

Differences in the Composition, Source, and Stability of Suspended Particulate Matter and Sediment Organic Matter in Hulun Lake, China

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1 **Differences in the composition, source, and stability of suspended particulate matter and sediment organic**
2 **matter in Hulun Lake, China**

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13
14 **Abstract:** Sediment (SOM) and suspended particulate (SPOM) organic matters are two important organic matters
15 in water. Their occurrence, migration and transformation, and stability have important effects on the
16 environmental behaviors of carbon, nitrogen, phosphorus, and other pollutants in a water environment. The
17 content, composition, fluorescence characteristics, source, and stability of SOM and SPOM in Hulun Lake, a
18 typical lake in cold and arid region of China, were compared by sequential extraction, three-dimensional
19 fluorescence spectroscopy, parallel factor technique, carbon–nitrogen ratio, and stable carbon isotope. SOM and
20 SPOM in north and west were higher than those in east and south. The average content of SPOM (24.70 ± 4.63
21 g/kg) was slightly higher than that of SOM (23.04 ± 10.27 g/kg), but the difference was not significant. Humin
22 was the dominant component in SOM and SPOM, accounting for 73.7% and 61.2%, respectively. Humus was the
23 main fluorescence component of water-extractable organic matter in SOM and SPOM, accounting for 79.9% and
24 70.4%, respectively, of the total fluorescence intensity. SOM and SPOM were derived from terrestrial sources
25 with relative contribution rate of about 70%. SPOM was more influenced by autochthonous sources and had
26 significantly lower humification degree and stability than SOM. Effects of climate changes on migration,
27 transformation, stability, and bioavailability of organic matters and endogenous pollutants closely related to
28 organic matters in lakes of cold and arid regions should be paid attention in the future.

29 **Keywords:** lake in a cold and arid area, Hulun Lake, sediment organic matter (SOM), suspended particulate
30 organic matter (SPOM), source, stability

31 **1. Introduction**

32 Organic matter exists widely in all kinds of water environments and is one of the important chemical
33 components in a water environment. Organic matter participates in the material circulation of the aquatic
34 environmental food web and affects the occurrence, migration, transformation, cycle, and biological availability,
35 and toxicity of water environment pollutants, including nitrogen, phosphorus, heavy metals, and toxic and harmful
36 organic matters (Guo et al. 2020; Shah et al. 2021; Slukovskii et al. 2020; Yao et al. 2020). Organic matters in a
37 lake water environment have complex and diverse compositions and structures and can be divided into dissolved
38 (DOM), suspended particulate (SPOM), and sediment (SOM) organic matters in accordance with different
39 attached media (Chen et al. 2021; Verdugo et al. 2004). The mutual transformation among DOM, SPOM, and
40 SOM can be realized through physical, chemical, and biological functions along with the migration,
41 transformation (He et al. 2016; Toosi et al. 2014), and changes in the environmental characteristics of pollutants
42 combined with organic matters and remarkably influences the environmental quality and ecological health of
43 water (Kurek et al. 2021; Tang et al. 2021). The stabilities of SOM and SPOM make an important contribution to
44 the occurrence of DOM and carbon burial in the lake (Chmiel et al. 2016; Lü et al. 2019; Wang et al. 2021b).
45 However, studies comparing the occurrence and stability of SPOM and SOM in lakes and the changes and
46 mechanism of migration, transformation, occurrence, and bioavailability of biogenic elements and pollutants
47 during the migration and transformation processes among SOM, SPOM, and DOM are few.

48 The lake ecosystem in cold and arid regions is fragile because of its special geographical location and basin
49 climate. This ecosystem is minimally affected by human activities and sensitive to climate change, human
50 interference, and environmental change (Li et al. 2021; Ma et al. 2013; Song et al. 2020; Zhang et al. 2015).
51 Therefore, once the human interference intensity increases or the climate and environment change significantly,
52 the water ecological environment of lakes in the cold and arid region may be seriously and irreversibly damaged.
53 The occurrence, migration, transformation, and sedimentary evolution of organic matters in lakes are bound to
54 change. Thus, the cold and arid regions are ideal study areas for observing the burial, migration, and
55 transformation behaviors and the influencing factors of lake carbon. However, historical data and research of lake
56 organic matters in cold and arid regions are insufficient. Hence, a systematic, comprehensive, and in-depth
57 understanding of the occurrence, transformation and transport, deposition, and evolution of organic matters in

58 lakes in cold and arid regions as well as the response process and mechanism to climate change and water
59 environment change are lacking, which remarkably restricts the protection and management of lake water
60 environment in cold and arid regions, and relevant research should be carried out.

61 The Hulun Lake is a typical lake in the Inner Mongolia–Xinjiang Lake region located in the cold and arid
62 region of China and is the largest lake in north China (Xie et al. 2021). Hulun Lake plays an irreplaceable role in
63 adjusting regional climate, conserving water resources, preventing desertification, protecting biodiversity, and
64 maintaining the ecological balance of Hulunbuir Grassland and the ecological security of northern China (Liu and
65 Yue 2017). The contents, components, fluorescence characteristics, sources, and stabilities of SOM and SPOM in
66 Hulun Lake are studied by the combined use of sequential extraction method, three-dimensional fluorescence
67 excitation emission matrix spectrum–parallel factor method (EEMs-PARAFAC), carbon-nitrogen ratio (C/N), and
68 stable carbon isotope ($\delta^{13}\text{C}$).

