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Research Article

Seasonal variations in dissolved organic matter concentration and composition in an outdoor system for bank filtration simulation

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ABSTRACT

Dissolved organic matter (DOM) in surface waters can vary markedly in character depending on seasonal variations such as rainfall intensity, UV radiations and temperature. Changes in DOM as well as temperature and rainfall intensity over the year can affect the biochemical processes occurring in bank filtration (BF). Identification and characterization of DOM in the surface water could help to optimize the water treatment and provide stable and safe drinking water. This study investigated year-long variations of DOM concentrations and compositions in a surface water of a circulated outdoor pond (research facility) connected to a BF passage. DOM was dominated by humic substances and a changing pattern of DOM in surface water was observed throughout the year. A significant increase of DOM (~ 38%) in surface water was noted in August compared to November. The fluorescent DOM showed that DOM in summer was enriched with the degradable fraction whilst non-degradable fraction was dominated in winter. A constant (1.7 ± 0.1 mg/L) effluent DOM was recirculated in the system throughout the year. DOM removal through BF varied between 4% to 39% and was achieved within a few meters after infiltration and significantly correlated with influent DOM concentration ($R^2 = 0.82$, $p < 0.05$). However, no significant ($p > 0.05$) change in the removal of DOM was observed in two subsurface layers (upper and lower). This study highlights the presence of a constant non-degradable DOM in the bank filtrate, which was not affected by temperature, redox conditions and UV radiations.

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Introduction

Dissolved organic matter (DOM) in the aquatic environment is a complex mixture of organic constituents such as polysaccharides, proteins, amino acids, humic substances, and numerous anthropogenic organic pollutants (Berg et al., 2019; Shi et al., 2021; Wang et al., 2018). DOM originates from different sources (terrestrial, algal, and anthropogenic) and undergoes transformation by photolytic and microbial degradation (Lee et al., 2019; Maqbool et al., 2021; Murphy et al., 2018; Zhang et al., 2019). Since too high DOM concentrations cause esthetic issues (color, odor, and taste), protect pathogens from disinfectants, and generate unwanted disinfection by-products, DOM removal is an essential target of drinking water treatment (DeMarini, 2020; Leenheer and Croué, 2003; Philibert et al., 2022; Richardson, 2009). Additionally, the transport of heavy metals and hydrophobic pollutants can be promoted by DOM (Liu et al., 2007; Müller et al., 2000; Park, 2009; Yan et al., 2008). DOM components showed different removal through physical, chemical, and biological treatment (Edzwald, 1993; Moona et al., 2021). Identification of DOM fractions and monitoring of changes provide a basis for process understanding and eventually optimization of treatment processes.

Bank filtration (BF) is a sustainable water treatment technique that has been used for more than 150 years in different countries (Hu et al., 2016). Previous studies reported significant removals of various organic micropollutants (OMP) and DOM through BF (Filter et al., 2021; Grünheid et al., 2005; Sandhu et al., 2019). The removal through BF is highly influenced by the environmental conditions such as temperature, redox conditions, travel time and raw water quality (Filter et al., 2021; Pan et al., 2018). Previous studies reported 44% to 50% removal in DOM through BF (Jylhä-Ollila et al., 2020; Maeng et al., 2008; Sandhu et al., 2019). In recent years, fluorescence spectroscopy has been increasingly applied to track DOM changes and related effects on drinking water treatment (Jutaporn et al., 2021; Maqbool et al., 2021, 2020; Sweetman et al., 2018). Quantitative and qualitative information can be extracted using parallel factor analyses (PARAFAC) applied to the excitation emission matrix (EEM) obtained from fluorescence spectroscopy (Murphy et al., 2011; Pucher et al., 2019). Several batch and lab-scale studies have evaluated the effect of DOM on BF processes using fluorescence spectroscopy. For example, Abdelrady et al. (2020b) and Xia et al. (2023) reported that DOM changes markedly increases As, Fe, and Mn release to the aquatic environment through BF. Abdelrady et al. (2020a) observed that humic substances in the source water decrease the removal of heavy metal (Cu, Zn and Ni) through BF. Relations between DOM compositions and OMP elimination was also studied (Abdelrady et al., 2019). To the best of our knowledge, the EEM-PARAFAC approach has not been applied for determining the simultaneous impacts of season on DOM composition and concentration in source water and bank filtrate of a full-scale BF. Therefore, it is highly important to assess the seasonal variation of DOM components of source water and their removal through BF.

