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Effects of Dissolved Organic Matter (DOM) on Perfluorooctanoic Acid (PFOA) in a Seagoing River—A Case Study of the Wanggang River Flowing into the East China Sea

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Abstract: Perfluorooctanoic acid (PFOA) is an emerging environmental pollutant that has attracted widespread attention. In this study, water samples were collected from the Wanggang River in the eastern coastal area of China, and the PFOA and dissolved organic matter (DOM) levels were measured. The results show that the PFOA concentration in the water bodies ranges from 3.2 to 52.9 ng·L^{-1}, and the average value is 27.1 ± 13.4 ng·L^{-1}, indicating an intermediate level. Two protein-like (C2, C3) and two humus-like (C1, C4) DOM components in the Wanggang River are attributed to rainfall and human activities. Differences are observed in the DOM components before and after the flood season. The humus-like components are higher in the post-flood season, and are one of the factors affecting PFOA distribution and concentration in the Wanggang River. The results provide data support for monitoring and evaluating PFOA in rivers and help formulate PFOA pollution management strategies. In future research, it might be better to define the interaction between DOM and emerging organic pollutants by using 17 PFASs as subjects.

Keywords: dissolved organic compounds; perfluorooctanoic acid; seagoing river; Wanggang River

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1. Introduction

Perfluorooctanoic acid (PFOA) is a synthetic macromolecular organic compound with the formula $C_8HO_2F_{15}$. It is one of the most reported perfluoroalkyl compounds and polyfluoroalkyl substances (PFASs) [1]. Due to the high bonding energy of the C-F bond (485.3 KJ·mol^{-1}), PFOA has excellent chemical and thermal stability, and hydrophobic and oleophobic properties [2,3]. Since it was first synthesized in the 1950s, PFOA has been widely used in the chemical industry and for consumer goods, such as oil- and waterproofing textiles, leather, and furniture [4]. It has also been widely added to commonly used products, such as lubricants, pesticides, flame retardants, and cosmetics [3,5]. As an emerging organic pollutant, PFOA represents a worldwide concern owing to its persistence, bioaccumulation, toxicity, potential negative impacts on humans and animals, and the potential for long-range transportation [6]. Some studies show that PFOA is widespread in surface water [7], soil [8], air [9], sediments [10], fish [11], and human blood [12]. PFOA has been detected in seven major river systems in China in recent years, because of its discharge by domestic sources in large cities and industrial sources in industrial parks along rivers.

Dissolved organic matter (DOM) is widely found in aquatic ecosystems. It has a complex structure, stable properties, and other characteristics [13]. It has been used to characterize the organic matter content in water bodies and the level of organic matter pollution [14]. It has exogenous and endogenous sources. Exogenous inputs include organic fertilizers and domestic sewage from rural and urban areas [15]. Endogenous sources are mainly related to the waste from aquatic animals and degradation by aquatic microbes. DOM can adsorb or bind to organic contaminants through hydrogen bonds, covalent bonds, hydrophobic interaction,

distribution, and other forces [16], influencing the degradation and bioavailability of organic contaminants, such as polycyclic aromatic hydrocarbons [17] and estrogen [18]. Studies show that the molecular weight of the DOM can alter the binding capacity of PFASs to the DOM, and proteins inhibit the bioconcentration of PFASs in Chironomus plumosus larvae [19]. In addition, salinity affects the adsorption of PFAS to organic components [20]. Since PFOA is a widespread PFAS, the relationship of PFOA with DOM, its influencing factors, and differences in the concentration between different periods need further study.

Rivers are a vital component of ecosystems. They are the dominant water resource, the location of the biogeochemical cycle of PFASs [21], and the main carrier of DOM. The Wanggang River is a key seagoing river flowing through Yancheng City, China. It has various tributaries, forming a large and dense river network. The land bordering the river is used for various purposes and represents a significant and complex pollutant source. Previous studies on the Wanggang River Basin found that the efficiency of domestic sewage collection and treatment in townships along the river was low. Industrial pollution sources in industrial parks, farmland runoff, and aquaculture drainage are the leading factors influencing water quality [22] and may affect the DOM and PFOA concentrations. Therefore, monitoring PFOA pollution and DOM components in different periods in the Wanggang River Basin provides information on the influence of DOM on the PFOA concentration. The objectives of this study are (1) to investigate the PFOA concentration and distribution before and after the flood season (May and September) in the Wanggang River Basin, (2) to analyze the DOM composition and distribution in the Wanggang River Basin, and (3) to explore the effect of DOM on the PFOA concentration and distribution.

