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Variations of total dissolved iron and its impacts during an extreme spring flooding event in the Songhua River

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A B S T R A C T

Total dissolved iron (TDFe) is a controlling factor in primary productivity in marine ecosystems, hence the variation of riverine TDFe output is attracting an increasing attention. Spring flooding is a key hydrological process in cold regions and the extreme spring flooding event (ESE) is thought to influence the output of TDFe considerably. In 2013, an ESE arose in the Songhua River, to reveal its impact on TDFe output and its species, water samples were collected in the river during this ESE as well as normal spring flooding period (NSP) in 2014 and 2015 to analyze the concentration and species of TDFe as well as other basic parameters. The speciation analysis was conducted by filtration and ultrafiltration methods. The results indicated the concentration of TDFe did not represent a significant difference during ESE, with an average concentration of 0.28 mg/L in ESE and 0.30 mg/L in NSP. The steady trend of TDFe concentration can be contributed to the limited intensity and duration of erosion by the snowmelt runoff and the construction of hydraulic works. Whereas, ESE intensified TDFe output increasing from 27.93 ton/day during NSP to 48.56 ton/day during ESE due to its high discharge. The species of TDFe was dominated by Fe(III) with the molecular weight lower than 10 kDa. The correlation analysis indicated that content and property of DOM were the main controlling factors of TDFe species. Furthermore, the dynamics and migration of TDFe were accompanied by the nutrient and heavy metal transport which may potentially influence the water quality in the river basin.

1. Introduction

As one of the most abundant element in the Earth's crust, iron widely exists in different aquatic ecosystems, participating in various physiological and ecological processes (Boyd and Ellwood, 2010). Recently, dissolved iron was revealed to act as a controlling role in primary production in the ocean areas known as high-nitrate and low-chlorophyll (HNLC), where abundant dissolved macronutrients (e.g. N, P, Si) cannot be sufficiently utilized due to the lack of it (Martin and Fitzwater, 1988). Consequently, as a main source in the ocean, the variations of riverine dissolved iron output is attracting increasing attention (Labatut et al., 2014).

The Sea of Okhotsk is characterized by a highly productive ocean region, contributed to the sufficient dissolved iron transported from the Amur River (Suzuki et al., 2014). As the largest tributary, the Songhua River was indicated to be a crucial source of iron (Wang et al., 2012). It has been intensively studied on the features of total dissolved Fe (TDFe) concentration and species during summer flooding season (July–August) and normal flow period (September–October) in the Songhua River (Pan et al., 2011; Levshina, 2012), however, characters of its output during spring flooding season were rarely reported.

Spring flooding is a key hydrological process in cold regions, the snowmelt is not only of considerable importance in water supply of soil and river systems, but also influences physicochemical properties of the river (Gao et al., 2015). Moreover, due to global climate change, the recession rate has been enhanced and snowfall has increased coupled with frequently occurred snowstorms, which may alter many aspects of hydrology during this period, such as flooding, erosion, hydrochemical and output (Stepanauskas et al., 2000; Ollivier et al., 2006; Vilímek et al., 2015), likewise the Songhua River Basin (Wang and He, 2013). During the winter season from 2012 to 2013, Northeastern China suffered from intensive and frequent snowfall, the precipitation reached 78 mm in the upper stream of Songhua River Basin (the Second Songhua River), and over 70 mm in the basin of mainstream, which was approximately 110% more than the annual average snowfall. The monthly average discharge at Harbin achieved 1290 m^3/s in April and 1910 m^3/s in May in 2013, which was 20% and 80% beyond the mean value (1067 m^3/s) during the normal spring flooding period (NSF), respectively. It ranked the second maximum water level and discharge ever recorded since 1960s, thus, the 2013 spring flooding event can be defined as an extreme spring flooding event (ESE) (Eisenbies et al., 2007). Various studies have reported an increase trend of

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dissolved iron flux during spring flooding in different boreal rivers (Pokrovsky and Schott, 2002; Andersson et al., 2006; Sarkkola et al., 2013). It was also revealed that TDFe output in the Songhua River increased significantly during the flooding events induced by the extreme rainfall events (Guan et al., 2015, 2016). Therefore, it is hypothesized that extreme spring flooding event may also alter the trend of TDFe output in the Songhua River. So the current study is aimed (1) to observe the characteristics of TDFe output and its species during ESE, (2) to investigate the critical controlling factors, and (3) to discuss its potential impacts on the water quality of the Songhua River.

