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Hydrological management affected dissolved organic matter chemistry and organic carbon burial in the Three Gorges Reservoir

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ABSTRACT

With the linkage between dissolved organic matter (DOM) and the characteristics of natural ecosystem assessed extensively, the properties of DOM in reservoirs, the typical human interrupted ecosystems, have been focused on in recent years, which is critical for the understanding of human impacts on watershed ecosystems and carbon cycling. This study aims to analyze the effect of hydrological management on the DOM chemistry and organic carbon burial in Daning River tributary of the world's largest Three Gorges Reservoir (TGR). Based on the application of a combined approach including bulk geochemical analyses, optical spectroscopy, and ultrahigh-resolution Fourier transform ion cyclotron resonance mass spectrometry, various sources of DOM (terrestrial, anthropogenic, and autochthonous sources) were revealed. An increasing trend of terrestrial and recalcitrant DOM was observed along the upstream to downstream transect of Daning River tributary, which was mainly caused by the water intrusion with a higher terrestrial and recalcitrant signature from mainstream to tributary resulted from hydrological management of TGR. Integrated with the analysis of sedimentary organic matter in Daning River tributary in the past decade (after the construction of TGR), our work suggests that organic carbon burial in the reservoir could be enhanced by hydrological management-induced variation in DOM chemistry. Further studies are needed to better constrain the effects of damming reservoirs on carbon cycling considering their booming all over the world.

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1. Introduction

With economic development all over the world, the construction of reservoirs increased dramatically over the past several decades, especially in developing countries. The number of reservoirs increased by more than 70%, and the total reservoir storage volume increased from about 3500 to nearly 6200 km^3 since 1980 (World Commission on Dams 2000; Maavara et al., 2017). As the most far-reaching human modifications of the flows of rivers, reservoirs take on various kinds of functions including flood control, power generation, navigation, drinking water supply. However, the construction and operation of reservoirs lead to significant changes in hydrological conditions, which give rise to various ecological responses, including both benefits and side effects (Mallik and Richardson, 2009). Issues such as water level alteration, flow changes, sediment trapping, disruption of nutrient balance, and algal blooms had arisen in most reservoirs due to the hydrological management (Cortes et al., 2002; Taleb et al., 2004; Bennett et al., 2007; Mims et al., 2013).

Since the hydrological management of reservoirs exerted a non-negligible effect on the chemical energy distribution in the watershed, the dynamic processes of natural organic matter, including dissolved organic matter (DOM), would be altered significantly (Hur et al., 2007; Maavara et al., 2020). DOM is an organic assemblage of numerous molecules with different sources and biochemical characteristics (Stedmon et al., 2003). As one of the most dynamic reactive carbon pools on earth, DOM plays a crucial part in global carbon cycling and has attracted substantial at-

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tention (Mopper et al., 2014). The significant influence of hydrology (e.g., discharge changes, rainfall alterations) on the quantity and quality of DOM has been assessed in natural aquatic ecosystems (Inamdar et al., 2011; Bao et al., 2019), however, the effect of reservoir hydrological management-induced variations in DOM cycling was less constrained (He et al., 2020a; Ma and Li, 2020; Maavara et al., 2020; Wang et al., 2021).

As the largest artificial reservoir in the world, the Three Gorges Reservoir (TGR) plays a crucial part in the economic development of China since its full operation in 2008. With a watershed area of 1080 km^2 along the Yangtze River from Chongqing to Yichang, TGR exerts a storage capacity of 39.3 billion m^3. With the operation of TGR, several hydrological alterations have taken place. The water-level fluctuation zone, the area of periodically submerged soils, is formed due to the management of TGR with a water level fluctuation of 30 m ranging from 145 m to 175 m (Bao et al., 2015). Hu et al. (2013) and Dai and Liu. (2013) found that the water velocity was significantly reduced in the mainstream of TGR since its impoundment, so as the flux of suspended particles. Algal blooms were observed in several segments of TGR (Cai et al., 2006). These alterations in physicochemical conditions of TGR have gained wide attention, especially for carbon cycling (Maavara et al., 2020). Our previous study has preliminarily depicted the relationship between hydrological management and CO_2 emission in TGR due to the degradation of labile components of DOM (He et al., 2020b). Previous studies have found that the recalcitrant component of DOM can devote to the in situ organic carbon (OC) burial (e.g., Schmidt et al., 2009), but whether and how it is affected by the hydrological management of reservoirs is poorly known. Therefore, the comprehensive understanding of alteration dynamics and fate of DOM would offer better insight into the carbon cycling in reservoirs.

Analytical techniques of DOM develop significantly in the past several decades from initial bulk chemical characteristic analysis to optical properties depiction of colored DOM (CDOM) and fluorescent DOM (FDOM), including ultraviolet-visible spectroscopy (UV–Vis) and excitation and emission matrixes (EEMs; Hansen et al., 2016). The combination of EEMs and parallel factor analysis (PARAFAC) is also developed (Stedmon et al., 2003). Humic-like and protein-like PARAFAC components from various sources could be identified and semi-quantified. The characteristics of DOM are revealed partly by these rapid and less expensive approaches (Huguet et al., 2009). More recently, Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR MS) is used to precisely depict the characteristics of DOM on the molecular level (Kujawinski et al., 2002; D'Andrilli et al., 2015; Pang et al., 2020). Thousands of molecular formulae (CHO, CHON, and CHOS classes) in DOM are identified and several molecular groups have been preliminarily linked with different sources such as anthropogenic sources and terrestrial inputs, which are not accessible through conventional analytical techniques. With the combination of these approaches, compositional details, lability and degradability (biodegradability and photodegradability) of DOM could be assessed, so as its environmental behaviors (McKnight et al., 2001; Sleighter et al., 2014).

In this study, we investigated one of the largest tributaries of TGR, Daning River. We collected surface and bottom water samples, surface sediments and sediment core samples to monitor DOM variations through space (spanning a ca. 40 km transect) and time (the sediment core covering the age of 10~15 years). Through the application of a series of approaches including bulk chemical techniques, UV–Vis, EEMs and FT-ICR MS, the objectives were to 1) comprehensively depict the characteristics, sources, and spatial variations of DOM along the tributary; 2) preliminarily explore the linkage between DOM properties (i.e., recalcitrant signature) and OC burial under the hydrological management of TGR.

2. Materials and methods

2.1. Site description

The TGR is located on the Yangtze River in south-central China (110°25′–111°06′E, 31°04′–31°34′N). With the function of controlling floods, generating electricity, and improving navigability, this reservoir project started in 1994 and run since 2008. The area of TGR has a subtropical monsoon humid climate, with an average annual temperature of 18.9 °C and precipitation between 817.1 and 1360.5 mm.

Daning River, one of the largest tributaries in the TGR region with an average discharge of 73 m^3/s, is located in the middle section of TGR and at a distance of ~123 km from the dam. The length and watershed of Daning River are ca. 162 km and 4170 km^2, respectively, covering both Wushan and Wuxi Counties (Fig. 1). The watershed of Daning River has a high mountain canyon topography and mountains cover $>$ 95% of the total area with an average elevation of 1197 m.