69 **2. Materials and Methods**

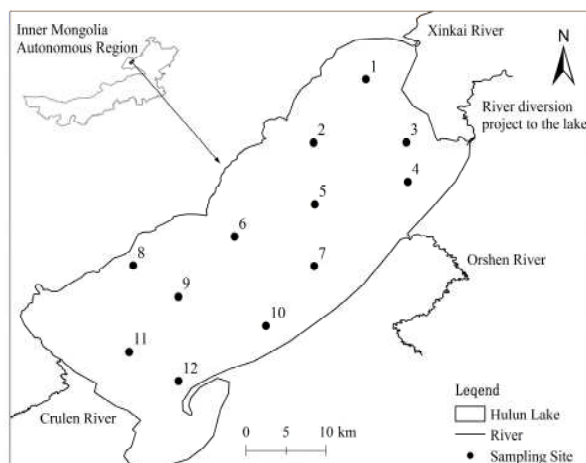
70 *2.1 Study area*

71 Hulun Lake (48.55°–49.33°N, 116.97°–117.81°E) is also known as Dalai Lake. The Hulun Lake basin
72 (including Halaha and Hailar River Basins) is located in China and Mongolia and has an area of $2.92 \times 10^5 \text{ km}^2$,
73 of which the area in China is $1.08 \times 10^5 \text{ km}^2$, accounting for 37% of the total area (Wang et al. 2020). The length,
74 width, circumference, and average water depth of Hulun Lake is 93 km, 32 km, 447 km, and 5.7 m, respectively
75 (Li et al. 2019). The temperature of the Hulun Lake Basin varies considerably at different times throughout the
76 year. The highest temperature is observed in summer for a short time, and low temperature occurs in winter with
77 the ice-forming period lasting for six months (Ao et al. 2020). The dominant wind in the basin is northwest wind,
78 with an average annual wind speed of 4.2 m/s (Zhang et al. 2018). The main land use type in the basin is grassland,
79 with an area of 20 393.57 km^2 , among which the natural grassland area is as high as 20 132.69 km^2 , accounting
80 for 81% of the total area of the basin (Wang et al. 2021a).

81 *2.2 Sediment and suspended particle sample collection*

82 Twelve surface sediment and suspended particle samples were collected from Hulun Lake in July 2019 (**Fig.**
83 **1**). At each sampling point, 40 L of the overlying water sample 0.5 m away from the water surface was collected
84 in a clean plastic bucket. After being transported back to the laboratory, water was filtered by a glass fiber filter
85 membrane (GF/F, $\Phi = 47 \text{ mm}$, Whatman, UK), which was burned at 450 °C for 3 h in advance. After the filter
86 membrane was freeze-dried, suspended particle samples were obtained and stored in a clean sample bag. Surface
87 sediment samples were collected using the Beeker cylindrical sampler (NL, $\Phi = 12 \text{ cm}$, Eijkelkamp, 04.23; SA,
88 the Netherlands) and stored in clean polythene sampling bags in the dark. Sediment samples were freeze-dried

89 after being transported back to the laboratory and sifted through a sieve (100 mesh, 0.149 mm). The prepared
90 sediment samples were stored in clean polythene bags.



91
92

Fig. 1 Sampling sites and location of sediments and suspended particles of Hulun Lake.

93 2.3 Experimental methods

94 2.3.1 C/N

95 Sample pretreatment before total organic carbon (TOC) analysis: An appropriate amount of sediment or
96 suspended particle samples were weighed and placed into a centrifuge tube and added with 20 mL HCl (3 mol/L)
97 to react fully for the removal of inorganic carbon. Samples were washed with ultrapure water to neutral, freeze-
98 dried, ground, and passed through a 100-mesh (0.15 mm) nylon sieve. Pretreated samples were stored in clean
99 polythene bags.

100 Sample pretreatment before total organic nitrogen (TON) analysis (Zhang et al. 2018): An appropriate
101 amount of sediment or suspended particle samples were weighed into a centrifuge tube and added with sufficient
102 amounts of 2 mol/L KCl and 0.5 mol/L HCl to react fully for the removal of inorganic nitrogen. Samples were
103 washed with ultrapure water to neutral, ground, freeze-dried, and passed through a 100-mesh (0.15 mm) nylon
104 sieve. Pretreated samples were stored in clean polythene bags.

105 The contents of TOC and TON were determined using the Elementar (elementar vario MACRO cube,
106 Elementar Analysensysteme GmbH, Germany). C/N was the ratio of TOC to TON.

107 2.3.2 Sequential extraction of organic matter components

108 The chemical components of SOM and SPOM included water-extracted organic matter (WEOM), humic
109 acid (HA), fulvic acid (FA), and humin (HM), and the contents of each component were determined using the
110 sequential extraction method (Zhang et al. 2017).

111 Step 1: Dry sediment or suspended particle sample (1 g) was placed into a 100 mL centrifuge tube and added
112 with 50 mL ultrapure water. The tube was oscillated in a water bath at 25 °C for 1 h and then centrifuged. The
113 supernatant was filtered with 0.45 µm membrane (the same as below) to obtain the WEOM extract.

114 Step 2: The residue obtained in step 1 was cleaned with saturated NaCl and added with 50 mL NaOH (1
115 mol/L)–Na₄P₂O₇ (0.1 mol/L) mixed solution. The pH of the mixture was adjusted to 13 by using 0.1 mol/L HCl.
116 The tube was oscillated at 25 °C for 1 h and centrifuged to separate the supernatant and residue.

117 Step 3: The residue obtained in step 2 was cleaned with saturated NaCl and dried at 55 °C to obtain HM.

118 Step 4: The supernatant (20 mL) obtained in step 2 was collected, and its pH was adjusted to 1.0–1.5 by
119 using H₂SO₄ (0.5 mol/L). The solution was kept in a 60 °C water bath for 1.5 h, allowed to stand for 8 h,
120 centrifuged, and filtered to obtain the FA extract.

121 Step 5: The residue on the filter paper after filtration in step 4 was dissolved in warm NaOH (0.05 mol/L)
122 solution to obtain the HA extract.

