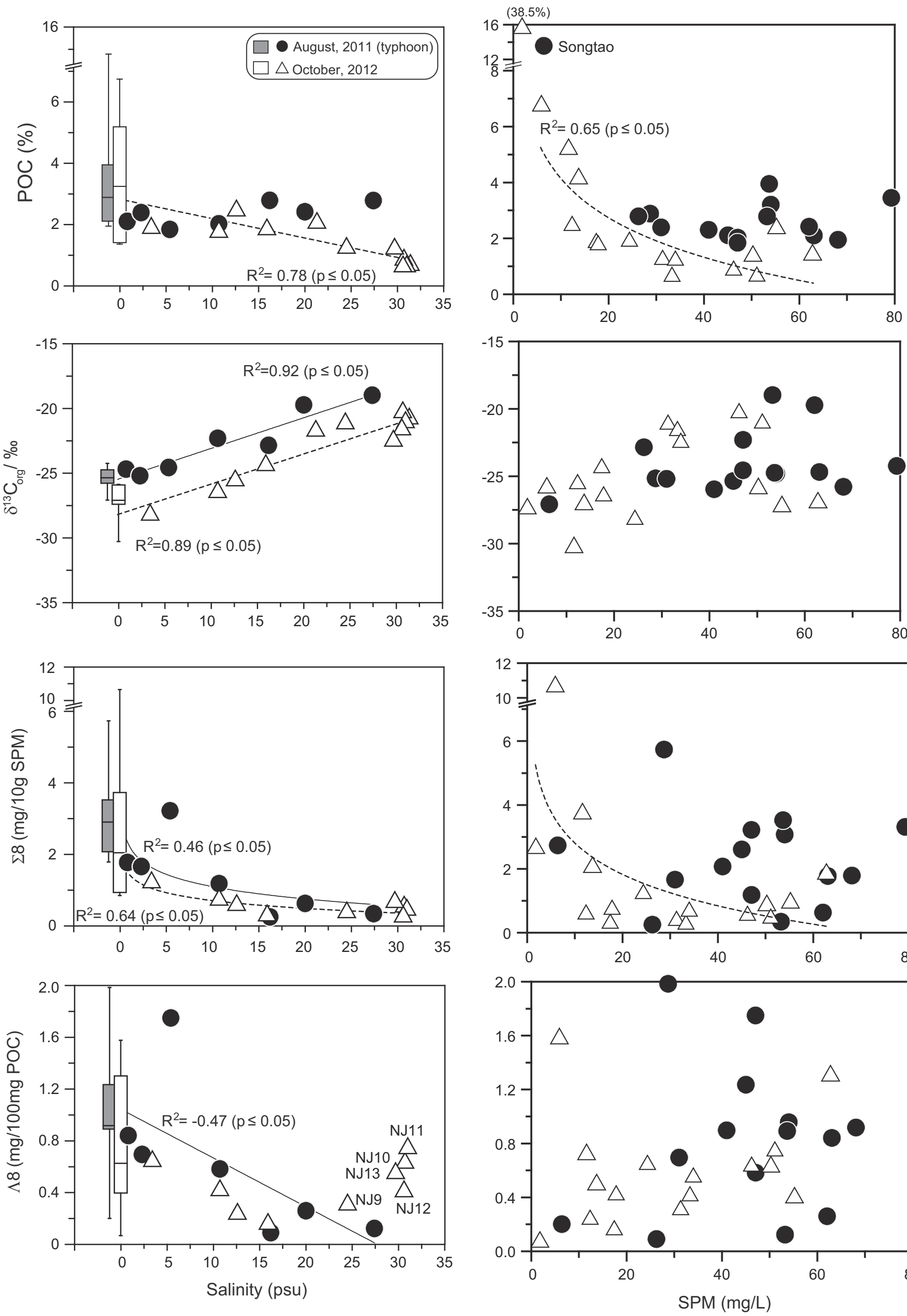

Fig. 4. Interrelationship of organic carbon contents, their isotopes and lignin phenols with salinity (left) and SPM (right) during August, 2011 (circles) and October, 2012 (triangles) campaigns in NRE. The values at riverine stations (with zero salinity) are displayed using box and whisker plot and their median (50% of data or middle of data) values were used for the regression line. Upper limit of box (above the median or horizontal line within box) represented upper quartile i.e. % of the data above median (50% of data or middle of data) and lower limit of box represented lower quartile i.e. % of the data less than the median values.

