

Article

The Composition of Dissolved Organic Matter in Arable Lands: Does Soil Management Practice Matter?

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Abstract: Dissolved organic matter (DOM) is a key soil quality property, indicative of the organic matter stored in the soil, which may also be a function of temporal variation. This study examines whether DOM is a robust property of the soil, controlling fertility, or if it may change with time. Altogether eight sets of soil samples were collected in 2018 and 2019 from the cultivated topsoil (0–10 cm) of cropland and from a nearby grassland near Martonvásár, Hungary. The study sites were characterized by Chernozem soil and were part of a long-term experimental project comparing the effects of manure application and fertilization to the control under maize and wheat monocultures. DOM was extracted from the samples with distilled water. The dissolved organic carbon (DOC), total dissolved nitrogen (DN), biological index (BIX), fluorescence index (FI), humification index (HIX), carbon nitrogen (C/N) ratio and specific ultraviolet absorbance at 254 nm (SUVA₂₅₄) index were studied in the arable soils, and the results showed that all the DOM samples were humified, suggesting relevant microbiological contributions to the decomposition of OM and its conversion into more complex molecules (FI = 1.2–1.5, BIX = ~0.5, and HIX = ~0.9). Temporal variations were detected only for the permanent grassland where higher DOM concentration was found in spring. This increased DOM content mainly originated from humified, solid phase associated, recalcitrant OM. In contrast, there were no differences among fertilization treatments and sampling dates under cropfield conditions. Moreover, climatic conditions were not proven as a general ruler of DOM properties. Therefore, momentary DOM alone is not necessarily the direct property of soil organic matter under cropfield conditions. The application of this measure needs further details of sampling conditions to achieve adequate comparability.

Keywords: dissolved organic carbon (DOC); total dissolved nitrogen (DN); DOM composition; SUVA₂₅₄; fertilization; C/N ratio; land use effects; Chernozem

1. Introduction

The soil system creates the foundation for ecosystem functioning and includes the most significant carbon pool. Dissolved soil organic matter (DOM) is only <4% of the soil organic matter (SOM) [1] and includes dissolved organic nitrogen (DON) and dissolved organic carbon (DOC) and is characterized as particles <0.45 µm in solution [2,3]. DOM occurs when soil pore water extracts the soluble components of SOM and becomes stable with drying out of the soil. Under wet conditions, therefore, it is mobile, reactive, and

available by diffusion and convection to all compartments of the soil environment, making it the most important labile fraction of SOM and an effective indicator of changes in soils.

DOM contains a wide variety of organic molecules, from easily decomposable sugars to the relatively stable phenolic lignin. The former, highly available molecules are generally related to the living organic part of the soil such as the microbiome, whereas the latter group reflects the humified and recalcitrant components with a higher residence time. The dominance of recalcitrant components indicates humus-originated, terrestrial DOM, and the higher ratio of sugars and amino acids reflects increased microbial activity [4]. DOM is often described as a direct function of SOM and, in this way, a useful soil quality indicator [5,6]. Nonetheless, the quantity and composition of DOM may also indicate the physiological activity of the soil (micro) biome. In this way, DOM is an ever-changing material reflecting the temporary environmental (temperature, moisture content, vegetation, etc.) conditions of the soil.

A stable ecosystem such as a native forest or pasture under natural conditions generally results in a well defined and specific SOM content and composition, which also reflects in the DOM properties. In contrast, if the soil is cultivated and therefore loses a considerable part of its SOM, the environmental conditions (e.g., nutrient and moisture content, plant cover and composition) will be extremely variable, causing frequent changes in DOM.