The current study investigated year-long variations of DOM concentrations and compositions in an outdoor recirculated BF facility. Specific objectives were: (1) to elucidate the impact of seasonal variations (of different environmental variables; temperature, UV radiation and rainfall) on DOM composition in source water and bank filtrate through fluorescence spectroscopy, (2) to determine degradable and non-degradable DOM in the recirculated BF facility and (3) to impacts of season dependent conditions on concentrations of dissolved oxygen (DO) in the surface water and bank filtrate.

1. Materials and methods

1.1. Site description

The research facility (at German Environment Agency, Berlin, Germany) consists of an artificial surface water pond (89 m length, 17.5 m width at the influent side) directly connected to a BF passage (Fig. 1a). The width of pond gradually increases to the bank with a 50 m width. The depth of the surface water in the pond increases by 1.5% from the influent end to the bank. The bank (35 m length, 4.0 m sand layer) consists of a medium sand layer (4 m along the length), followed by the two horizontal layers of fine (0.25 mm, upper layer) and medium (0.40 mm, lower layer) sand (Fig. 1b). The four embedded slow sand filters were not relevant for the study. Surface water samples were collected close to the bank (from both sides of the pond) and in 20 piezometers installed at different distances from the bank (Fig. 1b). Out of 20, 13 piezometers were installed in the lower layer of medium sand and 7 in the middle of the upper layer of fine sand. The surface water continuously infiltrates through the bank before collection through a drain (35 m away from the bank) with an average flow rate of 15.5 m³/hr and is then recirculated. Eventual evaporation losses in the pond are compensated by groundwater (from which Fe and Mn were removed through microbial precipitation). The system had been operating for decades, sufficient for developing of natural flora and fauna.

1.2. Sampling

Samples were collected monthly from December 2020 to November 2021. The piezometers were sampled using a peristaltic pump after discarding twice the dead volume of the piezometers (2 to 5 L). The samples were collected in glass bottles, filtered through membrane filter with 0.45 µm pore size (CHROMAFIL Xtra PA-45/25, Macherey-Nagel, Germany), stored at 4°C, and analyzed within 24 hr.

1.3. Analytical methods

DOC concentrations were quantified with a TOC analyzer (Vario TOC cube, Elementar Analysensysteme, Germany). Iron (Fe) and manganese (Mn) concentrations were determined photometrically according to standard procedures DIN 38,402-A51 and DIN 38,406-2EN. UV absorbance at 254 nm (UV₂₅₄) was measured with a spectrophotometer (Lambda 25 UV/Vis Spectrometer, Perkin Elmer, USA) and specific UV absorbance

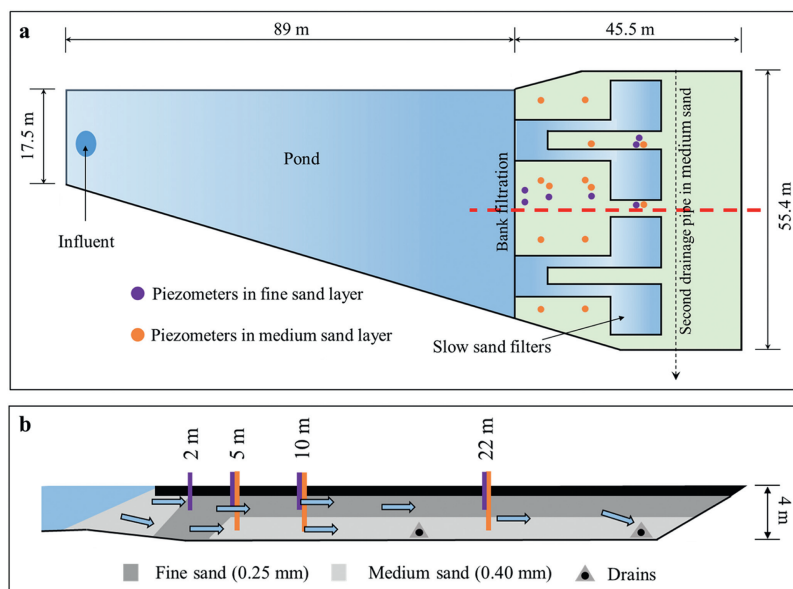


Fig. 1 – (a) Plan view of the system with locations of the 20 piezometers and second drainage pipe, and (b) sectional view along the dotted red line in (a) delineating the positions of the two different layers, the piezometers at different travel distances and the drainage pipes the first of which was not used (the four embedded basins are only included for completeness). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

(SUVA) was calculated by dividing the UV_{254} value by the DOC concentration.

Nitrate (NO_3^-) and ammonium (NH_4^+) were analyzed by ion chromatography (930 Compact IC Flex, Metrohm, Switzerland).

Climatic data is continually measured using an installed weather station.