basin (Yu et al., 2010) and river basins along west coast of India (Jennerjahn et al., 2008; Pradhan et al., 2014a). The N/C of POM samples of this study were ranged between 0.1 and 0.2, which is higher than the N/C of fresh plant tissues of Hainan Island (0.01–0.04; Bao et al., 2013), tropical Indian rivers (0.01–0.06; Pradhan et al., 2014a; Jennerjahn et al., 2008), and Pearl river basin (0.03–0.07; Yu et al., 2010). However, the values are close to the N/C of soil in tropical Indian rivers (0.06–0.08; Pradhan et al., 2014a), Pearl river basin (0.07–0.10; Yu et al., 2010) and Kalada river, India (0.05–0.20; Jennerjahn et al., 2008), but lower or nearly similar to the N/C of freshwater plankton (0.13–0.22; Bordovskiy, 1965; Pradhan et al., 2014b) and marine plankton (0.13–0.2; Bordovskiy, 1965; Liu et al., 2007b; Rezende et al., 2010). The mean N/C values such as 0.02 ± 0.004, 0.05 ± 0.01, 0.22 ± 0.024 and 0.13 ± 0.007 were used for end members of plant, soil, river plankton and marine plankton respectively.

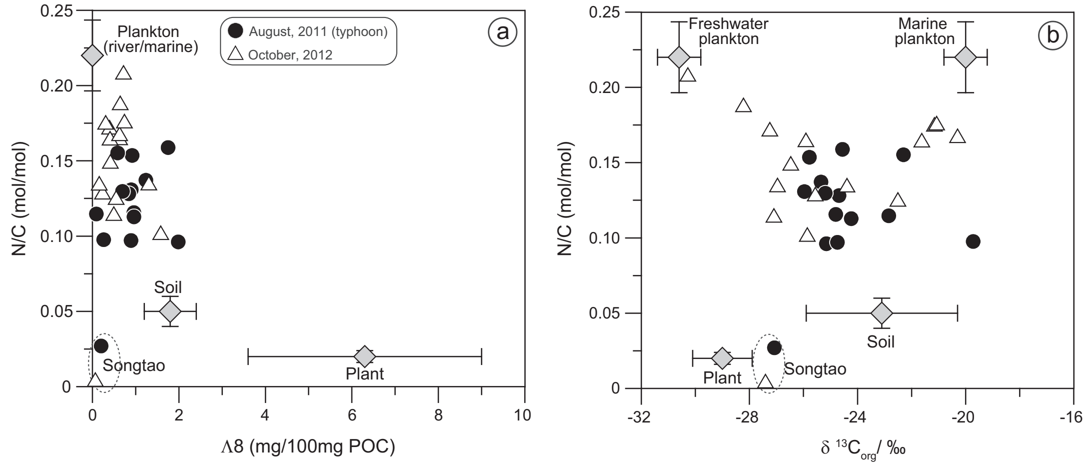

Fig. 5. Property–property plots between (a) Λ8 (mg/100 mg POC) and N/C (mol/mol), (b) $\delta^{13}C_{org}$/‰ vs. N/C (mol/mol), with end-member limits obtained adopted from literatures and discussed in Section 3.4.