2. Materials and methods

2.1. Study site

The Songhua River Basin (41°42′–51°38′ N, 119°52′–132°31′ E) is located in northeastern China (Fig. 1). The basin of Songhua River takes account for approximately 30% of the Amur River basin, with a total area of 5.61×10^5 km^2; the annual average temperature and precipitation are 3–5 °C and 400–750 mm, respectively; the frost-free period is about 130 days per year; icebound season is from November until April in the next year and the spring flooding season last from April to May (Meng and Mo, 2012). Harbin City and Jiamusi City are located along the middle and lower reaches of the Songhua River, respectively, and Tongjiang City is situated at the confluence of the Songhua and the Amur Rivers (Fig. 1).

2.2. Sample collection

Water samples were collected according to standard methods from Water Quality-Technical Regulation on the Design of Sampling Programs (HJ 495-2009, China EPA) conducted at Road Bridge (45°45′46″ N, 126°34′60″ E), Songpu Bridge (45°47′51″ N, 126°39′03 E) in the Harbin City, Fusui Bridge (47°14′14″ N, 131°58′16″ E) in Jiamusi City and Sanjiangkou (47°41′24″ N, 132°30′20″ E) in Tongjiang City (Fig. 1) from April to May during the 2013 ESE and NSP in both 2014 and 2015. The collected water sample of 1.0 L for each sampling event were immediately stored in a portable refrigerator (4 °C) and transported to the laboratory for further treatment and analysis.

2.3. Sample analysis

In current study, fractions of TDFe were determined by the ultrafiltration method (i.e. cross-flow filtration, CFF) established in previous researches (Pan et al., 2011). The samples were firstly filtered through Whatman GF/F membrane (Whatman, England) which are acid-cleaned and pre-combusted at 450 °C for 4 h to analyze TDFe concentration as well as other aquatic parameters. Then CFF was conducted to divide TDFe into $TDFe_L$, $TDFe_M$ and $TDFe_H$ with the molecular weight lower than 10 kDa (10 kDa MWCO PES), between 10 and 50 kDa (50 kDa MWCO PES), and larger than 50 kDa in the filtrate (Whatman GF/F), respectively. The recovery rate was 94.7–104.0% and the detection limit was 0.002 mg/L. All the pretreated samples were acidified to pH 2.0 to reduce Fe oxidation, and then stored at 4 °C prior to further analysis. In current study, colloidal Fe is defined as the sum of $TDFe_H$ and $TDFe_M$; $TDFe_L$ consists of Fe(II) and Fe(III), Fe(II) concentration were measured using ET7406 Fe Tester (Lovibond, Germany) with *o*-Phenanthroline spectrophotometric method in situ and Fe(III) concentration were calculated as the difference between $TDFe_L$ and Fe(II) (Borman et al., 2010).

The concentrations of TDFe as well as different molecular weight iron and Mn were determined by a flame atomic absorption spectrophotometer (GBC 932, GBC Scientific Equipment Pty, Ltd, Braeside, Australia), Fe and Mn concentrations of each samples were calculated from the corresponding linear regression equation ($R^2 > 0.999$) using six different dilutions of Fe and Mn standard solution (Standard Material Center of China). pH was measured by a portable pH meter (Rex, INESA Scientific Instrument, Shanghai, China). Dissolved organic carbon