In situ measurements of pH, DO, and conductivity were carried out using a pH probe (pH 3310, WTW, Germany), a DO flow-through cell (Fibox 4 trace, PreSens, Germany), and a conductivity meter (Cond 340i, WTW, Germany), respectively.

Fluorescence was analyzed using a FluoroMax-4 (HORIBA Jobin Yvon, USA) with excitation wavelengths from 220 to 500 nm (5 nm intervals), emission wavelengths from 250 to 600 nm (2 nm intervals) and a scan speed of 4800 nm/min. The EEM was analyzed for fluorescence regions, listed in Appendix A Table S1.

1.4. PARAFAC modeling

A dataset consisting of 528 EEM from samples of the research systems was processed using the staRdom package for R (Pucher et al., 2019). Briefly, the data processing includes (1) blank subtraction, (2) inner-filter effect correction, (3) Raman normalization, (4) scattering removal, and (5) excitation and emission correction. EEM blanks were acquired using an ultra-pure water sample. PARAFAC was also applied to process (corrected and normalized) EEM data and the most appropriate number of PARAFAC components was determined with split-half validation.

1.5. Statistical analysis

Two-way ANOVA was used to determine the significance of data at different sampling locations at a single distance. One-way ANOVA was used to identify the impact of season dependent boundary conditions on DOM composition. The criterion level of significance (p) was 0.05. Pearson correlation coefficients (r) were calculated to determine linear correlations between different water quality parameters and temperature changes. Principle component analysis (PCA) was performed on the monthly values of different water quality parameters. All the analyses were performed in OriginPro 2019b (OriginLab, USA).

2. Results and discussion

2.1. Water quality and climate parameters

The climate data recorded at the experimental site showed that the average monthly temperatures from December to March were similar and below $5^\circ C$ and a gradual increase in temperature was observed until August (Appendix A Fig. S1). The maximum average temperature of $21^\circ C$ was recorded in July. Averaged daily UVA, UVB and photosynthetically active radiation (PhAR) were below $5 W/m^2$ (mW/m^2 for UVB) from October to March. Maximum values for UVA, UVB and PhAR were recorded in June and July (Appendix A Fig. S2). Based on the rainfall (R_f) data (Appendix A Fig. S3), the site experienced a dry period from October to April and wet period from May