123 After measuring the dissolved organic carbon (DOC) concentration in WEOM, FA, and HA extracts by using
124 an automatic total organic carbon tester (TOC-V, SHIMADZU, Japan), the contents of WEOM, FA, and HA in
125 sediments and suspended particles were further calculated in accordance with the mass of the sediments. The HM
126 content (g/kg) was the difference between the TOC content and the total contents of WEOM, FA, and HA.

127 2.3.3 EEM data of WEOM

128 The WEOM extract in section 1.3.2 was subjected to ultraviolet-visible (UV–VIS) spectrophotometry
129 (D5000, hash, USA) at the wavelength range of 200–700 nm (1 nm intervals, medium speed). Ultrapure water
130 was used for the baseline correction of absorbance measurements. A fluorescence analyzer (Hitachi, F7000, Japan)
131 was used to scan the fluorescence spectrum of WEOM to obtain the EEM data. A 150 W xenon lamp was used as
132 the excitation source, and the PMT voltage was set to 400 V. The excitation (λ_{Ex}) and emission (λ_{Em}) wavelengths
133 were set to 210–450 and 250–530 nm, respectively, and the wavelength increment, slit width, and scanning speed
134 were set to 2 nm, 10 nm, and 12000 nm/min, respectively. The calibration methods of EEM data and the
135 determination of fluorescence components by the PARAFAC technology were performed by referring to the
136 methods of Wang et al (2018). The fluorescence intensities (F_{max}) of individual components were used to represent
137 their relative concentrations in the extract, and the total fluorescence intensity (F_{t}) of WEOM was the sum of all
138 components. F_{t} was calculated using Eq. (1):

$$139 \quad F_{\text{t}} = \sum_1^n F_{\text{max}(n)}, \quad (1)$$

140 where n is the number of the fluorescence components of WEOM in SOM or SPOM, and $F_{\max(n)}$ (R.U.) is the
141 relative intensity of the n th component of WEOM in SOM or SPOM.

142 The humification index (HIX) is the ratio of the peak area in the λ_{Em} range of 435–480 nm to the peak area
143 in the λ_{Em} range of 300–345 nm at λ_{Ex} of 255 nm (Huguet et al. 2009).

144 2.3.4 $\delta^{13}C$

145 The sediment and suspended particle samples were pretreated by adding HCl (3 mol/L) to react fully for the
146 removal of inorganic carbon. A proper amount of pretreated sample and 2–3 g CuO wire were placed into a quartz
147 tube that was preheated at 850 °C for 2 h in a muffle furnace. The tube was then welded, sealed in a high-vacuum
148 system, and burned at 850 °C for 5 h in a muffle furnace, and the CO₂ was purified in a vacuum system (Liang et
149 al. 2014). The $\delta^{13}C$ was analyzed using an isotope mass spectrometer (MAT252, Finnigan Mat, Germany) with
150 the Pee Dee Belemnite of Cretaceous in South Carolina, USA as the standard, and the analytical error was 0.2‰.
151 The $\delta^{13}C$ values were calculated using Eq. (2):

$$152 \quad \delta^{13}C = \left(\frac{R_t}{R_s} - 1 \right) \times 1000\text{‰}, \quad (2)$$

153 where R_t is the ratio of natural abundance of ¹³C to ¹²C of the sample, and R_s is the ratio of natural abundance of
154 ¹³C to ¹²C of the standard.

155 2.3.5 Relative contribution of organic matter sources

156 The relative contribution rates of terrestrial and autochthonous sources for SOM and SPOM were calculated
157 using end-member mixing models through the following equations (Wang et al. 2021a):

$$158 \quad C = C_T + C_A, \quad (3)$$

$$159 \quad N = N_T + N_A, \quad (4)$$

$$160 \quad R_T = C_T / N_T, \text{ and} \quad (5)$$

$$161 \quad R_A = C_A / N_A. \quad (6)$$

162 where C is the TOC content in sample; C_T and C_A are the TOC contents from terrestrial and autochthonous
163 sources, respectively, in the sample; N is the TON content of sediment or suspended particle sample; N_T is
164 the TON content of terrestrial organic matters in the sample; N_A is the TON content of autochthonous matters
165 in the sample; R_T is the C/N value of terrestrial organic matters; and R_A is the C/N value of autochthonous
166 organic matters.

167 The values of N_T , N_A , C_T , and C_A were calculated by Eqs. (3)–(6):

$$168 \quad N_T = (C - R_A \times N) / (R_T - R_A), \quad (7)$$

169
$$N_A = (C - R_T \times N) / (R_A - R_T), \quad (8)$$

170
$$C_T = R_T \times (C - R_A \times N) / (R_T - R_A), \text{ and} \quad (9)$$

171
$$C_A = R_A \times (C - R_T \times N) / (R_A - R_T). \quad (10)$$

172 N_T , N_A , C_T , and C_A were calculated by substituting the values of R_A , R_T , C , and N into Eqs. (7)–(10),
173 and the relative contribution rates of terrestrial and autochthonous sources were calculated by Eqs. (11) and
174 (12):

175
$$P_T = C_T / C, \text{ and} \quad (11)$$

176
$$P_A = C_A / C. \quad (12)$$

177 P_T and P_A are the relative contribution rates of terrestrial and autochthonous sources, respectively.

178 P_T and P_A based on $\delta^{13}\text{C}$ were calculated using Eqs. (13) and (14) (Koszelnik et al. 2018):

179
$$\delta^{13}\text{C} = \delta^{13}\text{C}_T \times P_T + \delta^{13}\text{C}_A \times P_A, \text{ and} \quad (13)$$

180
$$P_T + P_A = 1. \quad (14)$$

181 $\delta^{13}\text{C}_T$ is the $\delta^{13}\text{C}$ value of terrestrial organic matters in sample, and $\delta^{13}\text{C}_A$ is the $\delta^{13}\text{C}$ value of autochthonous
182 organic matters in sample.

183 2.4 Statistical analysis

184 Three parallel analyses were performed for all measured indices, and test results were expressed as the
185 average value of three parallel analyses (error range of the results of the three analyses < 5%).