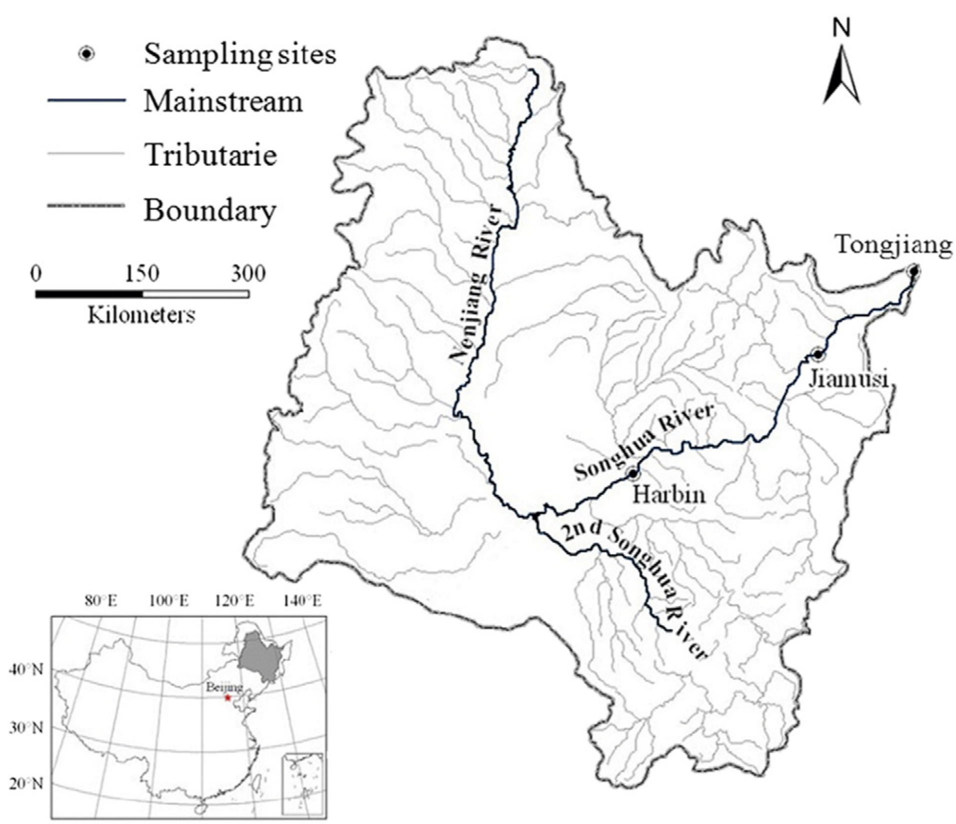

Fig. 1. Location of sampling sites.

(DOC) and dissolved inorganic carbon (DIC) were analyzed using a TOC-V_{CPH} (TOC-V_{CPH}, SHIMADZU, Kyoto, Japan). The concentrations of ammonium (NH_4^+-N), nitrate (NO_3^--N), phosphate phosphorus (PO_4^{3-}-P) in each sample were measured using a discrete auto analyzer (Mode Smartchem 200, AMS, Rome, Italy). The humification index (HIX) was obtained by a Fluorescence Spectrophotometer (Cary Eclipse, Varian, Palo Alto, USA). HIX was defined as the ratio of the area under the emission spectra between the quarter (Σ435–480 nm) and (Σ300–345 nm) at an excitation of 254 nm (λex = 254 nm, slit = 5 nm; λem = 290–490 nm, slit = 5 nm; scan speed = 600 nm/min).

2.4. Data analysis

All statistical analyses were conducted using SPSS 20.0 statistical software (SPSS Inc., Chicago, USA). By *Q-Q* probability plot analysis, the data did not show a normal distribution pattern. Therefore, Spearman correlation analysis was performed to analyze the relationship between the concentrations of different Fe species and the other analyzed parameters. One-way analysis of variance (ANOVA) was performed to compare the differences of concentrations monitored during different periods. The difference were considered significant if $P < 0.05$.