2.2. Sample collection

To capture the optimal effect of hydrological management of reservoir, we sampled in November of the year 2017 (storage period), during which the flow was reduced to store water in the reservoir and the hydrological regime was shifted from river to semi lake-like condition by management (Zhou et al., 2016; Zhao, 2017). Surface (ca. 1 m deep) and bottom water (ca. 50 m deep) samples were collected based on topography from 8 sites in Daning River (DN07~DN00). DN00 is the most downstream (estuary) site, and DN07 is the most upstream site (about 40 km to the estuary) (Fig. 1). Besides that, three surface water samples (01, 02, and 03) were collected in mainstream. All water samples were collected using hydrochloric acid (trace metal purity) precleaned Nalgene bottles, transported back to the lab on ice. All water samples were filtered through precombusted 0.7 μm glass fiber filters (Whatman GFF) and 0.22 μm membrane filters (Millipore) to remove particles, algal aggregates, and most bacteria within 24 hr of sampling to obtain DOM in water (Seidel et al., 2014).

To assess the DOM characteristics in buried sediments, surface sediments and a sediment core (12 cm length) were obtained at DN04. Considering the average annual sedimentation rate of ca. 1.12 cm (ranged from 0.70 to 1.54 cm, Tong, 2020), the sediment core (12 cm, obtained in 2017) could likely cover the sedimentary record from 2012 when TGR began to fully operate. Sediment core was obtained with a gravity corer and divided every 2 cm into 6 portions (0–2 cm, 2–4 cm, 4–6 cm, 6–8 cm, 8–10 cm, 10–12 cm). Sediment samples were warpped with a pre-combusted alumina foil, kept in vacuum-sealed plastic bags, and placed in an icebox during the transport back to the lab. DOM in sediments was obtained followed the procedure of Hur et al. (2014) within 24 hr of sampling. In particular, DOM in sediments was extracted by centrifugation of the sediments at 5000 rpm for an hour (Hur et al., 2014). The supernatant was carefully collected under an atmosphere of nitrogen and stored in tightly sealed tubes, then filtered through precombusted 0.7 μm glass fiber filters (Whatman GFF) and 0.22 μm membrane filters (Millipore) to get DOM in sediments. The remaining sediments were all air-dried, ground, and passed through a 0.2 mm sieve and stored at 4 °C for further analyses.

2.3. Bulk geochemical analyses

Concentrations of Na^+, K^+, Mg^{2+}, Ca^{2+}, and Cl^- in water samples were measured by Dionex ICS-3000. Nutrient concentrations

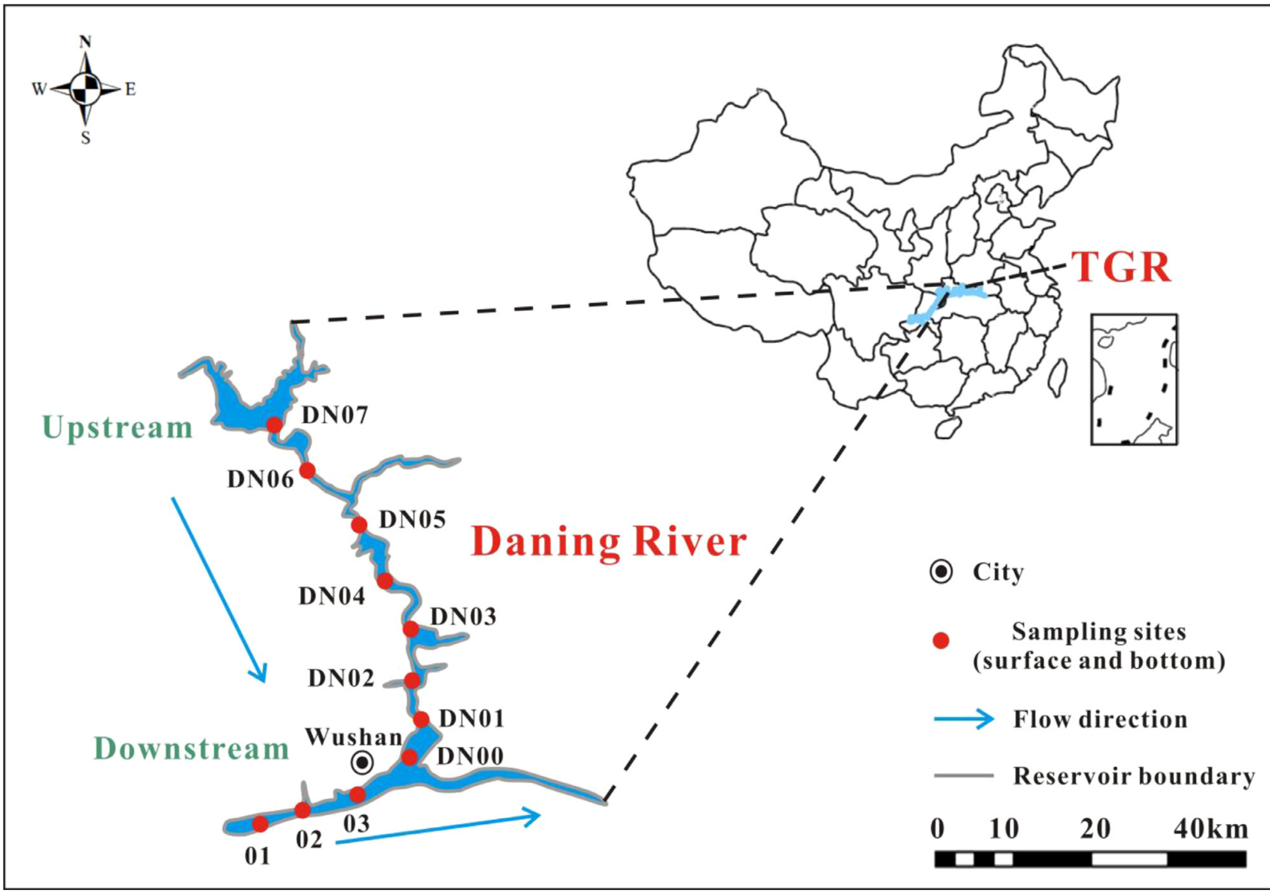

Fig. 1. Study area and sampling sites in Daning River of TGR. Sampling sites were selected along the upstream to downstream transect (DN07 to DN00) and mainstream (01, 02, 03).

in water samples were measured by UV–Vis spectroscopy (Multiscan Spectrum, Thermo Scientific). Dissolved organic carbon (DOC) concentrations were determined by high-temperature catalytic oxidation (TOC-L analyzer, Shimadzu) with a coefficient of variation of 2%. A calibration series, deep-sea and low carbon water standards provided by the University of Miami, Miami, FL, were used. Water samples in these measurements were all filtered by 0.22 μm membrane filters (Millipore).

The DOM in the 0.22 μm-filtered samples (0.5 L) was concentrated by solid-phase extraction (SPE) using 200 mg, 3 ml Agilent Bond Elut PPL cartridges (Dittmar et al., 2008). After SPE, the cartridges were dried completely via ultra-high purity nitrogen gas, eluted with 4 ml methanol to pre-combusted glass ampoules and stored in dark at −20 °C for further analysis.

The total organic carbon (TOC), total nitrogen (TN) contents and δ^{13}C values of whole sediments were analyzed by an elemental analyzer and an isotope ratio mass spectrometer (Finnigan MAT 253, Thermal Scientific, USA), following the previously published protocol (Liu et al., 2020).