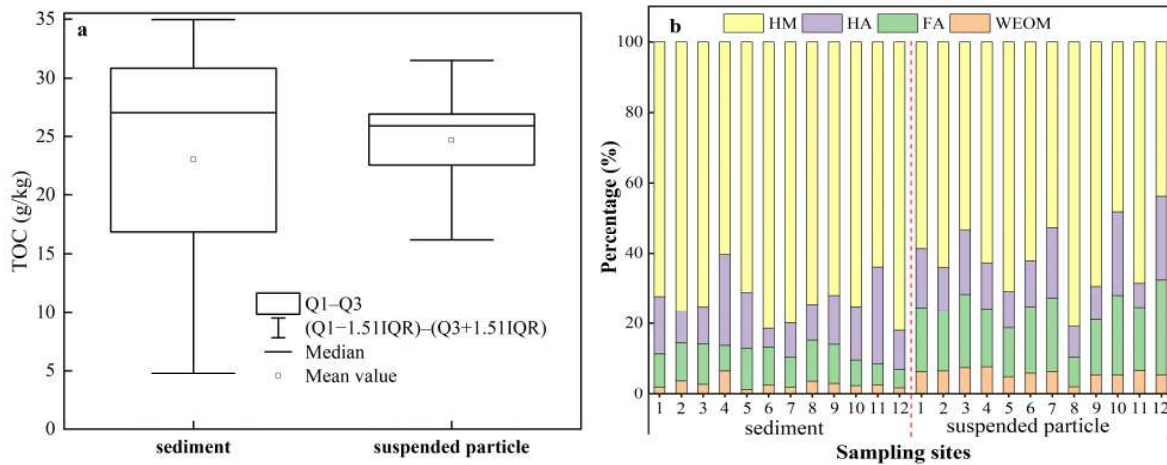
186 The distribution map of sampling sites was drawn using the ArcGIS 10.2. Figures about the contents,
187 composition, fluorescence intensity, and fluorescence components of the WEOM of SOM and SPOM and the C/N
188 and $\delta^{13}\text{C}$ values of sediment and suspended particle samples were created using the Surfer 14.0 software.

189 The minimum, maximum, mean, and standard deviations of the analytical indices were determined using the
190 SPSS 19.0 software. The correlation and significance analyses between two data sets were performed using the
191 Pearson correlation coefficient method and ANOVA, respectively, in the SPSS 19.0 software.

192 3 Results and Discussion

193 3.1 Contents and compositions of SOM and SPOM in Hulun Lake

194 The contents of SOM and SPOM in Hulun Lake were characterized using the TOC concentrations in
195 suspended particles and sediments, respectively. SOM and SPOM contents range from 4.79 g/kg to 34.95 g/kg
196 (mean = 23.04 ± 10.27 g/kg) and from 16.19 g/kg to 31.49 g/kg (24.70 ± 4.63 g/kg), respectively (**Fig. 2**). The
197 average content of SPOM was slightly higher than that of SOM, but the difference was not significant ($P > 0.05$).



198
 199 **Fig. 2** (a) Contents of total organic carbon (TOC) in sediments and suspended particles and (b) compositions of
 200 sediment (SOM) and suspended particulate (SPOM) organic matters in Hulun Lake. Q1: first quartile, Q3: third
 201 quartile, IQR: interquartile range, HM: humin, HA: humic acid, FA: fulvic acid, WEOM: water-extractable
 202 organic matter.

203 The contents of SOM and SPOM in the north and west were higher than those in the east and south. The
 204 spatial distribution characteristics of SOM and SPOM in Hulun Lake were affected by the land use around the
 205 lake, dominant wind direction, and sediment particle size distribution. The grass shoreline in the northwest of
 206 Hulun Lake is 150 km long (Song et al. 2011), and animal husbandry is developed. In addition, the perennial
 207 prevailing wind in the basin is northwest wind, which leads to a large amount of hay, animal manure, or soil from
 208 the semiarid grassland in the northwest into the lake followed by the surface runoff formed by wind or rainfall.
 209 According to statistics, the annual amount of hay into the Hulun Lake is about 3348 t (frozen period: 1580 t,
 210 nonfrozen period: 1768 t) (Zhang et al. 2019b). The decomposition and settlement of hay can introduce a large
 211 amount of dissolved and particulate organic matters. While the east bank of Hulun Lake is the lakeside dune zone,
 212 the organic matter input is less than that of the west lake. In addition, the sediments in southeastern part are sandy
 213 sediments with large particle size, whereas the sediments in the northwestern part are silts with small particle size.
 214 Most organic matters (more than 85%) are distributed in fine particles, and the lowest proportion is distributed in
 215 coarse particles (Shang et al. 2013). The median grain size (D_{50}) of surface sediments in Hulun Lake was
 216 significantly negatively correlated with TOC content ($D_{50} = -2.7197\text{TOC} + 104.38$, $R^2 = 0.6505$, $P < 0.01$). Hence,
 217 the SOM contents in the north and west lake areas with small sediment particle size were higher than those in the
 218 east and south lake areas with large sediment particle size. Hulun Lake is a shallow lake with a wide lake surface
 219 and an average water depth of 5.7 m. Under the disturbance of wind and waves, the sediment resuspension is also
 220 one source for SPOM (Ao et al. 2020; Liu et al. 2019b). Thus, the spatial distribution of SPOM is also affected
 221 by the SOM distribution.