3. Results and discussion

3.1. Basic aquatic quality parameter set

The statics of water quality parameters of the water samples are listed in Table 1. The values of pH, EC maintained steadily and did not show significant difference between ESE and NSP ($P > 0.05$). So did NO_3^-—N concentrations ($P > 0.05$) which averaged at 0.99 mg/L in ESE and 0.83 mg/L in NSP. The concentrations of DIC ($P < 0.01$) and Mn ($P < 0.05$) in NSP dropped significantly compared with that monitored in ESE, whereas PO_4^{3-}—P as well as NH_4^+—N concentrations demonstrated an increase trend in NSP ($P < 0.05$). The concentrations of NH_4^+—N averaged at 0.23 mg/L in ESE and 0.28 mg/L in NSP; the mean content of PO_4^{3-}—P was 0.22 mg/L in ESE and 0.23 mg/L in NSP.

DOC maintained steadily and did not present significant difference ($P > 0.05$) during ESE which fluctuating from 9.09 to 10.91 mg/L during ESE and 9.16 to 11.03 mg/L during NSP, with a mean value of 9.88 mg/L and 10.03 mg/L, respectively. The similar pattern was also reported in the Elbe River that DOC varied slightly during a spring flood event (Karrasch et al., 2004). In natural waters, HIX values usually varies between 2 and 18, which generally increases with processes of decomposition or sorption onto mineral surfaces, i.e. high values of HIX (10–16) indicate an increasing humification degree and the presence of organic matters with high molecular weight and aromaticity, whereas low values (<5) demonstrate fresh autochthonous dissolved organic matter (DOM) derived from plant biomass as well as animal manure (Huguet et al., 2009; Catalán et al., 2014). In current study, majority of HIX values fluctuated in a range from 6 to 10, implying DOM has an important terrestrial originated humic character while weak recent autochthonous component.

3.2. Concentration and output of TDFe

The concentration of TDFe vary between 0.16 and 0.42 mg/L during ESE and 0.19 and 0.45 mg/L during NSP, averaged at 0.28 and 0.30 mg/L, respectively (Fig. 2). During ESE, the concentration of TDFe did not represent a significant difference ($P > 0.05$). Whereas, in our previous researches, TDFe concentration in the Songhua River increased significantly during the flooding events induced by extreme rainfall due to the intensive territorial runoff and hydrological connection to the wetlands as a main pool of iron in the river basin (Guan et al., 2015, 2016). The stabilization of TDFe content during ESE may owe to the relatively limited intensity and duration of soil erosion and leaching by the snowmelt runoff (Heikkinen, 1994). Besides, the reduction of Fe (hydr)oxides is catalyzed by microorganisms (Frohne et al., 2014), the low temperature of river water (below 4 °C) may limit the microbial activity and retard the reduction and release of Fe. Additionally, the construction of large hydropower plants and reservoirs has given rise to considerable influence on the hydrological and hydrochemical regimes in the Songhua River. Currently, there are 34 large-scale reservoirs located along the river, with a total storage capacity of 41.5 billion m^3. Consequently, they may serve as geochemical barrier for TDFe, since it is a common feature that TDFe is accumulated in the reservoir as a result of its precipitation as colloids (Shamov et al., 2014). Therefore, TDFe that migrated from the tributaries into the river may be blocked in the reservoirs leading to a steady TDFe concentration in the Songhua River during ESE. However, the values were significantly higher ($P < 0.01$) in comparison with that (0.18 mg/L) monitored in summer flooding season (Wang et al., 2012). This pattern is agreed to other researches, indicating TDFe output in the spring flooding season may contribute largely to the annual output in boreal rivers (Heikkinen, 1994; Pokrovsky and Schott, 2002; Andersson et al., 2006).