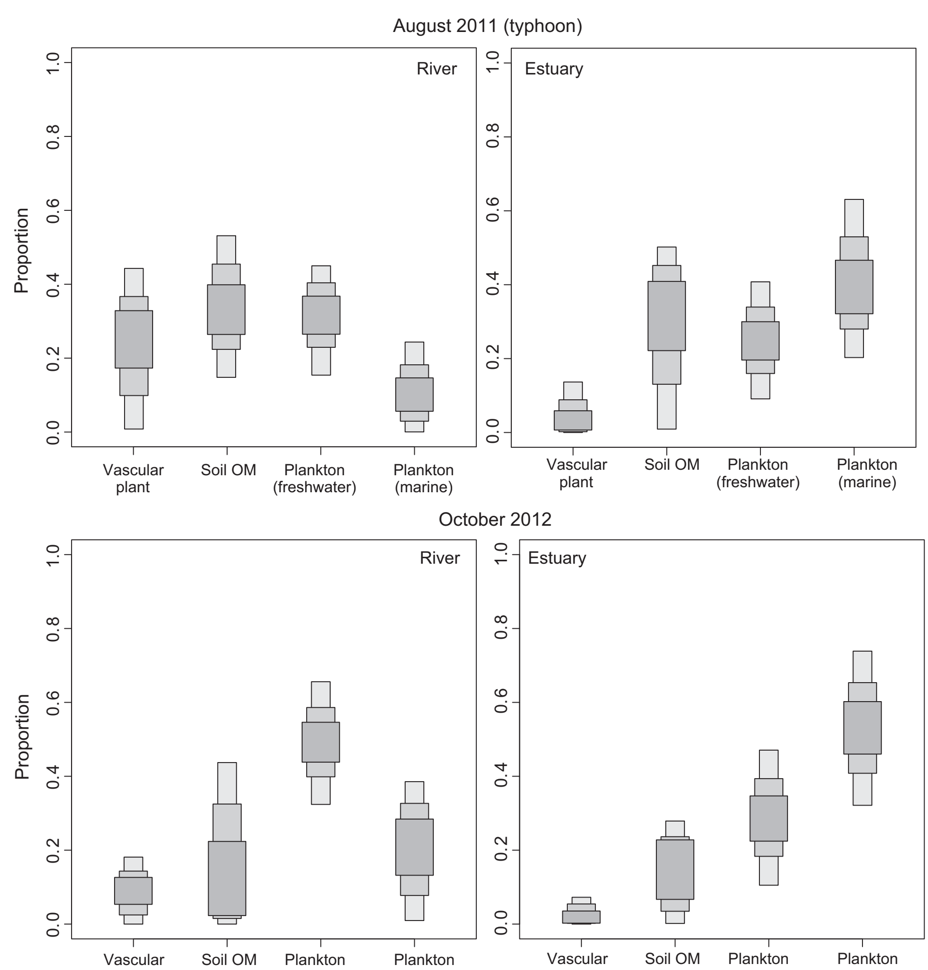

Fig. 6. Proportional contributions from source materials (plants, soil, freshwater plankton and marine plankton) to the riverine and estuarine POM of the NRE during August, 2011 (upper panel) and October, 2012 (lower panel).

Results from end member mixing model indicated that, in riverine region of NRE, contributions of soil OM (35%) and freshwater plankton (32%) to the POM was nearly similar during August, 2011. The estuarine region was predominated with OM contribution from marine plankton (44%), with equal contributions from soil OM (26%) and freshwater plankton (26%). Contrastingly, the OM contribution from freshwater plankton in the riverine region (51%) and marine plankton in estuarine region (56%) remained higher as compared to other sources during October, 2012 (Fig. 7).

3.5. Principal component analyses (PCA)

Results from PCA analysis indicated that, three factors had affected POM compositions in NRE, among which first two PCs are significant, therefore are discussed here. Factor 1 was associated with strong positive loadings of Λ8, Pn and DHBA, with strong positive correlations among them and appeared on right quadrant of PCA diagram (Fig. 8; Table 2). Factor 2 was associated with strong positive loadings of pH, salinity, SPM and $\delta^{13}C_{org}$ along with negative loadings of POC, PN and N/C (Table 2). Non-lignin products DHBA appeared within close proximity of lignin phenols. Samples from Songtao appeared on left quadrant and likely influenced by autochthonous OM sources. Stations of Haidianxi and Henggou channel were plotted in the left quadrant of PCA diagram during both the campaigns (Fig. 8).