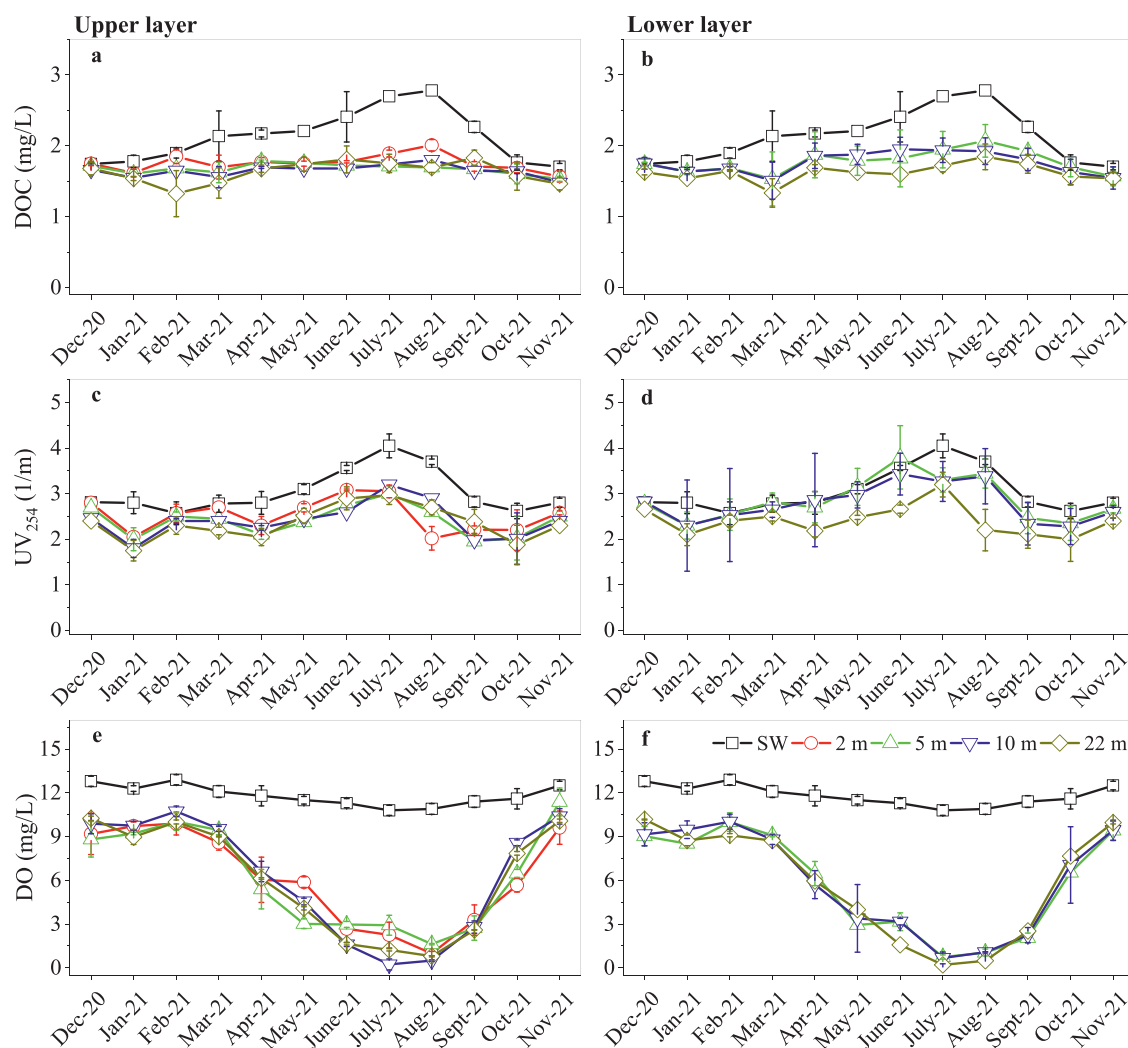


Fig. 2 – Averaged monthly variations in DOC concentrations ((a) and (b)), UV₂₅₄ ((c) and (d)), and DO concentrations ((e) and (f)) in the surface water (SW, included in the left and right columns for comparison) and in the upper fine sand layer (left column) and lower medium sand layer (right column) at defined distances from the bank.

to September. August was found to be the wettest month with 68 mm of rainfall and October (18 mm) was the driest.

The pH of the surface water and bank filtrate was almost constantly between 7.9 and 8.1 throughout the investigated period. The conductivity of the surface water and the bank filtrate did not show significant variation with $1087 \pm 18 \mu\text{S}/\text{cm}$. Fe and Mn were not detected in the surface water and bank filtrate. NO_3^- concentrations were below 1 mg/L and NH_4^+ concentrations were below 0.1 mg/L both in the surface water and bank filtrate throughout the year.

A significant ($p < 0.05$) effect of climatic variations was observed for UV₂₅₄, and DOC and DO concentrations (Fig. 2). The highest average DOC concentration ($2.8 \pm 0.1 \text{ mg/L}$) in the surface water was observed in August (during the wet and warm seasons) and the lowest DOC concentration ($1.7 \pm 0.0 \text{ mg/L}$) in November. DOC gradually increased (by up to 38%) in summer plausibly due to phototrophic activities as PhAR were high in summer (compare Appendix A Fig. S2). UV₂₅₄ in the surface

water showed a trend similar to that of the DOC. The SUVA in surface water was slightly higher in June, July and August (summer) compared to the rest of the year, indicating that the aromaticity of DOM was higher in summer (Appendix A Fig. S4).

The DOC concentration in after the BF passage was constant ($1.7 \pm 0.1 \text{ mg/L}$) throughout the year and no significant ($p > 0.05$) impact of the sand particle size on the DOC concentration was noted. It was observed that DOC removal only occurred before the first sampling points. These results are consistent with the findings of Munz et al. (2019) who reported maximum removal of DOC within the first few meters. No significant ($p > 0.05$) removal of DOC was noted between 2 m and 22 m in the upper sand layer. Similarly, DOC concentrations after 5 m and 10 m in the lower layer did not show significant differences ($p > 0.05$). The DOC at 22 m in the lower sand layer was slightly reduced, presumably due to dilution with water from the upper sand layer. The downward movement of water