2. Materials and Methods

2.1. Study Area and Sample Collection

The Wanggang River (120°17′~120°49′ E, 32°57′~33°12′ N) is located in the south-central part of the Dafeng region, Yancheng. It is the main trunk river that drains into the sea from the coastal reclamation area. It is 44 km long with a basin area of 787 km^2 and an average annual runoff of 346 million m^3 per year. According to the topographic and hydrological characteristics, the location of tributaries, and typical pollution types (key agricultural non-point sources, industrial sewage outlets, and urban centers), 30 sampling sites were selected in the Wanggang River from upstream to downstream (Figure 1). Surface water collections were carried out in May 2021 (before the flood season) and September 2021 (after the flood season). We collected 1 L of river water samples at each sampling site at a depth of 0.5 to 1.0 m, using three replicates. The water samples were cryopreserved and transferred to the laboratory as soon as possible and prepared for PFOA content and DOM analysis.

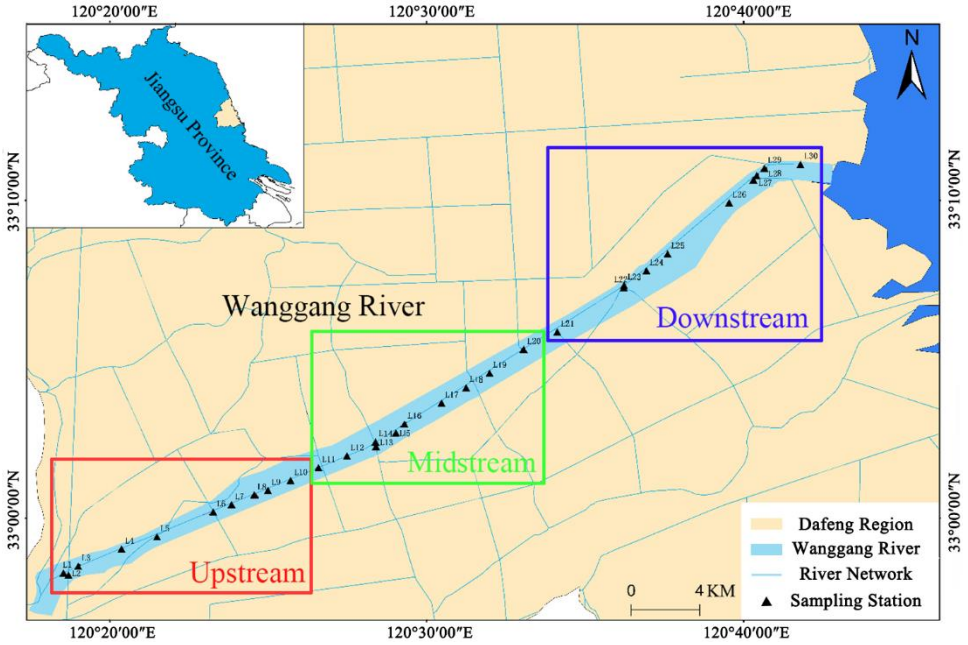

Figure 1. The study area and the sampling sites in the river.

2.2. Measurement of PFOA Content in the Water

The OASIS WAX solid phase extraction (SPE) column was activated sequentially with 4 mL of 0.1% ammonia/methanol solution, 4 mL of methanol, and 4 mL of ultrapure water. The water sample containing the internal standard was then passed through the column. The column was rinsed with 4 mL of ammonium acetate buffer (pH 4.0, 25 mM). Subsequently, the SPE column was naturally dried, and the PFOA in the SPE column was eluted with 4 mL of methanol and 4 mL of a 0.1% ammonia/methanol solution. The eluent was collected into a 15 mL polypropylene centrifuge tube. The collected 8 mL eluate was concentrated to 1 mL under high-purity nitrogen and filtered through a 0.2 μm nylon membrane. The filtrate was placed in a 1.5 mL brown glass vial and stored in a refrigerator at −20 °C for subsequent detection. Finally, the concentration of PFOA in the water samples was quantitatively determined using a high-performance liquid chromatograph with a mass spectrometer (1260HPLC-6400C MS/MS, Agilent Technologies, Santa Clara, CA, USA) and an electrospray ion source in negative ionization mode [23].