2.4. Spectroscopic analysis and PARAFAC modeling

UV-Visible absorbance and 3D-EEM were measured for both water column samples and water-extractable organic matter of the sediments using an Aqualog (Horiba, Japan) absorption-fluorescence spectrometer. The EEMs were calibrated for Raman scattering and Inner-filter effects. Various parameters including specific ultraviolet absorbance at 254 nm [$SUVA_{254}$ (L mg C^{-1} m^{-1})], fluorescence index (FI), humification index (HIX), biological index (BIX), freshness index (β:α), spectral slope ($S_{275-295}$), and slope ratio (S_R) were calculated (Hansen et al., 2016 and reference therein). Besides, parallel factor analysis (PARAFAC) was applied for further qualitative and quantitative analyses of EEMs (Stedmon and Bro, 2008). Detailed procedures were presented in the supplementary information.

2.5. Molecular characterization using FT-ICR MS

Methanol eluent of SPE-DOM was analyzed with a 9.4 T Apex-ultra X FT-ICR MS (the Heavy Oil Key Laboratory, China University of Petroleum) under negative mode with an electrospray ionization source (Bruker Apollo II). The whole process followed the instrument setting proposed by He et al. (2020a). After calibration with a list of known molecular formula mass peaks (Bruker Daltonics Data Analysis 3.4 software package), mass spectra range from 200 to 1000 Da was assessed with 128 scans accumulating per run. With the utilization of C, H, O, N and S formulas including CHO, CHON, CHOS and CHONS in which signal-to-noise ratio (S/N) >4 were screened out with a detection error of within 1 ppm in absolute mass (He et al., 2020a). Detailed procedures were presented in the supplementary information.

2.6. Ultra-high resolution mass spectrometry data related parameters integration

The relative peak intensities (mass peak magnitude or intensity divided by the summed magnitude of all mass peaks in a respective spectrum) were proposed as a manifestation of components with different intensities in percentage to assess DOM composition semi-quantitatively. The magnitude-weighted parameters including elements (C, H, O, N, S), formulae (CHO, CHON, CHOS, CHONS) and others (H/C, O/C, double bond equivalent (DBE), modified aromaticity index (AI_{mod})) were calculated as a basis for composition analysis of DOM (Koch and Dittmar, 2006, 2016).

Assigned molecular formulae were categorized as: polycyclic aromatics (PCAs, $AI_{mod} \geq 0.67$), polyphenols ($0.66 \geq AI_{mod} \geq 0.50$), highly unsaturated compounds, which include soil-derived products of lignin degradation ($AI_{mod} < 0.50$, $H/C < 1.5$), unsaturated aliphatic compounds ($2.0 \geq H/C > 1.5$, N = 0), and peptides ($2.0 > H/C \geq 1.5$, N > 0). Carboxylic-rich alicyclic compounds (CRAMs) were also identified (formulae with DBE:C = 0.3–0.68, DBE:H = 0.2–0.95, and DBE:O = 0.77–1.75) (Hertkorn et al., 2006; Wang et al., 2019).

2.7. Statistical analyses

To assess the variation of DOM, the Student *t*-test was conducted on R3.2.1 (R Core Team 2015). To assess the characteristics of DOM comprehensively, Spearman's correlation (considered significant at $p<0.01$ and $r>0.5$) between molecular information and spectral analysis was conducted (Lavonen et al., 2015). To demonstrate the variation of DOM along the transect, a principal component analysis (PCA) was also conducted to select from the optical and molecular parameters (HIX, $SUVA_{254}$, O/C, and H/C, etc.).

3. Results

3.1. Hydrological regime and distribution of conservative ions

The water intrusion from the mainstream to Daning River tributary has been supported by numerous studies in Daning River during November (storage period; same to our sampling month) (Fig. 2a, b), and drainage period (Xiong, 2013; Huang et al., 2017; Zhao, 2017; Han et al., 2020). Significant increasing trends ($p<0.01$) of concentrations in many cations and anions, including Na^+, K^+, Mg^{2+}, and Cl^-, along the DN07 to DN00 transect were also observed (Table S1), which supported the water intrusion process observed previously (Fig. 2b, c) (Yang et al., 2015).

3.2. Bulk geochemical characteristics

The DOC concentrations in Daning River ranged from 146 to 269 µM (Table S1). This range of DOC was smaller than that in the upstream of the mainstream of TGR, which ranged from 87.5 to 850 μM (Chen et al., 2012; Jiang et al., 2018). In general, there was an increasing trend ($p < 0.01$) in DOC concentration in both surface and bottom water from upstream to downstream (DN07 to DN00). Although there were no obvious spatial variation trends for concentrations of DIN and PO_4^{3-} along the upstream to downstream transect, a significant increasing trend ($p < 0.01$) was observed for SiO_3^{2-} in both surface and bottom water (Table S1).

3.3. Optical properties of CDOM

The values of HIX and $SUVA_{254}$, which represent the humification and aromaticity degree of DOM, ranged from 0.47 to 0.65 and 3.31 to 5.44 L mg-C^{-1} m^{-1}, respectively. There was an increasing trend ($p < 0.01$) in HIX and $SUVA_{254}$ from upstream to downstream for both surface and bottom water samples (Fig. 3, Table S3). $S_{275–295}$, which has an inverse relationship with humification and aromaticity degree of DOM (e.g., Fichot and Benner, 2012), ranged from 0.0148 to 0.0168 and showed an increasing trend ($p < 0.01$) from upstream to downstream in both surface and bottom water samples. Whereas, FI, BIX, β:α, and S_R which ranged from 1.72 to 2.04, 0.79 to 0.84, 0.78 to 0.82, and 1.47 to 2.08, respectively, showed no significant variation trends along the upstream to downstream transect (Table S3).

Four fluorescent components including two protein-like (C2 and C4) and two humic-like (C1 and C3) components were identified through PARAFAC (OpenFluor; congruence coefficients: 0.9) and validated by split-half analysis (Fig. S1, S2; Table S3). C1 humic-like component is associated with relatively high aromatic compounds or fulvic acid, while C3 humic-like component can exist in farmland environments. C2 and C4 were tryptophan-like component and tyrosine-like component respectively (Table S3). These two components have been reported in various ecosystems including inland waters and coastal wetlands (Stedmon et al., 2005; Yamashita et al., 2011). The averaged relative proportions of C1 and C3 components ranged from 35% to 54% with an increasing trend ($p < 0.01$) from upstream to downstream in both surface and bottom water, while C2 and C4 ranged from 46% to 65% with no trends along the transect.

3.4. Molecular composition of SPE-DOM detected by FT-ICR MS

FT-ICR MS showed that a total of 10,217 (9212 and 9431 in surface and bottom waters, respectively) unique formulas were detected in DOM from Daning River (Fig. S3). A total of 4341 (4051 and 4068 in surface and bottom waters, respectively) unique CHO compounds were detected. The averaged relative abundance of CHO compounds ranged from 68.8% to 76.1%, with no variation trend along the upstream to downstream transect. A total of 3671 (3302 and 3360 in surface and bottom waters, respectively) unique CHON compounds were detected. The average relative abundance of CHON compounds ranged from 14.9% to 22.3%. A total of 1664 (1396 and 1517 in surface and bottom waters, respectively) unique CHOS compounds were detected. The averaged relative abundance of CHOS compounds ranged from 6.8% to 14.5%. A total of 540 (457 and 486 in surface and bottom waters, respectively) unique CHONS compounds were detected. The averaged relative abundance of CHONS compounds ranged from 0.61% to 1.54%. The averaged relative abundance of heteroatom compounds including CHON, CHOS, and CHONS compounds showed no significant variation from upstream to downstream.