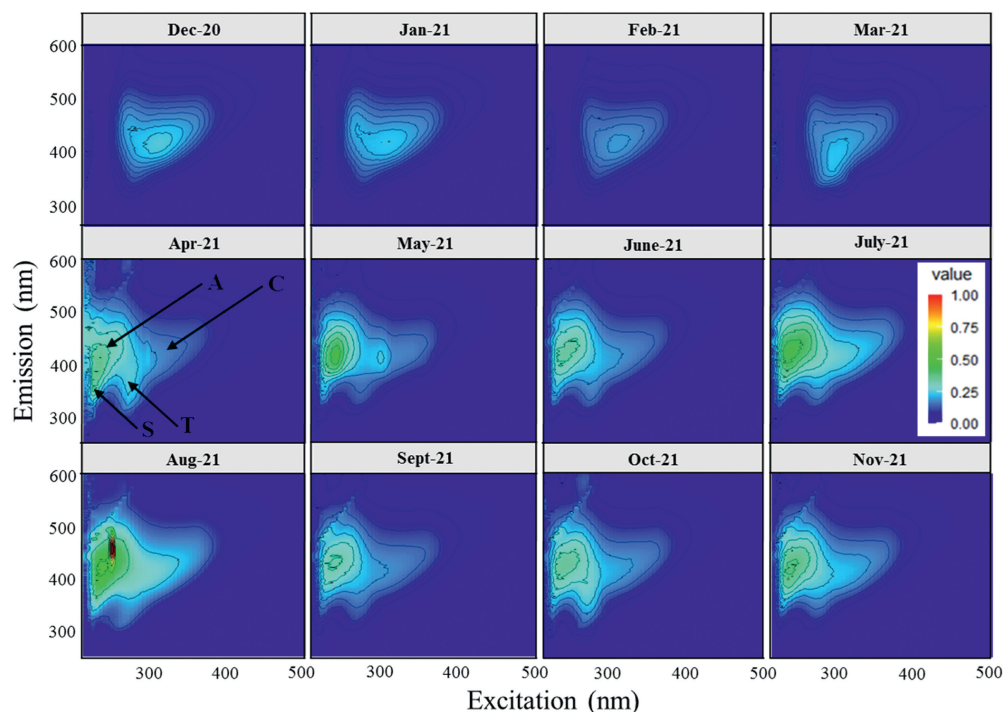


Fig. 3 – EEM of monthly surface water samples collected over a whole year with major signal intensities in the region of humic substance (the letters in the EEM of April indicate the position of components listed in Appendix A Table S1).

from the upper sand layer occurred because both layers infiltrated in a drain at 35 m (in the lower layer, compare Fig. 1). The DOC removal ranged between 4% in November and 39% in August, and the removal correlated with influent DOC concentrations ($R^2 = 0.82$, $p < 0.05$). However, the constant DOC concentration after BF indicated that a constant part of the DOC was recalcitrant while additional DOC in the summer was fully degradable. Similar to partially closed urban water cycles (Jekel et al., 2015), the recirculation of the water in the system leads to a circulation of persistent DOM that represents a constant background concentration. Additional DOM formed in the summer by phototrophic activity thus appears to be fully degradable.

The DO concentration in the surface water was constantly around 12 mg/L during the study. Both subsurface layers were oxic during the dry and cold seasons. The DO concentration in the subsurface layers decreased below 1 mg/L in July and August as the DOM in the surface water increased (Fig. 2). Maximum DO was utilized within the first few meters of the bank passage with almost no further depletion afterward. Specific oxygen demand during BF and before the first sampling point was between 0.0 to 0.4 mg in winters (October to February) and 1.0 to 2.3 mg in summer (March to September; Appendix A Fig. S5). Assuming 3 mg of oxygen required to complete mineralize 1 mg of organic carbon (Jekel and Grünheid, 2006). This theoretical amount showed that probably 0.2% to 25.2% oxygen was consumed by increased aerobic microbial conversion of DOM and 99.8% to 74.8% by particulate organic carbon (POC) deposited in the infiltration zone. A similar observation was reported by the previous studies that oxygen consumption

likely resulted from the degradation of accumulated POC or DOM in the sediments (Bayarsaikhan et al., 2018; Filter et al., 2017).

2.2. Fluorescent dissolved organic matter (fDOM)

The three-dimensional EEM of surface water samples collected throughout a year showed multiple peaks at different excitation and emission wavelengths (Fig. 3). Based on the literature, the peaks can be identified as tryptophan-like (T), SMP-like (S), fulvic-like (A), and humic-like (C) (Bierzo et al., 2009; Li et al., 2013; Maqbool et al., 2021; McKnight et al., 2001).

During the cold season ($T < 5$ °C, December to March) only two fluorophores (peaks A and C) were identified. As the temperature increased, intensities of peaks A and C increased and the other two peaks T and S were also identified, in conjunction with an increase in temperature and higher DOC concentrations in the surface water. The peak T represents protein-like DOM and its origin was reported to be linked to microbial activity and algal blooms leading to an accumulation of DOM in the euphotic zone (Fox et al., 2017; Sorensen et al., 2020; Yamashita and Jaffé, 2008). Stubbins et al. (2014) described that peaks A and C (humic-like) consisted of higher molecular weight and aromaticity with low nitrogen contents. Peaks T and S (protein-like) contained nitrogen-rich aromatics (Stubbins et al., 2014). Liu et al. (2014) reported that peak S contains both fluorescent (humic and proteins) and non-fluorescent (polysaccharide) substances. Overall, humic-like peaks dominated in the surface water throughout the year, whereas the protein-like peaks occurred seasonally.