Settings of the liquid phase were as follows: a Poroshell 120 EC-C18 (4.6 × 100 mm, 2.7 μm, Agilent, CA, USA) was used; the column temperature was constant at 40 °C, and the sample injection volume was 5 μL. We used a binary mixture solution as a mobile phase, where mobile phase A consisted of 10 mM of ammonium acetate solution, and mobile phase B was 100% chromatographic grade methanol (A: B 5: 95, the flow rate 0.4 mL·min^{-1}). The proportions of A and B and the flow rate over time in the mobile phases are shown in Table 1.

Table 1. Settings of flow rate and proportions of the mobile phases over time in liquid mass spectrometry.

Time (min)	A (%)	B (%)	Flow Rate (mL·min^{-1})
0.00	80	20	0.4
14.00	10	90	0.4
16.00	10	90	0.4
16.01	80	20	0.4
20.00	80	20	0.4

The mass spectrometry conditions were as follows. Multi-reaction monitoring (MRM) was used to scan fragment ions. The temperature of the carrier gas (helium) was 350 °C, and the capillary voltage was 4000 V. The auxiliary gas (nitrogen) temperature was 350 °C, the pressure was 345 kPa, and the flow rate was 100 L·min^{-1}. The atomization temperature was 380 °C, the ion source voltage was 180 V, the cycle was 10 min, and the ionic mass–mass ratio (*m*:*z*) was 413:169.

2.3. Quality Assurance and Quality Control (QA/QC)

Quality assurance and quality control were performed throughout the entire process of sample collection and transportation, PFOA extraction, and on-machine testing. In the sample collection, we avoided using PTFE or other fluoropolymer materials during sampling to prevent background contamination. The sampling bottles were rinsed three times with ultrapure water before collecting water samples. During the transportation of samples, container blanks and transportation blanks were set and ultrapure water was used as the monitoring medium.

Background contamination and cross-contamination should also be avoided during the extraction of PFOA. We avoided the use of PTFE or other fluoropolymer materials in extraction to ensure that the analysis results were not affected. PFOA extraction, enrichment, purification, and other processes were monitored in order to prevent background contamination and cross-contamination between samples. In addition, replicates were set up every 10 samples to maintain the reproducibility of the analysis.

2.4. DOM Measurement

The water samples were filtered through a 0.45 μm glass fiber membrane and placed in a washed 1 cm standard quartz cuvette. The three-dimensional spectra of the DOM were measured using a fluorescence spectrophotometer (F-4500, Hitachi High-Technologies Corp., Tokyo, Japan) with a 150 W xenon lamp and a photomultiplier voltage of 350 V. The excitation and emission slit widths were 10 nm, and the scanning speed was 2400 nm/min. The excitation wavelength was 200–450 nm with a 5 nm interval, and the emission wavelength was 250–600 nm with a 1 nm interval. Parallel factor analysis (PARAFAC) was used to analyze the three-dimensional fluorescence spectra using the DOM Fluor toolbox in MATLAB R2015a. The samples resulting from temperature differences, thawing time, and other influences were removed. The validity of the PARAFAC model was tested by split-half analysis and residual analysis to eliminate the overlap between different fluorescence peaks on the fluorescence intensity and decompose the fluorescence spectrum into independent components [24]. Dissolved organic carbon (DOC), another important parameter to evaluate DOM, was measured by a total organic carbon analyzer (TOC-5000A, Shimadzu Corp., Kyoto, Japan).

2.5. Data Processing

Microsoft Excel 2013 (Microsoft, Redmond, WA, USA) was used to calculate the fluorescence intensity of the DOM and PFOA. SPSS 24.0 (IBM, Armonk, NY, USA) was used to conduct a Shapiro–Wilk test to determine if the data followed a normal distribution. Student' *t*-test was chosen when data followed a nor-mal distribution. The fluorescence intensity of the DOM components before and after the flood season and the PFOA concentration in the river were plotted with SigmaPlot 14.0 (SYSTAT, CA, USA). The rainfall data in the Wanggang River Basin were obtained from The China Meteorological Data Service Center (CMDC). SPSS 24.0 software was used for Spearman correlation analysis of PFOA and DOM.