There was an increasing trend ($p < 0.01$) in AI_{mod} in both surface and bottom water along the upstream to downstream transect, whereas no significant trend was observed for other parameters (e.g., H/C, O/C, and DBE) (Fig. 4a, d; Table S4). The relative abundance of polyphenols and highly unsaturated compounds ranged from 6.3% to 7.5% and 81.7% to 87.6%, respectively, with significant increasing trends ($p < 0.01$) in both surface and bottom water from the upstream to downstream (Fig. 4b, c, e, f). The relative abundances of unsaturated aliphatic compounds and peptides ranged from 4.7% to 8.9% and 0.2% to 0.7%, respectively, with no significant trends observed along the upstream to downstream transect (Table S4).

3.5. Associations between molecular information and optical properties

3.4.1. Associations between molecular groups and PARAFAC components

All identified molecular groups correlated significantly with one or more PARAFAC components ($r > 0.5$, $p < 0.01$). The number (weighted average proportion of intensity) of formulae in the individual molecular group significantly correlated with PARAFAC components (C1, C2, C3, and C4) ranged from 8 (1%) to 87 (38%) in PCAs, 21 (1%) to 503 (52%) in polyphenols, 438 (1%) to 2417 (53%) in highly unsaturated compounds, 21 (7%) to 378 (51%) in unsaturated aliphatic compounds, and 6 (1%) to 76 (35%) in peptides, respectively (Table S6, Fig. S4).

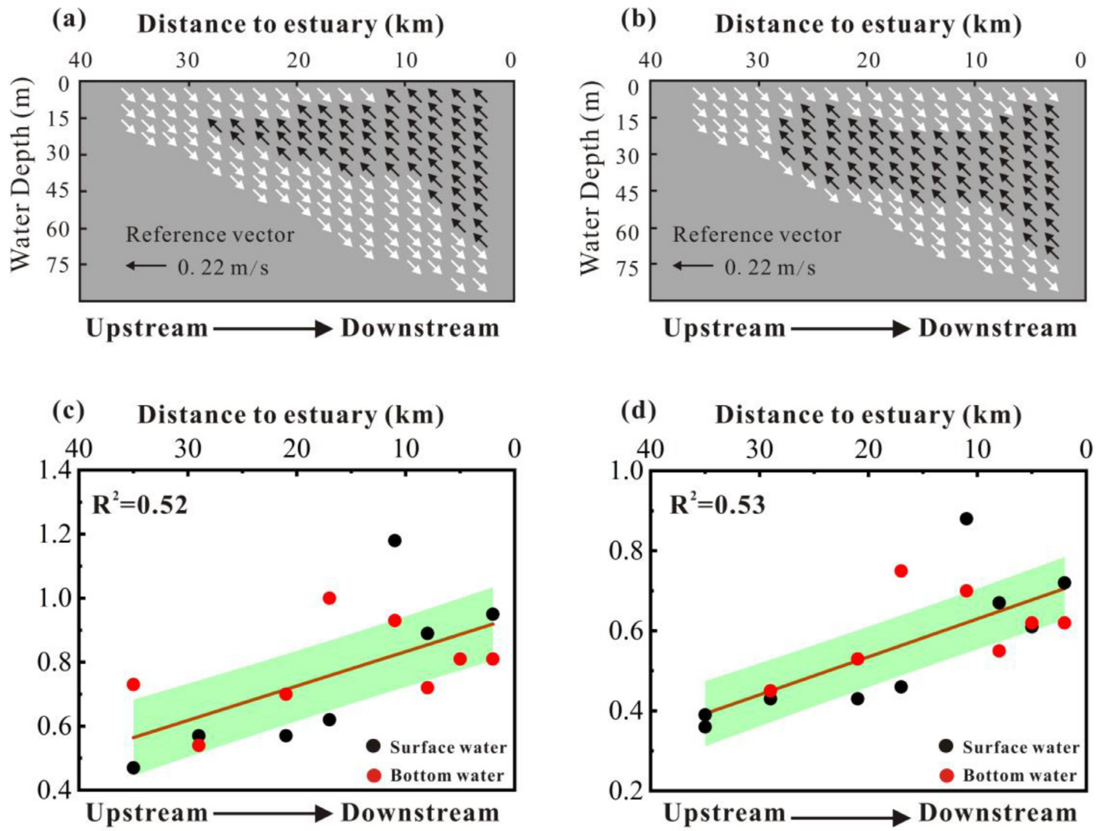

Fig. 2. Velocity distribution and conservative ion concentration plots: velocity distribution profile diagrams in Daning River during drainage (a) and storage (b) periods; concentrations of Na^+ (c) and Cl^- (d) in surface and bottom water along upstream to downstream transect ($p < 0.01$). Note: Black vector represents the water flowing from downstream (DN00) to upstream (DN07), and white vector represents just opposite. The velocity distribution profiles were modified from Huang et al. (2017) and Zhao (2017). Shaded area of green color in (c) and (d) indicates 95% confidence interval.