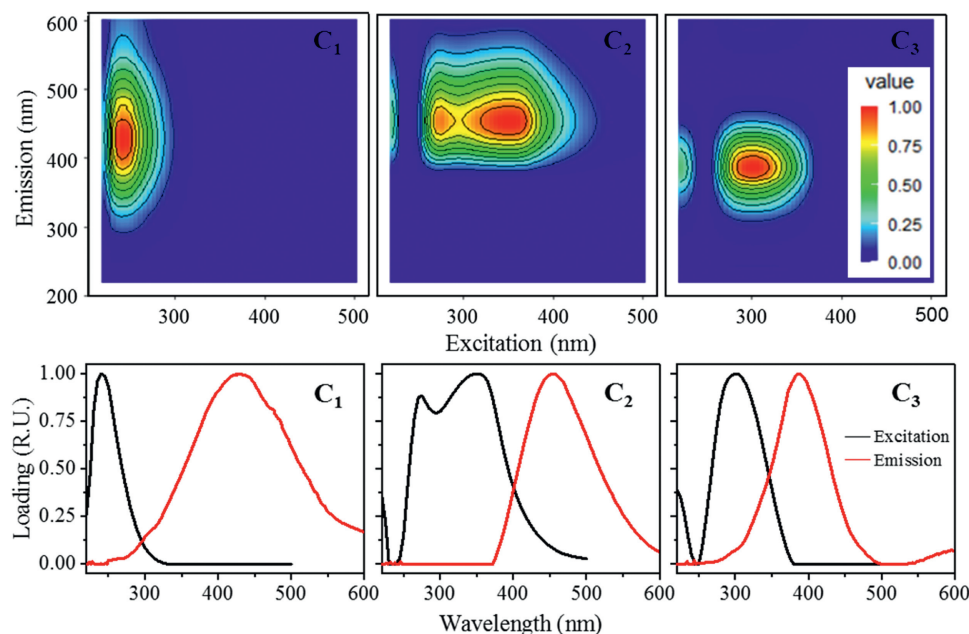


Fig. 4 – Calculated contour plots (first row) showing three humic-like components identified through EEM-PARAFAC and line plots (second row) for split-half validation.

2.3. PARAFAC modeling for fDOM

EEM-PARAFAC was applied to deconvolute the EEM datasets and three fDOM components (independent of peak A, C, T and S discussed before) were identified using PARAFAC modeling and components were validated via split-half analysis (Fig. 4).

The first (C_1), the second (C_2), and the third component (C_3) were identified as humic-like fDOM (Amaral et al., 2020; Chen et al., 2016; Lambert et al., 2017; Wauthy et al., 2018). C_1 was not detected from December to March, but occurred from April to November. The trend of C_1 occurrence (Fig. 5) was similar to the trend of UV radiations (increased in July and August and decreased in December to March; compare Appendix A Fig. S2). Maximum intensities of C_1 were detected in July and August with comparably high temperatures. Therefore, C_1 was probably produced during the exposure of DOM to UV or sunlight. Similarly, previous studies suggested C_1 as terrestrial-derived fDOM or transformed after DOM exposure to UV radiations (Amaral et al., 2020; Chen et al., 2016; Walker et al., 2009).

C_2 resembles a combination of fluorophores A and C and was named a terrestrial humic-like component (Lambert et al., 2017; Li et al., 2015). C_2 is associated with aromatic and larger (regarding molecular weight) components, and the intensities in the surface water and bank filtrate were constant. C_2 showed no degradation in the subsurface passage. There was a slight decrease in the intensities of C_2 in the surface water from March to June. The decreased concentration was presumably due to photo-degradation before the start of new build-up by phototrophic organisms (Osburn et al., 2011; Stedmon and Markager, 2005).

C_3 was classified by Murphy et al. (2008) and Chen et al. (2018) as a microbial derived humic-like component detected in freshwater bodies. Other studies showed the linkage of C_3 to the microbial degradation of algae and terrestrial humic-like compounds (Li et al., 2015; Wauthy et al., 2018).