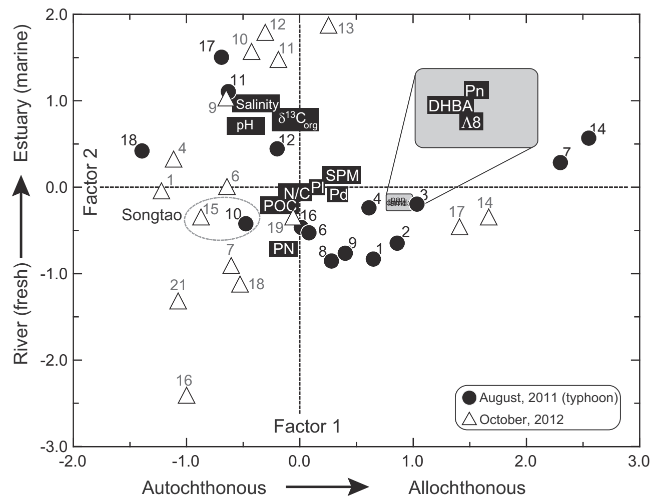

Fig. 8. Principal components diagram of variables merged with samples. Variables and their proximities are highlighted and the inset is an enlargement of the box. Factor 1 and Factor 2 are the two principal components. The numbers in black indicated stations of August, 2011 (followed by ND) and in grey indicated the stations of October, 2012 (followed by NJ) as marked in Fig. 1). Potential driving forces corresponds to the proximities of sample with parameters and between the two factors are marked on *X* and *Y* axis.

4. Discussion

Due to the effect of typhoon (Nock-ten), freshwater discharge in NRE during August, 2011 was higher in magnitude (nine times) than the freshwater discharge during October, 2012. The difference between SPM concentrations during both the sampling campaign was higher, although both the sampling were carried out during the wet seasons of NRE (Fig. 3; Table 1). Thus, the above mentioned conditions may correspond to POM composition alteration in NRE during this study. The distinct dry vs. wet climatic feature, with extreme precipitation during typhoon events strongly correspond to the POM composition alteration in NRE. Interestingly, such situations have also been observed in monsoon affected small river systems of India (Jennerjahn et al., 2008; Kessarkar et al., 2013) and typhoon affected small rivers of Taiwan (Kao and Liu, 2000) and Hainan Island (Wu et al., 2013).

4.1. Distribution and sources of POM in NRE

Due to effect of strong freshwater discharge and scouring of hinterland soil as well as bottom sediments, SPM concentrations were significantly higher during August, 2011 in NRE. The overall decrease of SPM towards sea is probably resulted due to the effects of sedimentation, mixing between riverine and marine waters and/or salt on particles aggregation under alkaline/saline conditions in the estuarine region (Fig. 3; Table 1). Combined effects of turbid condition and light inhibition on freshwater biological production might impinge on the scattered distribution of POC during August, 2011 (Ittekkot and Laane, 1991). Alternatively, dominant freshwater biological production and fairly lower SPM correspond to the negatively linear POC distribution during October, 2012 (Fig. 4). This may be due to POC generation from biological sources (plankton/algae), which often constitute lower SPM in river–estuary systems (Savoye et al., 2012). Overall, the significant POC variability along NRE hydrograph is resulted due to the combine effects of natural hydrological processes as well as potential anthropogenic activities, which is generally observed in tropical river systems (Ran et al., 2013). A fairly low amount of fresh plant derived OM (usually have C/N > 12) is present in the SPM of both campaigns, also the POM composition is largely varied along the NRE. This is indicated by lower C/N and highly scattered $\delta^{13}C_{org}$ values along the SPM gradient (Fig. 4; Table 1). The C/N ratios often get influenced by the mixing process of soil OM in highly dynamic river–estuary systems. Therefore, it cannot serve as a good tracer alone for POM source identification (Hedges et al., 1988). Transitional shift in OM contributions from terrestrial and marine sources in NRE was evidenced by positive correlations between $\delta^{13}C_{org}$ and salinity during both the campaigns (Middelburg and Nieuwenhuize, 1998). Lower Λ8 associated with higher C/N during both the campaigns in Songtao, might be related with diagenetic transformations of lignin phenols, which includes loss of methoxylated structural units of plant OM and delivered into the aquatic system through soil. Other possibility could be the presence of Fulvic acids, which have consistently lower lignin levels and higher C/N ratios