3. Results and Discussion

3.1. Profiles of PFOA in the Wanggang River

The detection rate of PFOA is 100% for the 30 sampling sites. The PFOA concentration ranges from 3.2 to 52.9 ng·L^{-1}, and the average is 27.1 ± 13.4 ng·L^{-1}. The pre-flood PFOA

concentrations range from 4.7 to 52.9 ng·L^{-1} and from 28.4 ± 13.8 ng·L^{-1}. The maximum pre-flood (post-flood) value occur at sample site L17 (L17), and the minimum value at L5 (L4). The PFOA concentration is generally lower after than before the flood season, with concentrations ranging from 3.2–43.7 ng·L^{-1} and an average of 25.8 ± 13.1 ng·L^{-1}.

The PFOA concentration in the Wanggang river is higher than that of the Jiulong river, Yangtze River, and Poyang Lake, similar to that of the Daling River and Taihu Lake, and lower than that of the Baiyangdian River and Pearl River (Table 2). In addition to differences in hydrological conditions, such as the water type, flow rate, and flux, the types and outputs of the surrounding industries and anthropogenic activities are also responsible for the differences in the PFOA concentrations in rivers or lakes. The northern catchment of the Pearl River Basin contains many industrial areas, such as Shaoguan (known for metal mining and related industries) and Qingyuan. The PFOA concentrations in these surface waters are much higher than in other regions, indicating that the PFOA distribution is closely related to regional industrialization [25]. There is no fluorine industry in the Wanggang River Basin, but the area is still affected by chemical, textile, and pharmaceutical industries in the region.

Table 2. Comparison of PFOA concentrations in different rivers.

Study Area	Date	Range (ng·L^{-1})	References
Wanggang River	2021	3.2–52.9	This study
Daling River	2018	6.5–37.9	[25]
Jiulong River	2014–2015	5.5–15	[21]
Yangtze River	2019	8.9–3.6	[2]
Taihu Lake	2019	16.9–39.7	[26]
Poyang Lake	2016	1.8–17	[27]
Baiyangdian Lake	2016	13.6–441	[28]
Pearl River	2019	0.125–1030	[29]

As shown in Figure 2, the PFOA concentration increases and decreases from upstream to downstream. It is the lowest in the upstream region (L1–L10) and the highest in the mid-stream region (L11–L20), and the difference is significant ($p < 0.05$); the PFOA concentration is significantly higher downstream than upstream ($p < 0.05$).

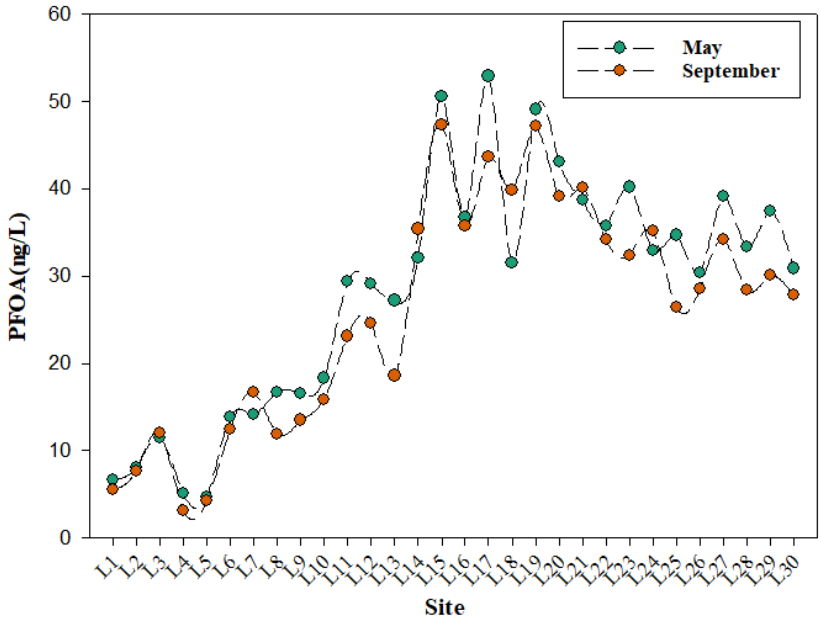

Figure 2. PFOA concentrations before the flood season (May) and after the flood season (September) in the Wanggang River Basin.