The concentration of TDFe in current study is comparable with that in the boreal blackwater rivers which are commonly characterized by a high TDFe content (Regina et al., 2010; Kritzberg et al., 2014). This may be attributed to the geological conditions of the Songhua River Basin surrounded by orogenic zones which are filled with iron-rich terrigenous sedimentary, volcanogenic, and intrusive rocks. The mountain is composed of the basic, ultrabasic, and intermediate rock whose iron contents range from 5% to 9%, and accumulative terraces are developed along the river covered with thick layers of clay that is rich in iron (the content of iron varies from 6% to 10%) (Kulakov et al., 2010; Wang et al., 2012). Such iron content is higher than the average iron abundance in the earth's crust (5%) and soils (3.8%). Moreover, over the past 50 years, the natural wetlands had gone through a large-scale reclamation into cultivated land, such as paddy fields (Wang et al., 2015). Intensive human activity had changed hydrological characteristics and iron distribution and species in the soil (Cary and Trolard, 2008). Agricultural drainage played a crucial role in material transportation from paddy fields or uplands to downstream water bodies (Fritsch et al., 2009; Zhang et al., 2014). Thereby, it may also exert a significant influence on the migration of TDFe, resulting in an increasing TDFe concentration in the river (Pan et al., 2011).

The average flux of TDFe during spring flooding was 48.56 ton/day in ESE and 27.93 ton/day in NSP. The comparatively high output of TDFe in

Table 1
The aquatic quality parameters in the Songhua River.

	Statistics	pH	EC	DOC	DIC	NH_4^+-N	NO_3^-N	PO_4^{3-}-P	Mn	HIX
			μS/cm	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	
ESE	Range	6.89–7.31	137–146	9.09–10.91	9.23–10.93	0.11–0.35	0.53–1.86	0.08–0.38	0.03–0.07	5.95–8.78
(N = 45)	Mean ± SD	7.16 ± 0.15	140 ± 3.64	9.88 ± 0.41	10.14 ± 0.44	0.23 ± 0.07	0.99 ± 0.31	0.22 ± 0.09	0.05 ± 0.01	7.39 ± 0.92
NSP	Range	6.97–7.29	138–143	9.16–11.03	8.25–10.35	0.17–0.45	0.50–1.20	0.12–0.39	0.02–0.06	6.08–9.25
(N = 50)	Mean ± SD	7.12 ± 0.11	140 ± 2.07	10.03 ± 0.49	9.17 ± 0.71	0.28 ± 0.08	0.83 ± 0.24	0.23 ± 0.07	0.04 ± 0.01	7.67 ± 1.10

ESE: extreme spring flood event; NSP: normal spring flood period; EC: electric conductivity; DOC: dissolved organic carbon; DIC: dissolved inorganic carbon; HIX: humification index