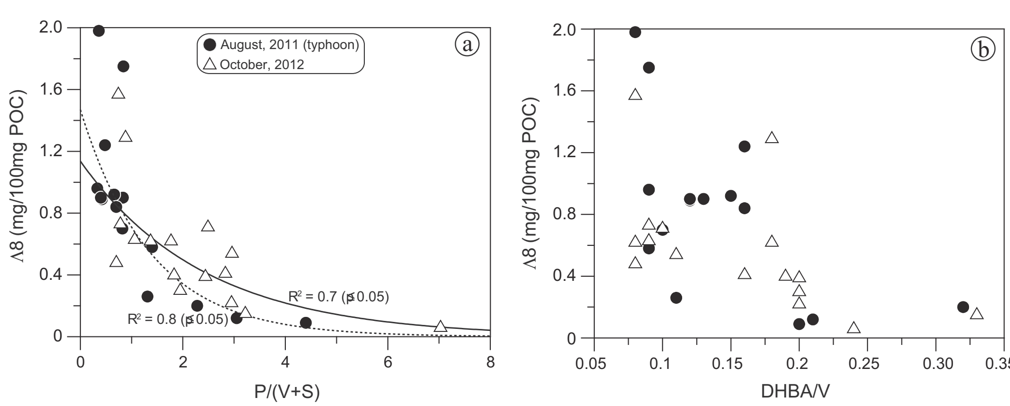

Fig. 7. Relationship between Λ8 and lignin phenol digenetic indicators (a) P/(V + S) and (b) DHBA/V in the POM from NRE during August, 2011 and October, 2012.

Table 2
Rotated mode factor analysis with loadings of principal components (PCs) on different biogeochemical parameters in NRE. Result of Pearson's two-tier correlation analysis among different parameters. Significant relationships are highlighted at 95% and 99% confidence levels.

Parameters	Factor 1	Factor 2	pH	Salinity	SPM	POC	PN	$\delta^{13}C_{org}$	N/C	Λ8	Pl	Pn	Pd	DHBA
pH	−0.349	0.817	1											
Salinity (psu)	−0.348	0.877	0.851**	1										
SPM (mg/L)	0.360	0.150	−0.260	0.028	1									
POC (%)	−0.169	−0.201	−0.107	−0.271	−0.447*	1								
PN (%)	−0.090	−0.695	−0.394	−0.456*	−0.289	0.009	1							
$\delta^{13}C_{org}$ (‰)	−0.089	0.875	0.614**	0.812**	0.386*	−0.309	−0.592**	1						
N/C (molar)	0.009	−0.104	0.058	0.108	0.145	−0.579**	0.233	−0.134	1					
Λ8 (mg/100 mg POC)	0.926	−0.177	−0.417*	−0.424*	0.250	−0.237	0.157	−0.217	0.168	1				
Pl (mg/100 mg POC)	0.179	0.031	0.158	0.143	−0.139	−0.180	0.280	−0.203	0.508**	0.207	1			
Pn (mg/100 mg POC)	0.907	−0.139	−0.347	−0.385*	0.191	−0.240	0.192	−0.213	0.192	0.912**	0.336	1		
Pd (mg/100 mg POC)	0.356	−0.047	0.096	0.006	−0.258	−0.110	0.472**	−0.254	0.388*	0.406*	0.796**	0.576**	1	
DHBA (mg/100 mg POC)	0.889	−0.172	−0.398*	−0.422*	0.311	−0.241	0.012	−0.291	0.217	0.837**	0.252	0.777**	0.349	1

Factor 1 (Eigen value: 5.3; variance: 40.5%); Factor 2 (Eigen value: 2.8; variance: 38.6%) and Factor 3 (Eigen value: 2.3; variance: 12.9%).
* Correlation is significant at the 0.05 level.
** Correlation is significant at the 0.01 level.