The flood season of the Wanggang River lasts from July to September. The influx of heavy rainfall and surface runoff during the flood season dilute the pollutants in the water [30]. According to the comparison in Figure 3, there is a higher PFOA concentration before than after the flood season downstream, implying contaminants in this reach are affected by the rain. A government report about the water quality of Wanggang River basin shows that the Wanggang River is slightly polluted, the water quality is significantly higher upstream than downstream, and the pollution sources are the seepage of wastewater treatment plants and the discharge of rural domestic sewage. The L1–L10 sites were located in the upstream region of the Wanggang river at the confluence of large tributaries. These areas have a high water flow, resulting in pollutant dilution. Therefore, the PFOA concentration is relatively low. The midstream region is surrounded by chemical industries, sewage treatment plants, and large leather factories that use chemicals to produce fiber textiles and leather products. PFOA is widely used in textile and leather products to make them waterproof and stain-resistant. It is also used in floor waxes, wax paper, and wires [31]. The PFOA concentration is significantly higher midstream than upstream due to pollutants discharged from Huafeng Industrial Park. The downstream region of the Wanggang River is surrounded by many livestock and poultry farms and farmland. PFOA is widely used in livestock feed and veterinary medicine products [32]. The PFOA pollution in the downstream region of the Wanggang River is attributed to intensive livestock and poultry breeding, and rural domestic sewage. Therefore, the PFOA concentration is relatively high in the downstream region.

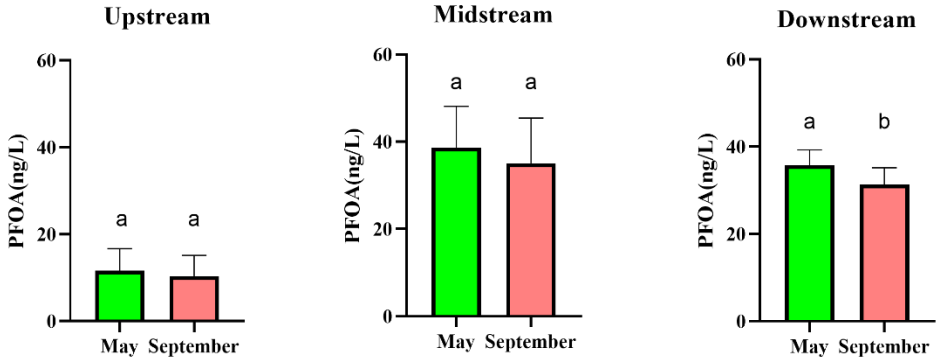

Figure 3. The PFOA concentrations were compared between May and September in different reaches. Note: different letters indicate significant differences, $p < 0.05$.

3.2. DOM Concentration and Distribution

By element composition, the DOM is mainly composed of C, O, H, N, P, and S. The DOC accounts for more than 50% of DOM. Therefore, the DOM content in the water is characterized by DOC concentration [24,33]. The DOC concentration is 2.76–14.79 mg/L (average of 7.21 mg/L) before the flood season and 1.82–16.2 mg/L (average of 4.34 mg/L) after the flood season. The spatial distribution of the DOC concentration follows the order downstream < upstream < midstream, and the maximum value is observed at sampling site L24.

Four fluorescent components (Figure 4) are detected by the PARAFAC method: a humus-like component (C1), a protein-like component (C2), a tryptophan-like protein (C3), and a humic-like component (C4) in the ultraviolet/visible region. The pre-flood DOM in the Wanggang River is dominated by protein-like components, accounting for 79.77% of the total fluorescence intensity. Components C1, C3, and C3 account for 20.23%, 33.42%, and 46.35% of the total, respectively. The humus-like components of the DOM are the dominant components after the flood season, accounting for 67.97% of the total fluorescence intensity. Components C1, C3, and C4 account for 40.05%, 18.55%, and 41.40% of the total fluorescence intensity, respectively.