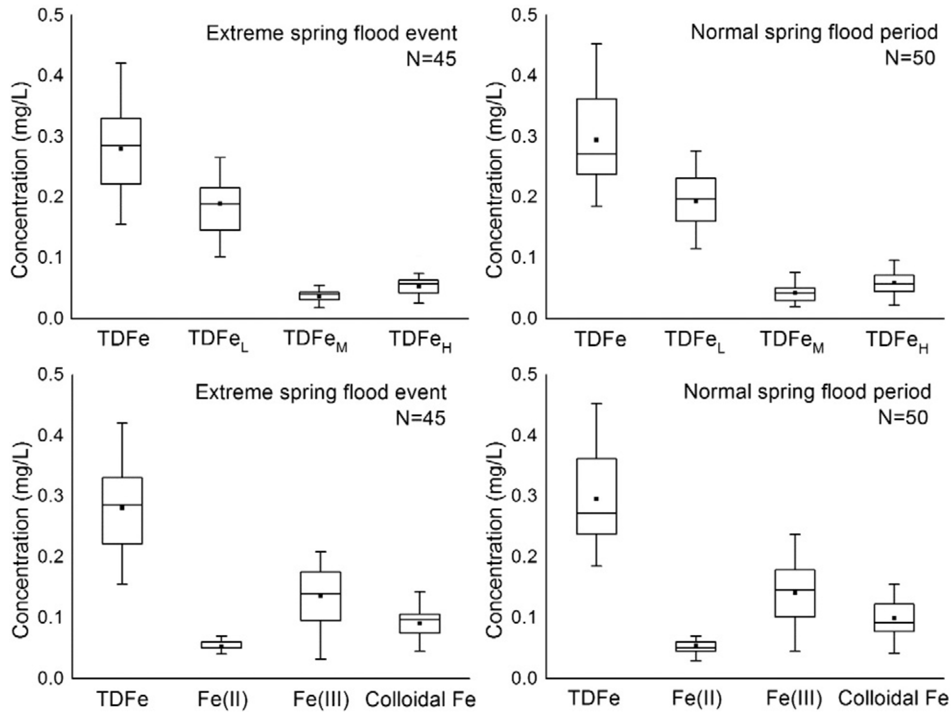

Fig. 2. Total dissolved iron (TDFe) concentration and its species during extreme spring flood event and normal spring flood period.

ESE can be contributed to the alteration in hydrological condition, i.e. the increment of discharge.

3.3. The species of TDFe

As shown in Fig. 2, the species of TDFe during ESE did not represent significant difference with that during NSP, it was both dominated by TDFe$_L$ form in the following order: TDFe$_L$ > TDFe$_H$ > TDFe$_M$, in which Fe(III) was the major species, accounting for approximately 50% of TDFe concentration.

In current study, a positive correlation was observed between TDFe and DOC (Fig. 3). DOM has high selectivity and affinity for Fe(III), in which the humic acids are assumed to be the active organic component in binding Fe(III), so that the greatest mass of truly dissolved Fe(III) present as Fe(III)-DOM complexes form and it can further control the solubility of TDFe (Aiken et al., 2011; Shimizu et al., 2013). Consequently, it is a typical feature that a high concentration of DOM in boreal rivers and the species of TDFe dominated by Fe(III)-DOM complexes and colloids (Björkvald et al., 2008; Ilina et al., 2013). A remarkable correlation between DOC and TDFe was also documented in extreme rainfall events in the Songhua River (Guan et al., 2016) as well as other rivers (Neal et al., 2008; Jiann et al., 2013) intimating the presence of DOM may exert a significant influence on the content and output of TDFe. However, it remains virtually unknown that the chemical structure of Fe(III)-DOM complexes represented by both soil fulvic DOM and autochthonous microbial exometabolites or DOM transformation products (Ilina et al., 2013). HIX indicated a weak recent autochthonous component of DOM, implying that the fulvic DOM with an important humic character may contribute largely in forming Fe(III)-DOM complexes (Ou et al., 2009). It is suggested that fulvic acids of high molecular weight and large size fractions may be the crucial chelation of Fe (Kritzberg et al., 2014). This is in agreement with a positive correlation between TDFe and HIX during ESE and NSP in this study (Fig. 4), indicating that fulvic DOM with high molecular weight and aromaticity may be the key carrier of TDFe in the river. Thus, the comparable high concentration of TDFe during the spring flooding season can owe to the rising DOC content compared with that (5.04 mg/L) documented in summer flooding season (Wang, 2011).