222 SOM and SPOM consist of four chemical components, i.e., WEOM, HA, FA, and HM. Among these
223 components, WEOM is soluble in water, an active component in SOM and SPOM, and has the highest
224 bioavailability and the lowest proportion (Li et al. 2018; Ni et al. 2021; Zhang et al. 2021). The WEOM contents
225 in SOM and SPOM were 0.15–1.34 and 0.45–2.28 mg/kg, respectively, and had mean values of 0.63 ± 0.35 and
226 1.49 ± 0.50 g/kg, respectively. WEOM took 3.0% and 6.0% of SOM and SPOM, respectively. HM is not soluble
227 in acid or alkali and is difficult to decompose (Pham et al. 2021; Zhang et al. 2019a). HM is the most stable form
228 of SOM and SPOM. The HM contents in SOM and SPOM were 2.89–26.73 and 7.32–22.33 g/kg, respectively,
229 and had mean values of 17.31 ± 7.81 and 15.33 ± 4.36 g/kg, respectively. These mean values accounted for 73.7%
230 and 61.2%, respectively, of the total amount of SOM and SPOM, respectively. FA and HA can be dissolved in
231 acid or alkali, and their stabilities are between those of WEOM and HM. The contents of FA in SOM and SPOM
232 ranged from 0.32 g/kg to 3.87 g/kg and from 1.72 g/kg to 5.61 g/kg, respectively, and accounted for 9.2% and
233 18.1%, respectively, of the total amounts of SOM and SPOM, respectively. The contents of HA in SOM and
234 SPOM ranged from 1.24 g/kg to 5.32 g/kg and from 1.77 g/kg to 5.40 g/kg, respectively, and accounted for 14.2%
235 and 14.8% of the total amount of SOM and SPOM, respectively. The relative ratios of the components
236 (WEOM:FA:HA:HM) in SOM and SPOM were 1.0:3.6:4.5:27.4 and 1.0:2.9:2.3:10.3, respectively. HM was the
237 dominant component of SOM and SPOM in Hulun Lake.

238 *3.2 Fluorescence components of WEOM in SPOM and SOM in Hulun Lake*

239 WEOM takes the smallest proportion in SOM and SPOM but is the most active component and is most easily
240 used by microorganisms. WEOM contains different fluorescence components with different characteristics in
241 structure, humification degrees, and sources (Derrien et al. 2019; Han et al. 2021; Liu et al. 2019a). Therefore,
242 further analysis of the fluorescence composition of WEOM can further understand the composition, source, and
243 biodegradability of SOM and SPOM. The WEOM fluorescence spectra of SOM and SPOM in Hulun Lake
244 reflected humus- and protein-like fluorescence peaks. The three-dimensional fluorescence spectra of EEM were
245 analyzed using the PARAFAC, and 4 and 3 fluorescence components with single-emission wavelength were
246 determined. As shown in **Table 1**, the four fluorescence components of WEOM in SOM included 1 terrigenous
247 fulvic acid-like component C1, 2 terrigenous humic acid-like components C2 and C3, and 1 tryptophan-like
248 component C4 formed by biodegradation. The three fluorescence components of WEOM in SPOM included
249 fulvic acid-like component C1', terrigenous humic acid-like component C2', and tryptophan-like component C3'.

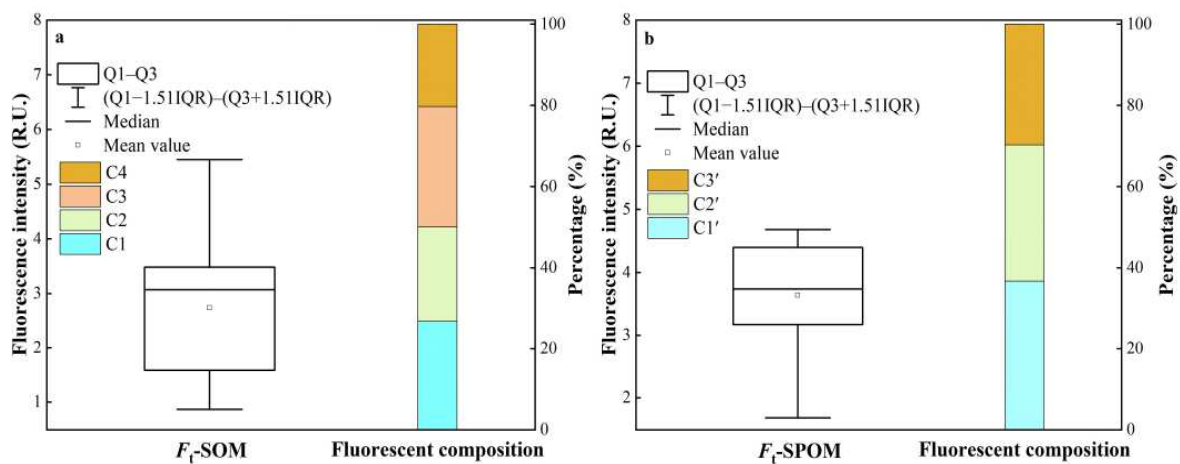
250

Table 1 WEOM components of SOM and SPOM in Hulun Lake.

Organic matter	Component	Ex _{max} (nm)	Em _{max} (nm)	Description
SOM	C1	238	410	fulvic acid-like fluorescence
	C2	276	490	humic acid-like fluorescence
	C3	252,362	454	humic acid-like fluorescence
	C4	220,276	340	tryptophan-like fluorescence
SPOM	C1'	244	434	fulvic acid-like fluorescence
	C2'	272, 368	480	humic acid-like fluorescence
	C3'	224, 276	346	tryptophan-like fluorescence

252 Ex_{max}: maximum excitation wavelength, Em_{max}: maximum emission wavelength, C1: component 1 of SOM, C2:
 253 component 2 of SOM, C3: component 3 of SOM, C4: component 4 of SOM, C1': component 1 of SPOM, C2':
 254 component 2 of SPOM, C3': component 3 of SPOM.