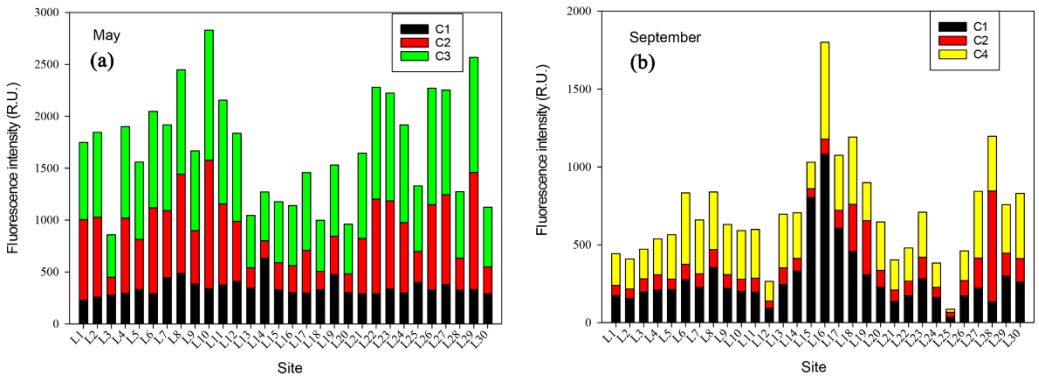

Figure 4. Fluorescence intensity of the DOM components before the flood season (May, (**a**)) and after the flood season (September, (**b**)) in the Wanggang River Basin.

Before the flood season, the minimum value of C1 occurs at sampling site L1 (227.02 R.U.), and the maximum value is observed at L14 (632.02 R.U.). The minimum values of components C1 and C4 occur at the L25 sampling site (34.22 R.U. and 20.46 R.U.), and the maximum values are at L16 (10,082.19 R.U. and 622.89 R.U.). The minimum and maximum values of C3 occur at L3 (409.21 R.U.) and L26 (1121.92 R.U.) before the flood season. The minimum value of C2 is observed at L25 (31.55 R.U.) and the maximum at L28 (712.56 R.U.) after the flood season.

The difference in the DOM composition in the Wanggang River is attributed to seasonal changes and precipitation. The Wanggang River received the majority of the rainfall between May and August 2021 (Figure 5). The protein-like components are higher before the flood season, and the humus-like components are higher after the flood season [34]. The spatial distribution of the DOM indicates that the contents of the C2 and C3 protein-like components are significantly higher in the middle reaches than in the upper and lower reaches ($p < 0.05$). The content of protein-like proteins is affected by high-intensity anthropogenic activities in cities and farmlands [15]. Since the precipitation level is lower before the flood season, pollutant emissions combined with the activity of microorganisms leads to an increase in protein-like components. The C1 content is higher in the midstream than in the upstream and downstream regions, which may be due to the presence of aquaculture facilities in the urban areas and surrounding areas of the middle reaches. They discharge pollutants into the rivers [22]. The total fluorescence intensity after the flood season increases from upstream to midstream and then decreases. Humus-like components in the upstream, midstream, and downstream regions account for 24.39%, 35.10%, and 21.12% of the total fluorescence intensity, respectively, indicating that the entire river is affected by land-based source inputs, resulting in an increase in humus-like substances in the water. The humus-like component C1 is significantly higher in the lower reaches than the upper and middle reaches ($p < 0.05$), indicating that the downstream area is more heavily contaminated and is affected by the input of upstream pollutants and sewage from human activities.

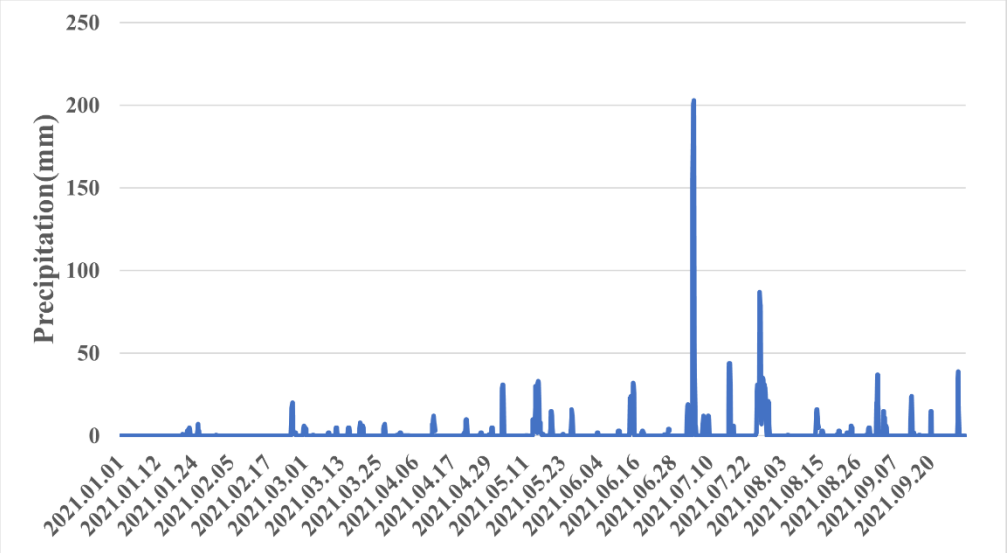

Figure 5. Precipitation in the Wanggang river Basin from January to September 2021.