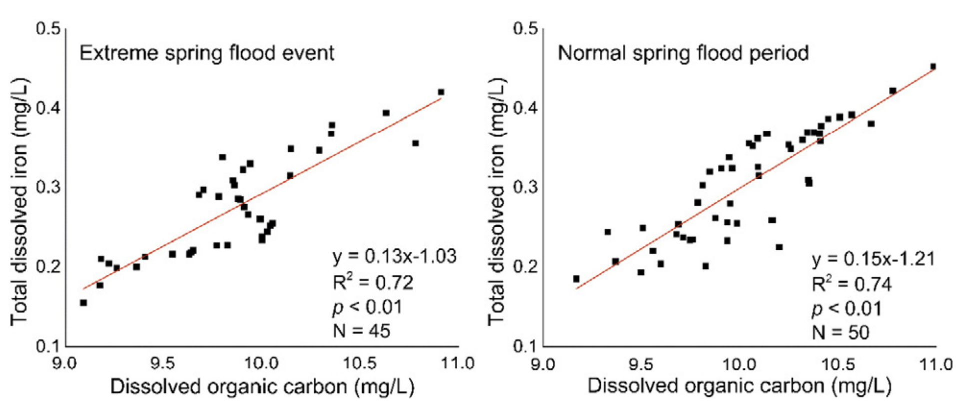

Fig. 3. Linear regression of total dissolved iron and dissolved organic carbon.

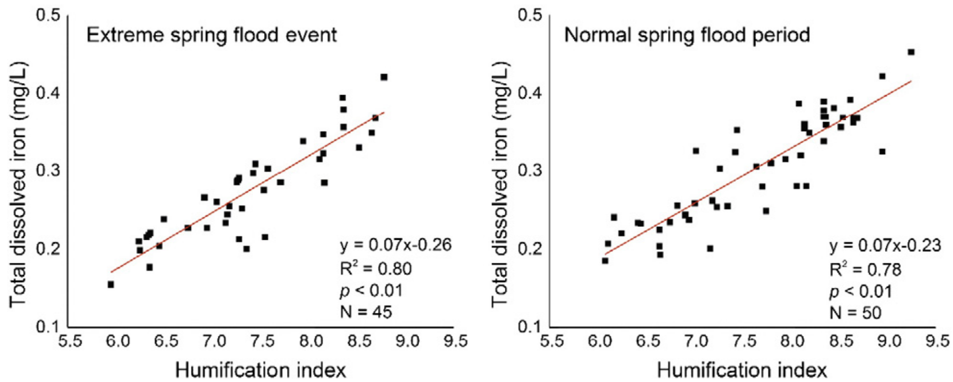

Fig. 4. Linear regression of total dissolved iron and the humification index.

3.4. Potential effects of TDFe flux during ESE

In aquatic ecosystems, the redox processes between Fe(II) and Fe (III) are central for iron cycling in aquatic environments and the biogeochemical cycle of iron is strongly associated to other elements in aquatic environments.

The Songhua River runs through the key grain producing region in China where excessive fertilizer application has become the principal source of N and P (Yang et al., 2010). As a consequence, the river could receive a large amount of nutrients leached from farm lands. While Fe dynamic has been intensively reported to provide a crucial sequence of nutrient cycling in aquatic ecosystems, since the behavior of N and P is strongly associated and/or controlled by the redox dynamic of Fe (Yang et al., 2012; Kleeberg et al., 2013). In current study, a significant positive correlation was observed between PO_4^{3-}-P and TDFe, colloidal Fe as well as Fe(III); NO_3^--N also weakly correlated to TDFe and Fe (III) during spring flooding season (Table 2). A positive relationship between nutrients (N and P) and dissolved metals (Fe and Al) was also observed in the Yangtze Huaihe region of China (Zhang and Shan, 2008). On the other hand, some studies had shown that P bound with Fe and Al was the main species in sediments of freshwater ecosystems (such as multi-pond system), accounting for 30%–60% of total P content, which directly affected the P exchange across the sediment-water interface (Tang et al., 2010). Moreover, the migration nutrients is controlled by the redox cycling between soluble Fe(II) and indissoluble Fe(III). Phosphate can be precipitated with Fe(III) oxyhydroxide under oxidizing conditions, and the co-precipitates will be released into the water column under reducing conditions (Voegelin et al., 2013). While the microorganisms can also take Fe(III) as electron acceptor to produce energy via oxidizing NH_4^+-N in sediments (Clément et al., 2005).