than humic acids in tropical river and lake systems (Ertel et al., 1986). Moreover, large variability in the plant OM source was evidenced by the scattered relationships among SPM, Σ8 and Λ8 during both campaigns in NRE (Fig. 4). The apparent dilution of lignin phenols due to freshwater biological production was predominant during October, 2012 as compared to August, 2011, which is indicated by lower Pn/P ratios as well as lower abundance of lignin phenols during October, 2012 campaign (Fig. 3; Table 1). Both Σ8 and Λ8 do not co-vary with each other across the salinity gradient in NRE, which is indicated by their distinct relationships with salinity during both campaigns (Fig. 4). Therefore it is likely that, the lignin entrained into NRE either during typhoon influenced fluvial erosion, dominated with soil OM (allochthonous) and/or diluted due to the mixing of freshwater phytoplankton (autochthonous). A weak interrelationship of Λ8 and salinity associated with apparently lower Pn/P is indicative of the second process to be active during October, 2012 (Table 1), whereas plant OM contribution from erosion products of soil is dominated during August, 2011 (Rezende et al., 2010). Estuarine region of NRE is affected by year-round strong tidal currents that cause sediment re-suspension. During the typhoons, turbid water from far north-eastern region of SCS carries large amount of suspended materials in west ward direction (i.e. towards Beibu; Fig. 1) through the Qiongzhou strait (Su and Weng, 1994; Tang et al., 2003). Such conditions expectedly enhance POM from existing floodplains especially along Haidianxi and Henggou channel during lower freshwater input conditions. The POM enhanced from floodplains is significantly altered in nature, which is indicated by higher $\delta^{13}C_{org}$ and lower Λ8 and LPVI values along Haidianxi and Henggou channel, also consistent with observations in other river systems (Alin et al., 2008; Ellis et al., 2012).

Vascular plants, plankton (freshwater and/or marine) and soil OM are primary contributors to the POM mixture in NRE, which is indicated by limited variation of C/N, with values close to plankton (4.6–7.5; Bordovskiy, 1965) during both campaigns. Nevertheless it is not an easy task to provide valuable insights into the provenance of POM only based on C/N, which undergoes into alteration during biological mixing. Lignin phenols are exclusively derived from terrestrial vascular plants therefore, when used along with $\delta^{13}C_{org}$ can provide semi quantitative estimate of OM contributions from plant and/or plankton/algae sources. The lower values of Λ8 found during both the campaigns indicated majority of POM is contributed from mixture of soil and/or algal sources (Fig. 5). POM contribution from freshwater phytoplankton is dominant in the riverine stations during October, 2012, which is also indicated by lower $\delta^{13}C_{org}$ values in those stations. Relatively fair amounts of lignin (0.5–0.8 mg/100 mg POC) present in riverine stations of NRE during both the campaigns, with higher values during August, 2011 are related with the OM contributions from mixture of degraded vascular plants (mobilized through soil) to the riverine stations (Fig. 6). Above condition is also supported by the fact that typhoon induced strong freshwater discharge during August, 2011 have caused scouring of soil OM into riverine stations, whereas apparently steady flow condition during October, 2012 caused production of biological OM in riverine region. The potential human activities such as deforestation in the past might trigger the soil erosion (42.6×10^{9} kg yr^{-1}) processes along NRE (Wangcheng, 1983; Zeng et al., 2009).

Similarly, the headwater of NRE (Songtao) is surrounded with rich diversity of Eucalyptus forest and rubber plantation, which is confined up to the upper reaches, whereas lowland regions are occupied with urban cities and industrial setups. Therefore, lower Λ8 values across Haidianxi and Henggou resulted due to less plant cover in lower NRE and/or digenetic alterations of plant OM in floodplain soils, which get mixed with estuarine SPM during lateral exchange processes (Alin et al., 2008). Estuarine POM is dominated with OM from marine plankton can be attributed to the higher biological production in the nutrient rich marine water across Qiongzhou strait (Liu et al., 2007b). This process is further affected by lower hydrologic stage during October, 2012 due to which the average OM contribution from marine plankton remained higher in estuarine waters, whereas typhoon induced strong hydrologic condition caused delivery of fairly good amount of soil OM (26%) and freshwater plankton OM (26%), which corresponds to apparently lower marine plankton OM (44%) in estuarine waters during August, 2011 (Fig. 6).