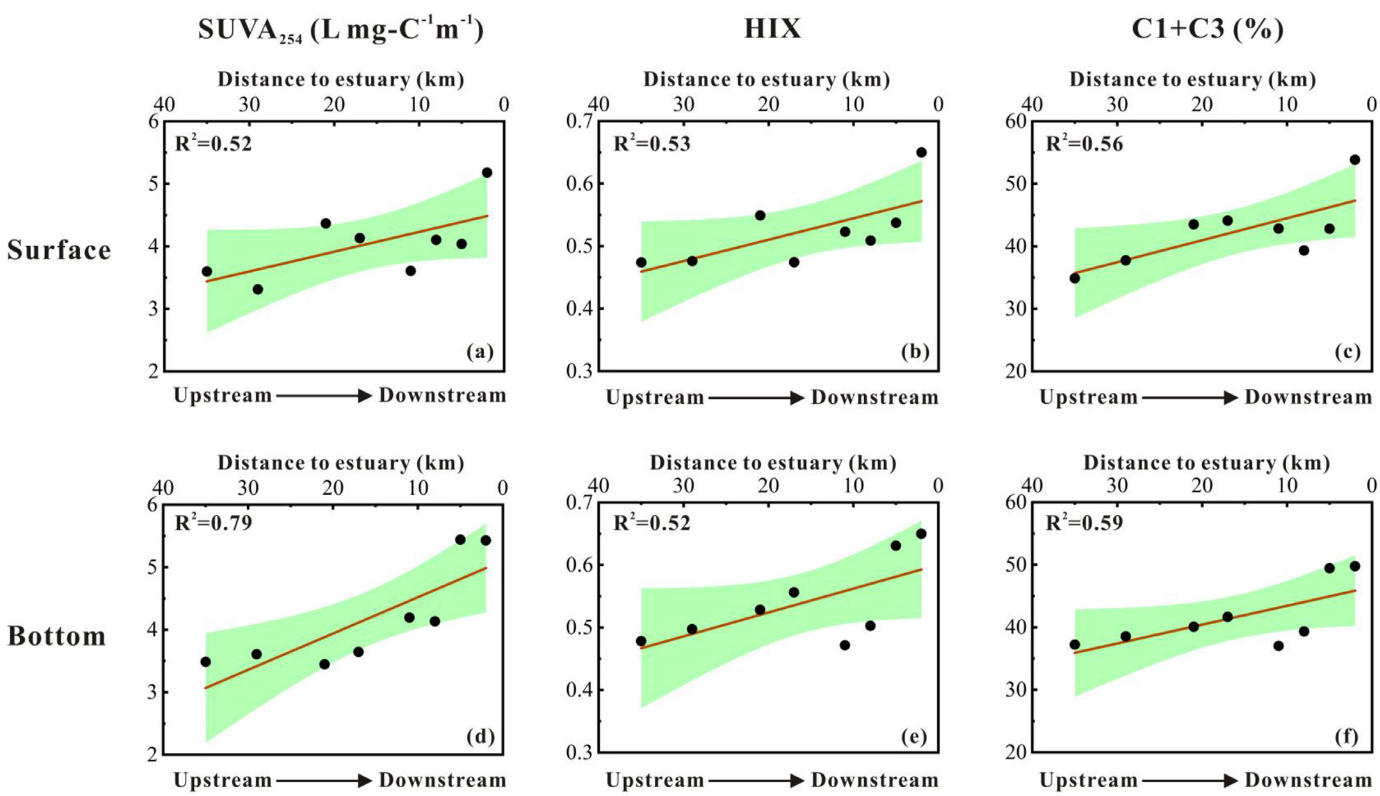

Fig. 3. Optical properties of DOM in surface and bottom water along the upstream to downstream transect ($p < 0.01$). (a)-(c): $SUVA_{254}$, HIX, and C1+C3 of DOM in surface water, (d)-(f): $SUVA_{254}$, HIX, and C1+C3 of DOM in bottom water. Note: The bottom X-axis is from upstream to downstream; the top X-axis is by distance (km) to estuary; shaded area of green color indicates 95% confidence interval.

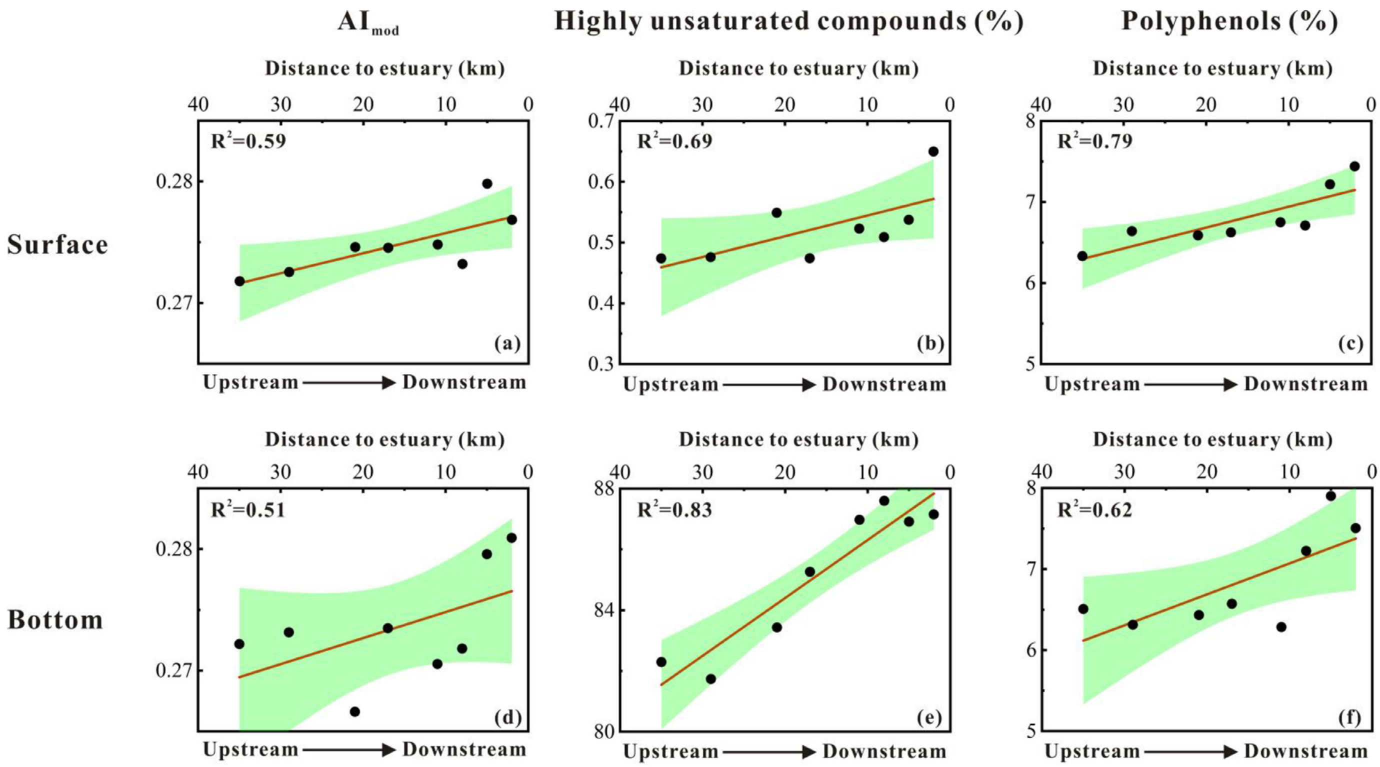

Fig. 4. Molecular variation of DOM in surface and bottom water along the upstream to downstream transect ($p < 0.01$). (a)-(c): AI_{mod}, highly unsaturated compounds, and polyphenols of DOM in surface water, (d)-(f): AI_{mod}, highly unsaturated compounds, and polyphenols of DOM in bottom water. Note: The bottom X-axis is from upstream to downstream; the top X-axis is by distance (km) to estuary; shaded area of green color indicates 95% confidence interval.

3.4.2. Associations between molecular groups and optical indices

The number (weighted average proportion of intensity) of formulae in the individual molecular group significantly correlated with optical indices ($SUVA_{254}$, $S_{275–295}$, S_R, BIX, and HIX) ranged from 33 (22%) to 85 (37%) in PCAs, 253 (25%) to 471 (48%) in polyphenols, 1218 (33%) to 2713 (50%) in highly unsaturated compounds, 102 (17%) to 387 (52%) in unsaturated aliphatic compounds, and 21 (16%) to 63 (31%) in peptides, respectively (Table S6, Fig. S4).

3.4.3. Molecular covariance among PARAFAC components and optical indices

The averaged molecular weight, H/C, O/C, and AI_{mod} of the formulae associated with PARAFAC components varied from 368 to 481, 1.14 to 1.29, 0.40 to 0.52, and 0.23 to 0.29 respectively (Table S6). The averaged molecular weight, H/C, O/C, and AI_{mod} of the formulae associated with optical indices ($SUVA_{254}$, $S_{275–295}$, S_R, BIX, and HIX) varied from 353 to 475, 1.14 to 1.30, 0.39 to 0.52, and 0.24 to 0.28, respectively (Table S6). The H/C, O/C, AI_{mod}, and mass distribution of molecular formulae associated with PARAFAC components and the typical optical indices were shown (Fig. S4).