Results of the PCA analysis (Fig. 6) of different parameters in surface water and bank filtrate showed that principle component 1 (PC_1) has a large (56.7% in surface water, 55.7% in bank filtrate) positive association with DOC, temperature, C_1 , UV_{254} , and fDOM ($C_1 + C_2 + C_3$) during the considered year. The loading for DO and SUVA in the surface water was negatively linked with PC_1 . Principle component 2 (PC_2) represented a positive linkage with C_2 and C_3 in surface water. In bank filtrate PC_2 is largely related to C_3 . Results of PCA suggested that the removals of DOM and DO in the groundwater passage correlated with the temperature. Additionally, changes in UV_{254} , C_1 , and C_2 were also affected by temperature.

Similarly, regression analyses between combined fDOM components ($C_1 + C_2 + C_3$) and DOC showed a correlation (Pearson's $r = 0.61$, $p < 0.05$) for all months. The low DOC of the surface water in winter (December to March) was mainly due to the absence of component C_1 . A correlation between DOC and temperature (Pearson's $r = 0.82$, $p < 0.05$), temperature and UV_{254} (Pearson's $r = 0.69$, $p < 0.05$) and DOC of surface water and DOC removal through BF (Pearson's $r = 0.91$, $p < 0.05$) was also observed. Thus, seasonal variations have significant impacts on DOM in the surface water and its removal through BF. This information can help to make changes and optimized the post BF treatment of surface water based on seasons and to meet the standard for portable water supplies to the com-