4.2. Diagenetic state signatures of POM in NRE

The range of C/V and S/V indicated that, SPM of NRE was composited mixture of plant OM that are dominated by angiosperm leaf and wood tissues during both campaigns (Hedges and Mann, 1979). The diagenetic state of plant OM in composited SPM from NRE was judged based on $(Ad/Al)_V$ ratios of lignin phenol. The $(Ad/Al)_V$ showed higher values in river–estuary systems (0.5 to >2) due to processes such as humic substance input and/or sorption of plant OM in mineral soil (Houel et al., 2006). Alternatively, $(Ad/Al)_V$ in fresh plant tissues with rich in lignin produce lower ratios (0.3–0.4; Opsahl and Benner, 1995). The ranges of $(Ad/Al)_V$ found during both campaigns in NRE, suggests that, primarily plant OM in SPM has undergone into advanced degradation state (Table 1). However, it is not clear that the degradation has taken place prior to or after plant OM input into NRE. The degradation of plant OM in NRE estuary could be influenced by other processes such as hydrodynamic sorting and/or selective transport, albeit not necessarily occur within SPM, which is indicated by higher values of $(Ad/Al)_V$ along the channel stations during August, 2011 as compared to the October, 2012 (Table 1). Generally, the coarser particles retain more lignin and deposited easily at delivery site, whereas fine particles, presumably enriched in acidic compounds and enhanced into SPM of estuarine stations during strong hydrologic conditions in August, 2011. Such trend was not clear during October, 2012, probably due to mixing of biological OM that is devoid of lignin. The ranges of DHBA/V (0.1–0.3) were higher (0.05; Houel et al., 2006) and identical during both the campaigns, thus effectively indicate towards dominance of soil OM in NRE. Goñi and Hedges (1995) proposed that the likely precursors of DHBA are not lignin, rather than tannins and other flavonoids, which have hydroxyl groups in alternative positions on aromatic ring. Furthermore, the relative increase of tannins in mineral soils might lead to degradation and/or humification of plant OM, which generally increase the DHBA/V ratio (Houel et al., 2006). However, limited variability of DHBA/V ratios and no significant relationship between DHBA/V and $(Ad/Al)_V$ ($R^2 = 0.04$; $p \geqslant 0.05$; not shown), during both campaigns probably corresponds to specific lignin degradation process in soil, which was then delivered into riverine/estuarine SPM. Specific lignin degradation mainly due to side chain oxidation, lead to demethylation of V and S constitutes, therefore increase the P/(V + S) in CuO products (Dittmar and Lara, 2001; Houel et al., 2006).

The LPVI decreases with diagenesis given that C and S phenols are preferentially removed relative to V phenols during degradation (Rezende et al., 2010). However, there was no significant relationship found between LPVI and $(Ad/Al)_V$ ($R^2 = 0.03$; $p \geqslant 0.05$; not shown), also between DHBA/V and P/(V + S) together reinforcing the idea of selective sorption and transport effect, which presumably caused disproportionate degradation of plant OM along NRE during both campaigns. Furthermore, significant spatio-temporal variations of Pn/P ratios indicated an obvious input of autochthonous OM, specifically during October, 2012 owing to freshwater biological production. In addition, apparently higher Pn/P observed during August, 2011 are mainly derived from lignin constituents (Dittmar and Lara, 2001). Therefore, in NRE, conservative mixing of OM pools from lignin rich plants and lignin poor soil is affected by typhoon during August, 2011 and steady hydrologic stage characterized with freshwater biological OM during October, 2012. This is further evidenced with significant relationship between $\Lambda 8$ and lignin ratios P/(V + S) and DHBA/V during both campaigns (Fig. 7). However, the relationship between $\Lambda 8$ and DHBA/V is apparently weaker, therefore may not solely represent the differences in the degradation of soil OM, but the strong relationship of P/(V + S) with $\Lambda 8$ definitely confirms the role of selective transport of plant OM, presumably related to the particle size, density and lignin input along the NRE basin.