3.6. Geochemical properties of sedimentary organic matter

$\delta^{13}C$ values of sedimentary organic matter (whole sediments) varied from −26.5‰ to −25.1‰, showing a significant ($p < 0.01$) increasing trend (enrichment of ^{13}C) with depth from the surface to bottom (8–10 cm) layers (Fig. 5b). The total organic carbon percentages (OC%) of the sediments ranged from 0.57% to 0.80%, showing a significant ($p < 0.01$) decreasing trend with depth (Fig. 5c).

The HIX of DOM in the sediment core ranged from 0.82 to 0.87, showing a significant ($p < 0.01$) decreasing trend with depth (Fig. 5a). Significantly higher (p <0.01) averaged HIX value was observed in DOM of sediments than that of both surface and bottom water samples.

4. Discussions

4.1. Multiple sources of DOM in Daning River

Several DOM sources including anthropogenic, terrestrial and autochthonous sources were revealed in Daning River through the analyses of DOM by UV–Vis, EEMs, and FT-ICR MS. Our study identified two humic-like components (C1 and C3) in DOM, which are considered to be derived from terrestrial sources. These two humic-like components were also identified in the lakes and estuaries (Stedmon et al., 2005; Murphy et al., 2014). Besides, the range of HIX and $SUVA_{254}$ values of CDOM (0.47–0.65, 3.31–5.44 L mg-$C^{-1}m^{-1}$, respectively) suggested terrestrial inputs as well (Hansen et al., 2016). Considering the parameter ranges of aquatic ecosystems including rivers and lakes ($SUVA_{254}$: 0.6 to 5.3 L mg-C^{-1} m^{-1}; HIX: 0.39 to 0.87), the values of these parameters indicated that CDOM in Daning River was in the high end of aromaticity and humification degree (Weishaar et al., 2003; Kothawala et al., 2012b). In terms of molecular composition, the detection of polyphenols and highly unsaturated compounds in SPE-DOM, which could be indicators of vascular-plant-sourced organic matter and products of lignin degradation, respectively, demonstrated the input of terrestrial organic matter (Seidel et al., 2015). Besides, the significant positive correlations (Spearman's rank correlation, $r > 0.5$, $p < 0.01$) between terrestrial indicators of FDOM (HIX, C1, and C3) and SPE-DOM (AI_{mod}, polyphenols, and highly unsaturated compounds) further confirmed the terrestrial source of DOM (Table S6).

The protein-like C4 has a similar EEMs peak range to the C5 in the inland water system and C6 in Liverpool Bay, UK,

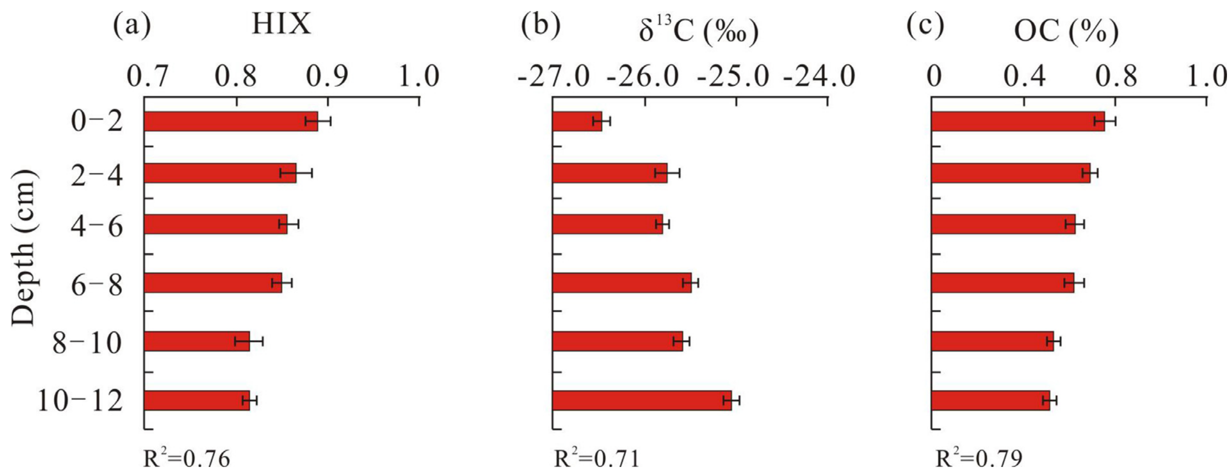

Fig. 5. Geochemical properties of sedimentary organic matter in Daning River ($p < 0.01$). (a) HIX of DOM in sediments, (b) (c) properties of sedimentary organic matter.

which were associated with the aquatic organisms (Osburn et al., 2016; Yamashita et al., 2011). The identification of peptides and unsaturated aliphatic compounds in SPE-DOM by FT-ICR MS also indicated the contribution of DOM from aquatic organisms (Seidel et al., 2015). The significant correlations (p <0.01) between peptides or unsaturated compounds and FI or BIX further confirmed the aquatic input (Table S6). No significant positive correlation was observed between C4 and peptides or unsaturated aliphatic compounds, which might result from the limitation of the analytical window of FT-ICR MS for high molecular weight (e.g., >700 Da) compounds associated with C4.

The protein-like C2 is similar to the C5 component reported by Murphy et al. (2014), which was identified in wastewaters and indicated the anthropogenic input. In terms of molecular composition, O_3S and O_5S classes of compounds identified in FT-ICR MS, which might relate to surface-active ingredients in cleaning and personal care products, also signify the anthropogenic input (Melendez-Perez et al., 2016). In addition, a significant correlation (p <0.01) between C2 and O_3S and O_5S compounds further supported the input of anthropogenic sourced DOM (Table S6).

4.2. The effect of hydrological management on DOM chemistry in Daning River

A significant ($p < 0.01$) decreasing proportion of terrestrial-derived DOM and an increasing ($p < 0.01$) proportion of autochthonous and anthropogenic-derived DOM along the upstream to downstream (DN07 to DN00) transect were observed (Figs. 1, 3, 4). The values of HIX, $SUVA_{254}$, and AI_{mod} increased significantly ($p < 0.01$) along the upstream to downstream transect, suggesting that DOM in downstream had higher aromaticity and humification degree but a smaller proportion of microbial or freshly produced DOM that of the upstream (Figs. 3, 4). No significant differences were observed for the optical and molecular indices between the surface and bottom waters, which demonstrated that the DOM chemistry showed limited vertical variations during the sampling period.

The variations in DOM chemistry along the upstream to downstream transect were further revealed by the PCA (Fig. 6). The loadings of the first two principal components captured 73% of the total variations. The first principal component (PC1) showed strong positive loadings for parameters associated with humification (e.g., $SUVA_{254}$ and AI_{mod}) and PARAFAC components or molecular groups associated with humic-like compounds (e.g., C1, C3, polyphenols, and highly unsaturated compounds) and negative loadings for parameters associated with freshly produced DOM (e.g., H/C, C4, peptides, and unsaturated aliphatic compounds). Samples at downstream sites (e.g., DN00, DN01, and DN02) have positive scores on PC1 and are plotted near AI_{mod}, $SUVA_{254}$, C1, C3, polyphenols, and highly unsaturated compounds, suggesting a higher proportion of terrestrially sourced DOM than samples in upstream (e.g., DN05, DN06, and DN07), which have negative scores on PC1.