3.3. Relationship between DOM and PFOA

The correlation between the four components of the DOM and the PFOA in the Wanggang River is listed in Table 3. The protein-like components C2 and C3 show a significant positive correlation in the pre-flood season ($p < 0.01$), indicating that C2 and C3 have the same sources of protein-like substances. There is no correlation between the humus-like components C1 and protein-like substances, indicating that the sources of different elements of DOM in the Wanggang River Basin might be different before the flood season. The main source of humus-like substances is terrestrial inputs, where domestic sewage and microbial activity are sources of protein-like substances [16]. The components C1 and C4 show a significant positive correlation ($p < 0.01$) in the post-flood season, indicating that they have the same sources of humus-like substances. Moreover, C1 and C4 are significantly positively correlated with the protein-like component C2 ($p < 0.01$), indicating that the sources of the three DOM components in the Wanggang River Basin are mainly land-based inputs after the flood season. Surface runoff caused by rainfall, agricultural runoff, and aquaculture pollution are the likely sources of humus-like substances.

Table 3. Spearman correlation coefficients between the PFOA and DOM contents.

	C1	C2	C4	DOC	PFOA
C1		0.564 **	0.658 **	0.081	0.364 *
C2	0.015		0.740 **	0.150	0.254
C3	0.088	0.955 **		0.128	0.041
DOC	0.075	0.182	0.254		0.194
PFOA	0.126	0.141	0.046	0.182	

Note: ** significant at 0.01 level; * significant at 0.05 level. The blue area shows the relationships before the flood season, and the red area shows the relationships after the flood season.

There is no significant correlation between the three components of the DOM and PFOA in the pre-flood season, indicating different sources of PFOA and DOM. The sources of the DOM are domestic sewage and agricultural non-point source pollution, whereas the PFOA source is primarily industrial wastewater discharge. The post-flood C1 component is positively correlated with PFOA. This phenomenon might be related to the types of DOM. The research performed in western Lake Chaohu and its inflow rivers finds a positive correlation between PFAS and DOM, and no significant correlation between PFAS and DOC [18]. They investigate not only PFOA, but also 10 other PFAS homologues, and reveal that the compositions of DOM could influence the distributions of PFAS in the aqueous

phase in the lake. The Wanggang River Basin receives most of the rainfall in May and August, resulting in increases in the humus-like substances in the river. The C1 component may affect the degradation of PFOA or bind to PFOA, influencing the concentration level of the two compounds. Studies show that humus-like substances may be associated with PFASs, including PFOA, and may bind strongly to PFOA [35]. Humus-like substances can reduce the PFOA concentration [36], affecting the PFOA distribution in water. Therefore, the PFOA concentration and distribution in the surface water of the Wanggang River Basin are significantly affected by the DOM composition. Taking consideration of the strong correlation of PFOA with post-flood C1 and the non-existent correlation with other components, humus-like substances are one of the factors affecting PFOA distribution and concentration after the flood season.

3.4. Outlook on Further Studies

In consideration of the complex pollutant sources in the Wanggang River Basin, detection of PFOA is not sufficient to explore the interaction between DOM and emerging organic pollutants. In future research, the monitoring of the 17 PFAS homologues might address the limitation. On this basis, research into the cause of PFAS concentration variation across reaches and seasons, as well as the correlation between PFAS and DOM, is required, and the connection of DOM with PFAS might become more evident to discuss.

4. Conclusions

This study investigated the spatio-temporal PFOA distribution and the DOM composition and distribution in the surface water of the Wanggang River basin. The PFOA content is significantly higher in the middle reaches of the Wanggang River than in the upper and lower reaches, and the PFOA concentration is at an intermediate level. The DOM concentration is affected by rainfall and human activities. Two protein-like components (C2, C3) and two humus-like components (C1, C4) were analyzed. The humus-like components have higher contents in the post-flood season, affecting the PFOA concentration. This finding indicates that the post-flood PFOA content and distribution in the surface water of the Wanggang River Basin are affected by humus-like substances. The results of this study provide insights into the PFOA concentration and distribution in seagoing rivers, and the link between pollutants and organic matter.

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Data Availability Statement: The data used during the study appear in the submitted article.

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