255 The total fluorescence intensity of WEOM in SOM (F_i -SOM) ranged from 0.87 R.U. to 5.45 R.U. (mean =
 256 2.74 ± 1.33 R.U.), and humus-like components (C1 + C2 + C3) took 79.9% of F_i -SOM (**Fig. 3a**). The total
 257 fluorescence intensity of WEOM in SPOM (F_i -SPOM) ranged from 1.60 R.U. to 4.68 R.U. (mean = 3.64 ± 0.88
 258 R.U.), and humus-like components (C1' + C2') took 70.4% of F_i -SPOM (**Fig. 3b**). Humus-like components were
 259 the dominant fluorescence components for WEOM in SOM and SPOM, but the proportion of humus-like
 260 components in SOM was slightly higher than that in SPOM.

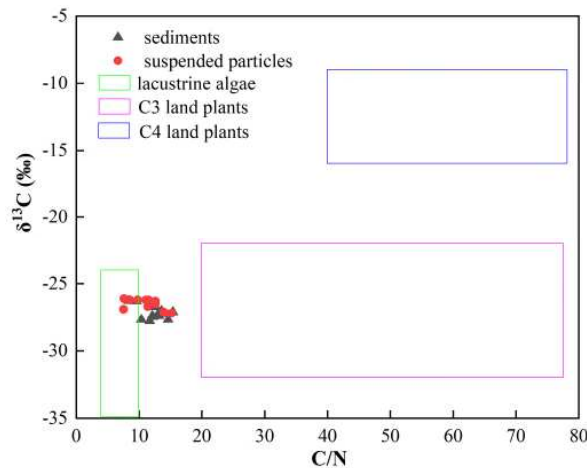


261 **Fig. 3** Total fluorescence intensities (F_i) and fluorescent compositions of WEOM in (a) SOM and (b) SPOM
 262 organic matters in Hulun Lake.
 263

264 3.3 Sources of SPOM and SOM in Hulun Lake

265 The organic matter carriers from different sources have different ranges of C/N and $\delta^{13}\text{C}$ and are conservative
 266 in the physical mixing process (Yu et al. 2010). The combination of C/N and $\delta^{13}\text{C}$ can effectively distinguish the

267 source of organic matter (Pan et al. 2019; Yu et al. 2010). The C/N values of aquatic phytoplankton range from 4
 268 to 10 (Meyers 1994), and the $\delta^{13}\text{C}$ values usually range from -42‰ to -24‰ (Liu et al. 2021). The C/N of most
 269 terrestrial higher plants is > 20 (Meyers 1994), and the C/N and $\delta^{13}\text{C}$ values of C3 plants range from 20 to 80 and
 270 from -32‰ to -22‰ , respectively (Kendall et al. 2001; Ogrinc et al. 2008). The values of C/N and $\delta^{13}\text{C}$ for C4
 271 plants generally range from 40 to 80 and -16‰ to -9‰ , respectively (Kendall et al. 2001). The C/N values of
 272 organic matters from terrestrial soils range from 8 to 15 (Ogrinc et al. 2008), and their $\delta^{13}\text{C}$ values are similar to
 273 those of terrestrial plants. C/N is less than 8, indicating large lake aquatic biomass, high primary productivity, and
 274 high proportion of autochthonous sources. The C/N is between 8 and 15, indicating that SOM and SPOM are
 275 influenced by terrestrial and autochthonous sources. C/N is greater than 15, indicating that SOM and SPOM come
 276 from terrestrial input. The C/N ranges of surface sediments and suspended particles in Hulun Lake were 8.37–
 277 15.39 (mean = 12.21 ± 2.01) and 7.53–15.20 (mean = 11.33 ± 2.55), respectively. The values of $\delta^{13}\text{C}$ in sediments
 278 and suspended particles ranged from -27.78‰ to -26.25‰ (mean = $-27.18\text{‰} \pm 0.52\text{‰}$) and from -27.20‰ to
 279 -26.10‰ (mean = $-26.57\text{‰} \pm 0.43\text{‰}$), respectively. According to the relationship of C/N and $\delta^{13}\text{C}$ of different
 280 end-member substances proposed by Meyers (1994) as shown in **Fig. 4**, SOM and SPOM in Hulun Lake were
 281 influenced by the joint effects of autochthonous and terrestrial sources, and the influence of autochthonous inputs
 282 on SPOM might be greater than that on SOM.



283
 284 **Fig. 4** Carbon-nitrogen ratio (C/N) and stable carbon isotope ($\delta^{13}\text{C}$) values of lacustrine algae, C3 land plants, C4
 285 land plants, and sediments and suspended particles in Hulun Lake.

286 The relative contributions of terrestrial (P_t) and autochthonous (P_a) sources were estimated using two end-
 287 member mixing models based on C/N and $\delta^{13}\text{C}$, respectively, to further analyze the relative contributions of
 288 autochthonous and terrestrial sources to SOM and SPOM in Hulun Lake (**Table 2**).

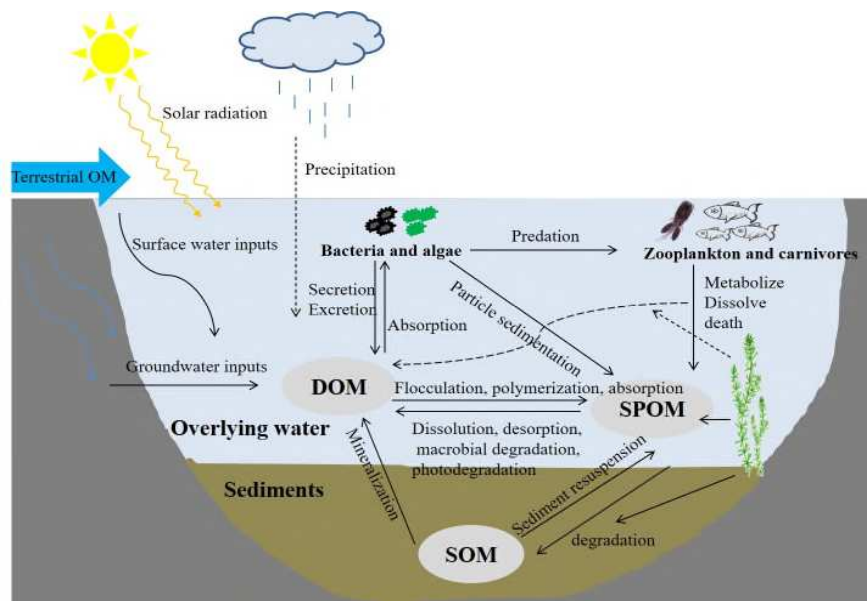
289 **Table 2** Relative contributions of terrestrial (P_t) and autochthonous (P_a) sources of SOM and SPOM in Hulun
 290 Lake.