Since there were similar vegetation distribution, land use, and geomorphology along the short (ca. 35 km distance) upstream to downstream transect, the spatial variation in DOM chemistry (e.g., the relative abundance of terrestrial DOM) might result from other factors, especially the water exchange between mainstream and tributary due to the reservoir hydrological management (He et al., 2020b). Several studies have focused on the interaction processes of river waters between the tributaries and mainstream of TGR mainstream due to hydrological management (Ji et al., 2010). These studies found that the mainstream of the TGR intruded into all tributaries (e.g., Xiangxi River, a tributary close to the Three Gorges Dam) as a density current with different plunging depths and would result in 70% of tributary water come from mainstream (Ji et al., 2010). Cl^- was used as a tracer to estimate the water source in the Daning River and the results showed that on average 73% of the water in Daning River is from the TGR mainstream (Ran et al., 2010). Xiong (2013) proposed a water exchange model in Daning River during November (same as our sampling month), which is characterized by water intrusion from the mainstream to Daning River tributary. This water exchange model in Daning River is found during both drainage and storage periods (Huang et al., 2017; Zhao, 2017; Han et al., 2020). Although the water velocity was not directly measured in this study, the concentrations of conservative cations and anions (e.g., Na^+, Cl^-) measured in this study also supported the water intrusion process (Table S1). For instance, it is known the mainstream water is characterized by higher concentrations of cations and anions (Ran et al., 2010), and there was a gradual increase of both cations and anions (e.g., Na^+, Cl^-) along the upstream to downstream (DN07 to DN00) transect (Table S1). In this regard, the DOM variation along the upstream to downstream can be mainly explained by this water exchange model, and a conceptual model of DOM dynamics is developed (Fig. 7). The hydrological management of the TGR resulted in the water intrusion from the mainstream to Daning River tributary (Figs. 2, 7). Since the DOM in the mainstream had a significantly higher (p <0.01) proportion of terrestrial signal (Table S7), the increase of terrestrial signal from the upstream to downstream could result mainly from the mainstream water intrusion. Therefore, the hydrological man-

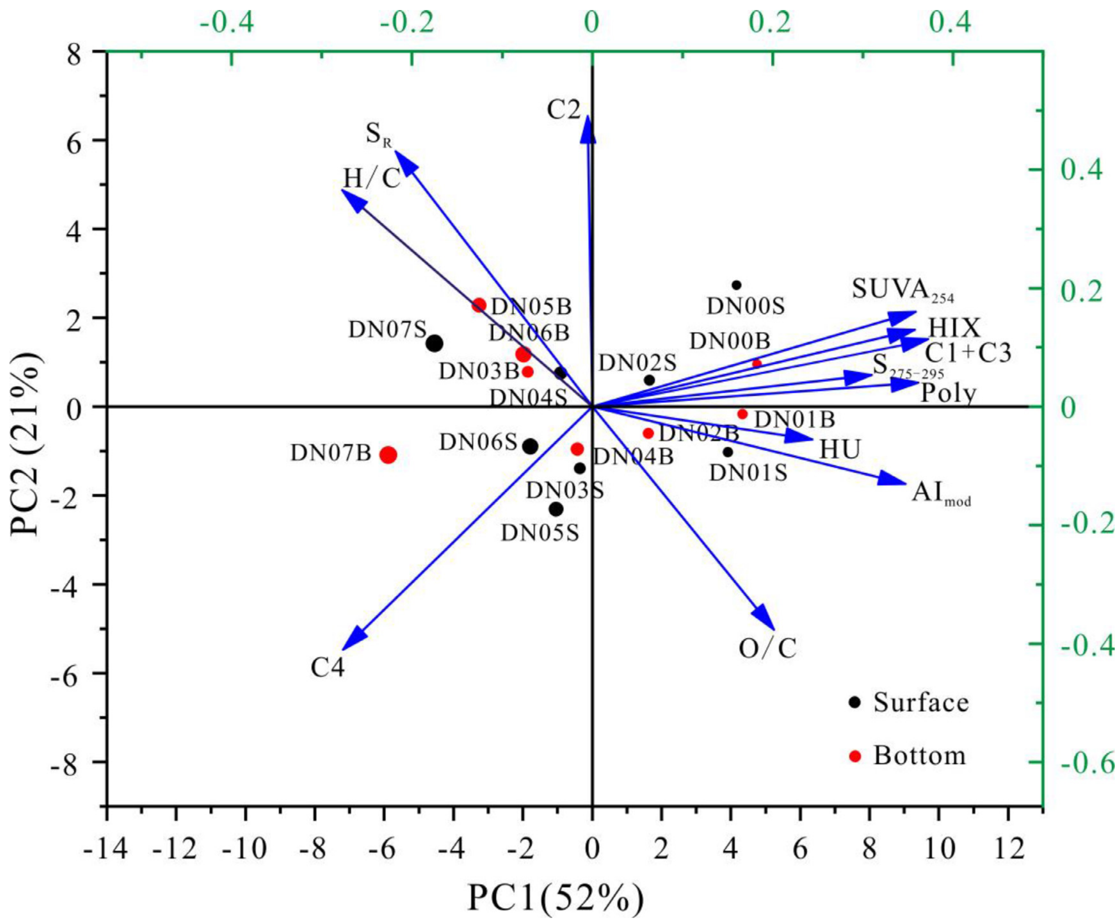

Fig. 6. Results of PCA analysis based on different parameters (optical parameters: C1+C3, C2, C4, HIX, $SUVA_{254}$, $S_{275-295}$, S_R; molecular parameters: H/C, O/C, AI_{mod}, HU, Poly). HU: Highly unsaturated compounds, Poly: Polyphenols. The size of sample symbol (surface and bottom samples) increases with the distance from estuary; coordinate axis in green color exhibits the loadings of parameter.

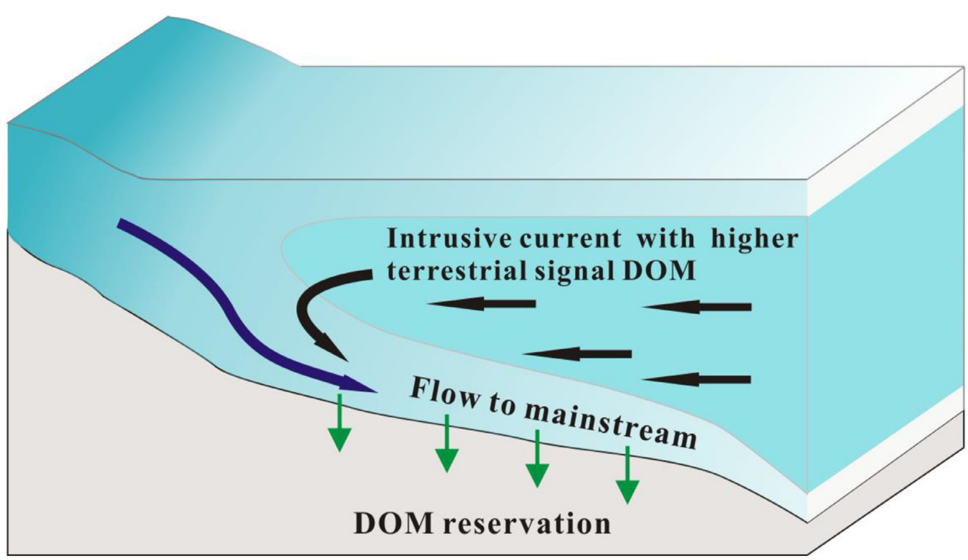

Fig. 7. Conceptual model relating reservoir hydrologic management to DOM composition and reservation.

agement of the TGR introduced additional terrestrial DOM to Daning River tributary. Since the water intrusion to Daning River tributary has been observed from January to December (Xiong, 2013; Zhao et al., 2015), the addition of DOM from the mainstream to Daning River tributary likely exists in multiple months (Zhao et al., 2015; Zhao, 2017; Han et al., 2020).