In aquatic ecosystems, the overall oxidation rate of Fe(II) can be accelerated at the presence of DOM, such as humic acids, moreover, the generated organic Fe(III) complexes may slow down the formation of iron colloids and stabilize Fe(III) in dissolved fraction in fresh waters (Pullin and Cabaniss, 2003; Henneberry et al., 2012). The interaction between Fe and DOM also provides a crucial sequence in various geochemical processes including the transportation of trace metals which is remarkably affected by the adsorption to surfaces of in-stream iron particles, affecting the fate of various heavy metals such as Pb, Cr, and As that are bound to the iron (oxy)hydroxides (Neubauer et al., 2013; Blake et al., 2015). The growth and agglomeration of iron (oxy)hydroxides will results in larger particles that deposit in the slow-flowing sections of streams, lakes, or estuaries (Karlsson and Persson, 2012). Whereas, Compared with iron (oxy)hydroxides, Fe(III)-DOM complexes are less likely to aggregate and settle, therefore, the complexes are supposed to transport over a longer distance (Sundman et al., 2014). Moreover, floodplain soils often act as net sinks for heavy metals and other pollutants, whereas the wet-dry cycles during the flooding may convert the reducing and oxidizing conditions in the soils, which could facilitate the mobility of the redox-dependent pollutants via alteration of Fe/Mn dynamics (Frohne et al., 2014; Shaheen et al., 2014a). Shaheen et al. (2014b) found redox potential played an essential role in heavy metal and metalloid release in the periodically flooded north Nile Delta rice soils, correspondingly, the concentrations of Co, Ni, and Se in soil solution were correlated with Fe, Mn and DOM dynamics, the increase of metal and metalloid solubility in the reducing conditions may be attributed to the reduction of Fe/Mn oxides during the flooding, while DOM might be a controlling factor of temporal dynamics of soluble metals. Therefore, the migration and dynamics of TDFe during ESE and NSP may be accompanied by the heavy metal transport, exerting a potentially influence on the water quality in the river basin.

Table 2
Spearman correlation between iron species and aquatic quality parameters.

	Period	NH_4^+-N	NO_3^--N	PO_4^{3-}-P	DIC	Mn	HIX
Fe(II)	Extreme spring						
Fe(III)	flood event		0.55[b]	0.75[a]	−0.53[b]	0.52[b]	0.84[a]
Colloidal Fe	(n = 45)			0.73[a]		0.52[b]	0.55[a]
TDFe			0.54[b]	0.78[a]	−0.50[b]	0.51[b]	0.80[a]
Fe(II)	Normal spring flood	0.54[b]					
Fe(III)	period (n = 50)		0.47[b]	0.67[a]	−0.44[b]		0.86[a]
Colloidal Fe				0.58[a]			0.48[b]
TDFe			0.49[b]	0.68[a]		0.44[b]	0.78[a]

TDFe: total dissolved iron; DIC: dissolved inorganic carbon; HIX: humification index
[a] Correlation is significant at the 0.01 level (2-tailed).
[b] Correlation is significant at the 0.05 level (2-tailed).

4. Conclusion

This study illustrates that the spring flooding season is in an important position to TDFe transportation in the Songhua River. The concentration of TDFe averaged at 0.28 mg/L in ESE and 0.30 mg/L in NSP, with an output of 48.56 and 27.93 ton/day, respectively. Unlike that during the extreme flooding events induced by the rainfall, the concentration of TDFe did not represent an increasing trend during ESE, owing to the limited intensity and duration of erosion by the snowmelt runoff and the construction of large hydropower plants and reservoirs. Whereas, TDFe output largely depends on hydrological condition, i.e. the intensive and frequent snowfall may intensify TDFe output during spring flooding season. The species of TDFe was dominated by Fe(III) at $TDFe_L$, accounting for nearly 50% of total concentration during both ESE and NSP, the content and property of DOM were the main controlling factors of TDFe species. Furthermore, the dynamics and migration of TDFe are accompanied by the nutrient and heavy metal transport which may potentially influence the water quality in the river basin.

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