Item	Calculated by C/N			Calculated by $\delta^{13}\text{C}$			
	C/N	P_a (%)	P_t (%)	$\delta^{13}\text{C}$ (‰)	P_a (%)	P_t (%)	
SOM	Min	8.37	12.8	40.5	-27.78	14.3	55.6
	Max	15.39	59.5	87.2	-26.25	44.4	85.7
	Mean	12.21 \pm 2.01	29.3 \pm 13.4	70.7 \pm 13.4	-27.18 \pm 0.52	26.2 \pm 10.3	73.8 \pm 10.3
SPOM	Min	7.53	10.6	34.8	-27.20	17.5	59.4
	Max	15.20	65.2	89.4	-26.10	40.6	82.5
	Mean	11.33 \pm 2.55	32.8 \pm 18.8	67.2 \pm 18.8	-26.57 \pm 0.43	31.1 \pm 9.3	68.9 \pm 9.3

291 The P_t values of SOM and SPOM estimated by C/N were 70.7% \pm 13.4% and 67.2% \pm 18.8%, respectively,
292 and the P_a values of SOM and SPOM were 29.3% \pm 13.4% and 32.8% \pm 18.8%, respectively. The P_t values of
293 SOM and SPOM estimated by $\delta^{13}\text{C}$ were 73.8% \pm 10.3% and 68.9% \pm 9.3%, respectively, and the P_a values of
294 SOM and SPOM were 26.2% \pm 10.3% and 31.1% \pm 9.3%, respectively. The estimation results of the two methods
295 were basically consistent, and SPOM was relatively more affected by autochthonous sources than SOM, and this
296 finding was consistent with the results of preliminary judgment. These results might be because samples were
297 collected in July when the cyanobacterial bloom occurred in Hulun Lake area in summer. During the sampling
298 period, the chlorophyll a concentration ranged from 4.37 mg/m³ to 60.56 mg/m³ (mean = 18.39 mg/m³). Therefore,
299 planktonic algae contribute a certain amount of SPOM as a part of suspended particles.

300 3.4 Stabilities of SPOM and SOM in Hulun Lake

301 The organic matters in a lake water environment exist in forms of DOM, SOM, and SPOM, which can
302 transformed into one another through physical and chemical processes, such as adsorption–desorption, dissolution,
303 condensation, redox, photochemistry, and biological processes of phytoplankton and large and medium-sized
304 aquatic plants and microorganisms (He et al. 2016; Wu et al. 2008) (**Fig. 5**). The stabilities of SOM and SPOM
305 directly affect the migration and transformation of organic matters and pollutants and the environmental toxicities
306 of the pollutants in different media in the lake water environment (Hu et al. 2019; Lipczynska-Kochany 2018;
307 Miller et al. 2020). Relatively stable SOM and SPOM are not easy to biodegrade, which is conducive to carbon
308 deposition and accumulation, and the risk of rerelease and biotoxicity of the pollutants combined with them are
309 relatively small. By contrast, poor stabilities of SOM and SPOM are not conducive to the deposition and
310 accumulation of carbon, and the ecological risks of combined pollutants are relatively high. Therefore, evaluating
311 the stabilities of SOM and SPOM is important in lake water environment protection. The stabilities of SOM and
312 SPOM were evaluated on the basis of source, composition, The percentage of HA in humus acid (PQ value), and
313 HIX value of organic matters.



314

315 **Fig. 5** Occurrence, migration, and transformation of organic matters in a lake environment. OM: organic matter.

316 SOM and SPOM in Hulun Lake came from terrestrial sources, and the contribution rate of terrigenous source
 317 was about 70%. Organic matters from terrigenous sources are humus-like matters, and the humification degrees
 318 are high. In terms of composition, the predominant component of SOM and SPOM in Hulun Lake was HM, which
 319 is difficult to degrade. HM accounted for 73.7% and 61.2% of the total amount of SOM and SPOM, respectively,
 320 and its maximum values could reach 81.9% and 80.9%, respectively. However, WEOM accounted for only 3.0%
 321 and 6.0% of the total amount of SOM and SPOM, respectively. In addition, the fluorescence components of
 322 WEOM were humus-like components, which had a large molecular weight and were relatively difficult to degrade,
 323 whereas tryptophane-like components with small molecular weight and easy to biodegrade accounted for less than
 324 30%. The results of the composition survey were in good agreement with the results of source identification. HIX
 325 can also reflect the humification degree of WEOM. $HIX < 4$ indicates low humification degree of WEOM, and
 326 HIX of 4–10 means the strong humification degree of WEOM and that WEOM is difficult to biodegrade (Huguet
 327 et al. 2009). The HIX values for WEOM in SOM and SPOM of Hulun Lake ranged from 4.21 to 8.54 (mean =
 328 6.40) and from 4.07 to 7.40 (mean = 6.09), respectively. The HIX values were all in the range of 4 to 10, indicating
 329 that the WEOM in SOM and SPOM of Hulun Lake had a strong humification degree and were difficult to
 330 biodegrade. Compared with those of Taihu Lake, with the same area and eutrophication level as Hulun Lake, the
 331 HIX values of WEOM in SOM and SPOM of Hulun Lake were significantly ($P < 0.05$) higher (Wang et al. 2018),
 332 indicating significantly higher humification degrees (**Table 3**).