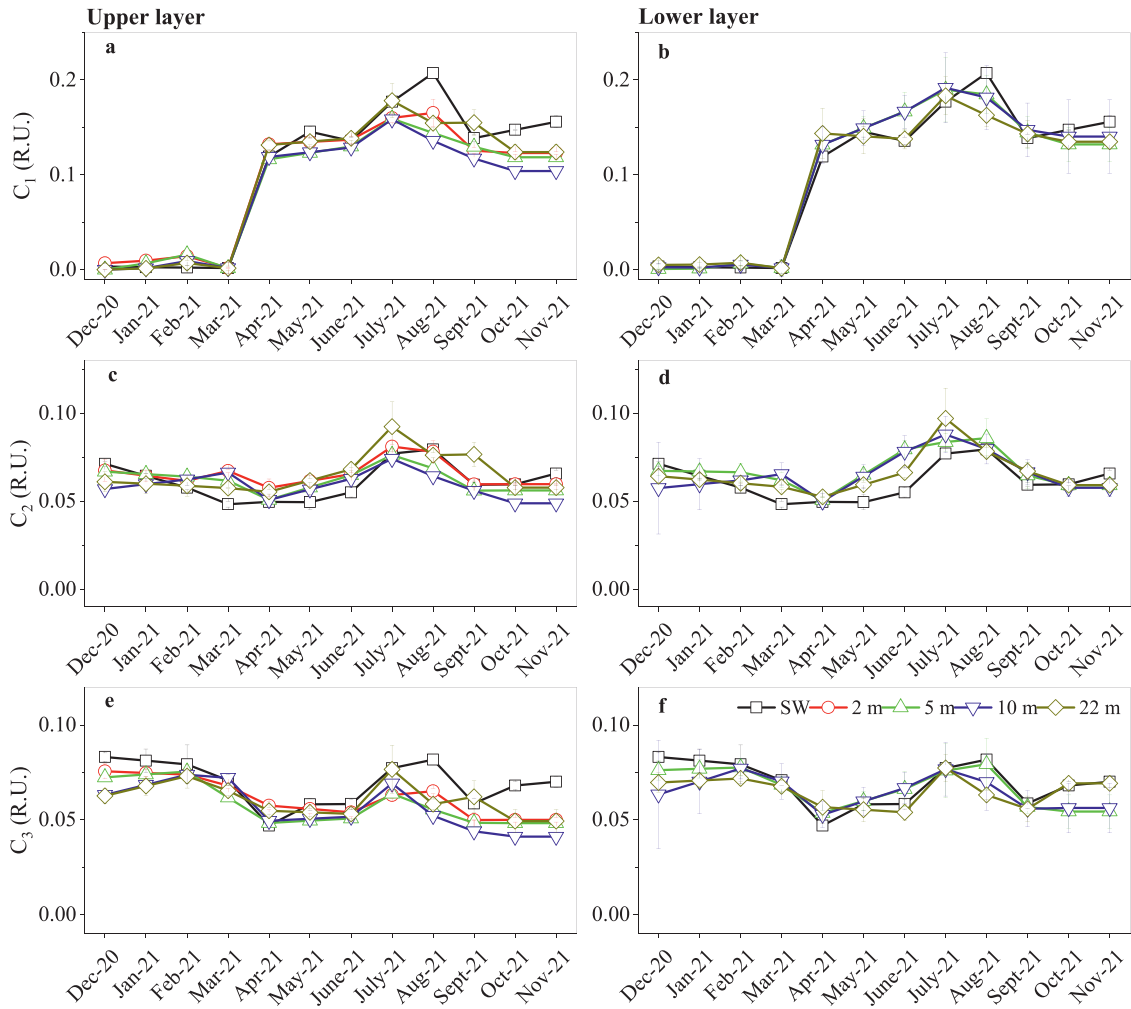


Fig. 5 – Averaged loadings values of the fDOM components C₁ ((a) and (b)), C₂ ((c) and (d)) and C₃ ((e) and (f)) over the year in the surface water (SW) and at different distances from the infiltration in the upper (fine sand) and lower layer (medium sand).

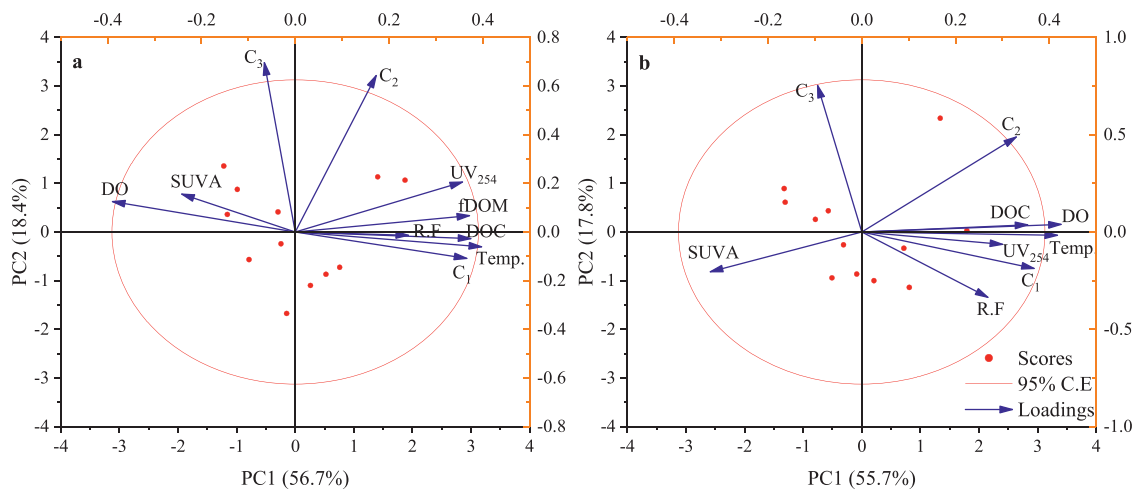


Fig. 6 – PCA (with a 95% confidence ellipse) (a) between monthly averaged values of water quality parameters in the surface water, and (b) averaged monthly removal of dissolved organic carbon (DOC), dissolved oxygen (DO), UV₂₅₄, specific UV absorbance (SUVA) and fluorescent dissolved organic matter through bank filtration. Whereas, “Temp” is averaged monthly temperature and “R.F” is the monthly average of rainfall.

munities throughout the years. As discussed in the introduction section the presence of DOM effects different pollutants removal, based on the seasonal variation in DOM and fDOM data appropriate solutions can be applied to avoid their deleterious effects on post treatment methods.

3. Conclusions

In this study, the impact of seasonal variations (such as temperature, UV radiation and rainfall intensity) on DOM composition and concentration in surface water, and removal through BF was investigated using fluorescence spectroscopy.

Three humic-like fDOM components ($C_1 + C_2 + C_3$) identified through EEM-PARAFAC were significantly affected by seasonal changes: Intensities of C_1 were low during winter and high in summer. Therefore, a direct relation between C_1 and UV radiations was observed. There was a basis humic DOM that was persistent, increased DOM concentration was presumably due to C_1 in the surface water. SUVA and fDOM suggested that the aromaticity of DOM in the surface water was slightly high (15%) in winter compared to summer.

The reduction of DOM through BF was within few meters and no removal was observed during subsurface flow. DOM removal was correlated with the reduction of C_1 and C_3 , whilst C_2 was constant and considered recycled DOM. DO in the bank filtrate was negatively correlated with temperature and DOC. During the summer, probably increased biological activity with oxidative biotransformation of POC accumulated at the infiltration zone caused the increased DO utilization from month April to month October. DOM produced in the summer by algal blooms and phototrophic activity were fully degradable and removed by BF. In a recirculated BF system DOM residual equal to the non-degradable fraction of DOM represents a constant background concentration and was not affected by temperature, UV radiations and redox conditions.

Declaration of Competing Interest

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

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Appendix A Supplementary data

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jes.2023.01.006.

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