4.3. Implications for organic carbon burial and further considerations

The linkage between human activities and OC burial has been increasingly emphasized (Sundquist et al., 1993; Stackpoole et al., 2014). In particular, the relationship between reservoir operation and OC burial has been paid attention (Tranvik et al., 2009;

Maavara et al., 2020). Although the OC burial is influenced by sediment transport, it is known that the DOM transported through the water column to the sediments could be an important way of OC accumulation in sediments as well (Tranvik et al., 2009). For instance, recalcitrant DOM compounds with carboxylic and hydroxylic groups (e.g., CRAMs) allow for ligand exchange with metals on mineral surfaces (Kothawala et al., 2012a), resulting in preferential adsorption to particles and further deposited in sediments (Kalbitz et al., 2005). Besides, the flocculation of recalcitrant DOM with metals such as Fe has also been observed in various aquatic ecosystems (Yu et al., 2015; Jilbert et al., 2017), and this association has devoted to stabilize OC in sediments and promote carbon burial (Lalonde et al., 2012). In particular, there was a positive relationship between the contents of recalcitrant DOC and total OC in sediments (Schmidt et al., 2009).

With the significant addition of terrestrial and recalcitrant DOM from the mainstream, the effect of reservoir hydrological management on the OC burial in Daning River tributary is further explored. Firstly, since the river reservoir is constructed, it will result in longer water residence time (compared with the natural river) and thus increase the in situ primary production (e.g., Cai et al., 2006). This increased in situ primary production will lead to increased deposition of autochthonous organic matter through time. Li et al. (2015) estimated that the $\delta^{13}C$ of phytoplankton-derived particulate organic carbon in Changjiang River is ca. −32.0‰ to −30.0‰, which is even more depleted in ^{13}C than that of the higher plants. Nevertheless, the DOM derived from phytoplankton usually has lower HIX values (e.g., Lee et al., 2019; Wang et al., 2019). Considering the overall high HIX values observed in this study, the DOM is likely mainly derived from terrestrial sources. Therefore, we suggest that the autochthonous DOM from phytoplankton is usually characterized by higher $\delta^{13}C$ and lower HIX values than those of terrestrial plant-derived organic matter, which will lead to increased $\delta^{13}C$ for the TOC of sediments and decrease of HIX for water-extractable organic matter with time. However, our surface sediment showed lower $\delta^{13}C$ (TOC of the sediments) and higher HIX (water-extractable organic matter of the sediments) than those of deeper sediments. As such, increased deposition of autochthonous organic matter through time cannot explain the vertical distribution of $\delta^{13}C$ and HIX.

The second factor to consider is that OC will undergo consistent degradation during its burial. Nevertheless, the degradation seems not to be a dominant factor affecting the distribution of OC quantity and quality with depth, because during biodegradation of DOM, the HIX value will increase due to the preferential removal of protein-like components and preservation of humic-like components, which has been observed in multiple studies (e.g., Hansen et al., 2016), and our previous incubation experiments at Xiangxi River tributary of the Yangtze River (He et al., 2020b), leading to increase of HIX values with depth. However, this is not the case observed here.

Therefore, integrated with the conceptual model (Fig. 7), we suggest that the increasing HIX value of extractable organic matter from bottom to top layers of the sediment core is mainly caused by the increasing terrestrial signal in Daning River tributary through time, which might be related to the addition of terrestrial and recalcitrant DOM from the mainstream (Fig. 5). Moreover, the quality association of DOC and OC was observed in the sediments of Daning River tributary by the significant positive correlation ($p < 0.01$) between the HIX value of DOM and OC% for the sediment core samples (Fig. S5). Specifically, there were significantly increasing trends ($p < 0.01$) of HIX and OC% values and a decreasing trend ($p < 0.01$) of $\delta^{13}C$ values from bottom to top layers of the sediment core, demonstrating the increase of terrestrial OC for both TOC and DOM of sediments in Daning River tributary with damming time increase (Fig. 5). Considering the increasing terrestrial signal from the bottom to top layers of the sediment core of Daning River tributary, and the established water intrusion regime from the mainstream to tributary, we speculate that the hydrological management of TGR makes an important devotion to the OC burial in tributaries (with similar hydrodynamics to Daning River tributary) through the addition of DOM with more terrestrial and recalcitrant signature. About 26 Tg OC per year has been estimated to bury in global reservoirs, which would increase to 52 Tg OC per year by 2030 due to the construction and operation of reservoirs, a fourfold increase compared with that in 1970 (Maavara et al., 2017). Considering the linkage between hydrological management and DOM chemistry in Daning River tributary, the hydrological management-induced variations in DOM chemistry should contribute to the estimated increase of OC burial in reservoirs around the world.

This study has provided novel insights into the OC burial mechanism in a typical tributary of the world's largest reservoir. This investigation was carried out in November with low river flow which likely facilitates DOM adsorption on particles and sedimentation in the lentic environment, future seasonal investigations and more sedimentary cores are needed to make a more comprehensive assessment of the linkage between DOM chemistry and OC burial. In addition to hydrological management, other factors may also affect the DOM chemistry and OC burial in reservoirs. For instance, both biodegradation and photodegradation may affect the spatial variations in DOM chemistry along the upstream to downstream transect. The mutual transformation among DOM, particulate organic matter (POM), and sedimentary organic matter would also influence the OC burial in reservoirs (He et al., 2016). In this regard, further qualitative and quantitative assessments of the factors affecting DOM chemistry and OC burial are encouraged to address the environmental and management control of carbon cycling in reservoirs.

5. Conclusions

This study provides comprehensive evidence that the hydrological management of the TGR has a substantial effect on the quantity and quality of DOM in Daning River tributary. Significant changes in optical and molecular properties of DOM were revealed in both surface and bottom water along the upstream to downstream transect of Daning River tributary. A significant increasing trend of aromaticity and humification degree (revealed by $SUVA_{254}$, HIX, and AI_{mod}) was observed along the upstream to downstream transect. Besides, the alteration of various sources of DOM (anthropogenic, terrestrial and autochthonous inputs) was uncovered by the changes in a variety of optical and molecular parameters identified by EEMs and FT-ICR MS. A significant increasing trend of terrestrial signal from the upstream to downstream transect was observed, which was mainly caused by water intrusion from the mainstream to tributary due to the hydrological management of TGR. The hydrological management of TGR likely further contributes to the increase of OC burial in Daning River tributary. Collectively, this study emphasized the role of hydrological management in alteration of DOM chemistry and OC burial in a typical tributary of TGR, and provided insights into the interaction between anthropogenic perturbations and carbon cycling in fluvial ecosystems.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests that could have appeared to influence the work reported in this manuscript.

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Supplementary materials

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