4.3. Factors affecting OM composition in NRE

In this study, the primary factor controlling POM compositions in NRE can be termed as fluvial erosion, which indicates allochthonous OM input from terrestrial region and evidenced with negative loadings of pH and salinity along with positive loading of DHBA (Table 2). The DHBA has both autochthonous (brown macroalgae) and allochthonous (soil and humic substances) source (Goñi and Hedges, 1995). In NRE, DHBA is derived from allochthonous sources (i.e. soil) during both campaigns, which is evidenced by strong positive correlation between lignin phenols and DHBA (Pearson's $r = 0.83$; Table 2). The secondary factor controlling POM compositions in NRE can be termed as biological production (freshwater and/or marine), which influence bulk chemical characteristics (POC, PN and $\delta^{13}C_{org}$) and indicated by positive loadings of salinity and pH (Pearson's $r = 0.85$; Table 2). The factor fluvial erosion have had significant influence on riverine POM composition during August, 2011 as most sampling stations are associated with lignin phenols in right quadrant, which represents POM origin from allochthonous sources. Similarly, the factor biological production predominated during October, 2012, which is evidenced by the association of most riverine stations in the left quadrant representing POM origin from autochthonous sources (Fig. 8). The POM from the stations in Haidianxi, Henggou and Beiganliu showed mixed signals of both autochthonous and allochthonous OM input during August, 2011 as well as October, 2012 owing to varying freshwater input and strong hydrodynamic condition of Qiongzhou strait.

Climate dynamics and steep catchment of small mountainous rivers in tropical region affects the quantitative as well as qualitative capacities of continental OM transport during high discharge condition and due to perennially high terrestrial primary production. These two factors predominantly affects POM compositions, exported by small rivers to be naturally bio-available in marine region and affect the carbon cycling of coastal ocean (Alin et al., 2008; Hilton et al., 2008). The changes in frequency of precipitation and rapid flushing rates in association with higher recurrence of natural disasters (e.g. tropical cyclones) and potential land-use changes (e.g. deforestation; intensive fertilized agriculture) significantly influence the POM composition in NRE as revealed in this study.

5. Conclusion

This study represents comprehensive isotopic and lignin phenol characterization of POM sources during two different wet seasons in NRE across the Hainan Island. Based on changes in elemental, isotopic and lignin phenols of POM, we draw the following conclusions:

(i) short term heavy precipitation owing to typhoon during August, 2011 had caused significant changes in freshwater discharge pattern as well as POM composition across NRE;
(ii) results of PCA analysis indicated that fluvial erosion and biological production are two important factors correspond to variability in the OM contribution from allochthonous and autochthonous sources in NRE;
(iii) POM constituted nearly similar OM input from soil (35%) and freshwater plankton (32%) during August, 2011, in contrast POM dominated with OM from freshwater plankton (51%) during October 2012 in riverine regions of NRE. In the estuarine region, POM constituted dominant inputs from marine plankton during August, 2011 (44%) and October, 2012 (56%) as compared to other sources;
(iv) results from an end member mixing calculation indicated that POM in riverine regions of NRE sourced from nearly similar inputs of soil OM (35%) and freshwater plankton (32%) during August, 2011. In contrast, POM was dominantly contributed by freshwater plankton (51%) during October 2012. POM in the estuarine waters constituted predominant inputs of marine plankton during August, 2011 (44%) as well as October, 2012 (56%); and
(v) lignin phenol compositions suggested that, SPM from NRE constitutes OM mainly from previously degraded angiosperm plant tissues and the conservative mixing of OM pools from lignin rich plants and lignin poor soil is associated with their selective transport mechanism affected by typhoons over Hainan Island.

The nature of compositional POM changes due to natural and potential anthropogenic activities across the rivers of Hainan Island underscores the need for further research into carbon cycling in these rivers in inter-annual scale to understand their importance in carbon cycling of SCS. This study will serve as first baseline study in this region and a model study for tropical rivers on island affected by natural disasters and potential human activity.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.jhydrol.2016.01.046.

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