333 **Table 3** Humification index (HIX) values of WEOM in SOM and SPOM of Hulun and Taihu Lakes.

Lake	HIX	Min	Max	Mean	SD
------	-----	-----	-----	------	----

Hulun Lake	SOM	4.21	8.54	6.40	1.29
	SPOM	4.07	7.40	6.09	0.85
Taihu Lake	SOM	2.37	3.68	3.02	0.08
	SPOM	2.81	5.32	4.04	0.15

334 Min: minimum value, Max: maximum value, SD: standard deviation.

335 The PQ value can also be used to characterize the stabilities of SOM and SPOM (Bulosan-Atendido et al.
336 2005; Satisha and Devarajan 2005; Yang et al. 2019; Zhang et al. 2020). A high PQ value results in complete
337 humification process of organic matters, stable organic matters, accumulation of organic matters in sediments and
338 suspended particles, and low contribution to the carbon cycle. By contrast, a low PQ value results in poor stability
339 of organic matters, recycling of organic carbon, and increased contribution to the carbon cycle. PQ values were
340 calculated using Eq. (15):

$$341 \quad PQ = HA / (WEOM + HA + FA). \quad (15)$$

342 The PQ values of SOM and SPOM in Hulun Lake ranged from 0.29 to 0.76 (mean = 0.52 ± 0.13) and from
343 0.22 to 0.46 (0.37 ± 0.07), respectively. The PQ values of SPOM were significantly lower than those of SOM (P
344 < 0.01). The comprehensive evaluation results of source, component, and HIX and PQ values showed that the
345 stability of SPOM in Hulun Lake was significantly lower than that of SOM. As one of the important forms of
346 organic matters in the lake water environment, SPOM plays an important role in carbon cycle and pollutant
347 migration and transformation in the lake (He et al. 2021a, b; Hu et al. 2019; Lehmann et al. 2004). Compared with
348 SOM, SPOM is more likely to be transported in water bodies. For shallow lakes, under the effects of wind
349 disturbance and hydrodynamic force, the frequency of migration and transformation between SPOM, DOM, and
350 SOM is increased, and the mechanism is complex. The results of this study indicated that the stability of SPOM
351 in Hulun Lake was significantly lower than that of SOM, which might be because algae was one of the important
352 sources of SPOM during the cyanobacterial outbreak in July. The main components of algal organic matters are
353 protein-like substances with low humification degree and high bioavailability (Lee et al. 2016; Nicolau et al. 2015;
354 Villacorte et al. 2013). In recent years, the climate of Hulun Lake shows a trend of warming and drying, and bloom
355 outbreaks have become normal in summer (Bao et al. 2021; Chen et al. 2012). With increasing temperature, the
356 area of the bloom increases, and the duration of the bloom is prolonged, which may contribute to SPOM. Studies
357 showed that the release effects of SOM and SPOM were enhanced, and stabilities deteriorate with increasing
358 temperature (Gudasz et al. 2010; Lipczynska-Kochany 2018). This finding may be a common phenomenon in
359 other lakes located in cold and arid regions. Therefore, studies on the occurrence characteristics of organic matters
360 in different media, and the migration and transformation of organic matters and endogenous pollutants, which are

361 closely related to the environmental behavior of the organic matters, in different types of lakes in China especially
362 for the lakes in cold and arid regions under climate change are important for lake environmental protection.
363 Moreover, these areas should be further studied in the future.

364 **4. Conclusion**

365 The occurrence, sources, and stabilities of SOM and SPOM in Hulun Lake were investigated and compared.
366 Results showed that SOM and SPOM were different in content, chemical composition, fluorescence composition
367 of WEOM, source, and stability.

368 The average content of SPOM in Hulun Lake was slightly higher than that of SOM, but no significant
369 difference was observed. The contents of SOM and SPOM showed a spatial distribution, i.e., higher in the north
370 and west than in the east and south, due to the influences of land use, dominant wind direction, and sediment grain
371 size distribution. The dominant component of SOM and SPOM was HM. The average proportion of HM in SOM
372 was 73.7%, which was higher than that in SPOM (61.2%). The proportions of WEOM in SOM and SPOM were
373 the smallest, but the WEOM proportion in SPOM was higher than that in SOM. The dominant fluorescence
374 component of WEOM in SOM and SPOM was the humus-like component.

375 According to the analysis results of C/N and $\delta^{13}\text{C}$, SOM and SPOM were influenced by terrestrial and
376 autochthonous sources but came from terrestrial input. The relative contribution rate of terrestrial source was
377 about 70%. SOM was more affected by terrestrial source than SPOM.

378 The SOM and SPOM in Hulun Lake had stronger humification degrees than those in Taihu Lake and were
379 not easy to biodegrade. Moreover, the stability of SPOM was significantly lower than that of SOM, which might
380 be affected by the cyanobacterial bloom in summer. In recent years, the climate of Hulun Lake showed a trend of
381 warming and drying, and the normal occurrence of cyanobacterial bloom might have a remarkable influence on
382 the stability of SOM and SPOM. In the future, the studies on the occurrence, migration, and transformation
383 characteristics and stability of organic matters and endogenous pollutants in different media in lake water
384 environment in cold and arid regions under climate change should be strengthened.

385

386 **Declarations**

387 ● **Ethics approval and consent to participate:** Not applicable.

388 ● **Consent for publication:** Not applicable.

- 389 ● **Availability of data and materials:** The datasets used and/or analyzed in the study are available from the
390 corresponding author upon reasonable request.
- 391 ● **Competing interests:** The authors declare no competing interest.
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395 writing the manuscript. Junyi Chen collected and analyzed the samples. Shuhang Wang formulated the
396 sampling and experimental schemes. Wei Li checked the quality of the paper. All authors read and approved
397 the final manuscript.

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