Previous studies focused on the role of either sampling dates or land-use effects in forest soils on DOM properties. For example, seasonal trends in DOC were found in forest floor leachates [7], while seasonal variation was shown to have a significant impact on the DOC concentration and composition in the forest floor [8]. Similarly, DOM was studied in various soil systems such as the gley soils of a moorland [9], soil samples taken after cattle manure addition [10], and soils with a sandy loam texture [11]. Embacher et al. [12] reported that DOM exhibited seasonal fluctuations in several soil types, specifically Cambisol, Luvisols, and Chernozem. However, limited information is available on how the DOM properties in arable soils respond to the combined effects of sampling dates and agrotechnical effects. It is also important to study the comparability of DOM concentrations in the same soil on grassland and cropland. In the case of different land-use practices and crop covers, the best management practice to preserve soil quality by increasing soil DOM has still not been satisfactorily determined, as limited information is available on the dynamics of DOM in arable soils [12,13]. Furthermore, it has been recommended that the factors controlling the response of DOM dynamics to changes in soil conditions (C contents) should be studied [6,14].

Former cropfield related works investigated the effect of the tillage system on DOM distribution across soil depth [15,16] or on that of sampling times on the soil DOM in forest and peaty soils [8,9,17]. It was suggested that land use, soil erosion on agricultural soils, and temporal variations should be considered in predicting the chemical composition of fluvial DOM [18,19].

On this basis, the present research hypothesized that the combined effects of seasonal variation, land use, and nutrient management affect both the quantitative and qualitative parameters of DOM. Furthermore, we aimed to answer two main questions: 1. What is the importance of the sampling date in DOM properties? 2. Did the sampling date or land nutrient management have a more significant effect on DOM?

In line with these aims, the specific objectives were to (a) study the effects of sampling dates and weather conditions; (b) study the effects of land use on DOM (grassland vs. cropland); and (c) study the effects of nutrient management on DOM.

2. Materials and Methods

2.1. Site Description

Soil samples were collected from arable lands (cropland) and a nearby semi-native grassland in the vicinity of Martonvásár (47.331196 N, 18.789660 E), Hungary, which were part of a long-term experiment, established in 1958, involving the same treatment each year (Figure 1). Mean annual temperature and precipitation values of 10.6 °C and 539 mm were

recorded between 1958 and 2018 [20]. The distance between the two sites is approximately 80 m. The permanent grassland used as a site control (intact soils with no fertilizers) was compared with cropland, on which the crops were given three treatments: NPK fertilizer alone ($110 \text{ kg ha}^{-1} \text{ yr}^{-1}$ nitrogen, $45 \text{ kg ha}^{-1} \text{ yr}^{-1}$ phosphorus and $50 \text{ kg ha}^{-1} \text{ yr}^{-1}$ potassium), NPK combined with $8 \text{ t ha}^{-1} \text{ yr}^{-1}$ farmyard manure (as organic fertilizer) and an unfertilized control.



Figure 1. Location of the long-term field trial in Martonvásár, Hungary (Google Earth Pro).

The soil of the study site was a Chernozem [21], the main parameters of which are summarized in Table 1. The two-factorial split-plot crop rotation had four replications, with maize and wheat, in the main plots (245 m^2) and the above-mentioned fertilizer treatments, applied in a random design, in the subplots (49 m^2).

Table 1. Main parameters of the studied soils [22]. Values are means \pm SE. The profiles included A, and C horizons. SOC: soil organic carbon; TN: total bound nitrogen; clay: $<5 \mu\text{m}$ fraction; silt: $5 \mu\text{m}$ – $50 \mu\text{m}$ fraction; sand: $50 \mu\text{m}$ – $2000 \mu\text{m}$; ND: not determined; * measurements were made in the top 10 cm.

Horizon	Maize and Wheat Monocultures		Grassland	
	A	C	A	C
Depth (cm)	0–40	60–	0–30	60–
SOC (g kg^{-1})	10.9 ± 0.2 *	0.5 ± 0.1	26.1 ± 0.6 *	0.5 ± 0.1
C:N	18.8 ± 4.0 *	ND	20.7 ± 1.6 *	ND
pH(KCl)	7.7 ± 0.2 *	7.9 ± 0.0	7.1 ± 0.2 *	7.6 ± 0.0
Clay (% <i>v/v</i>)	24.9 ± 3.3 *	24.5 ± 0.7	20.7 ± 0.6 *	18.8 ± 0.3
Silt (% <i>v/v</i>)	45.6 ± 1.9 *	44.9 ± 0.9	56.3 ± 3.6 *	30.7 ± 3.1
Sand (% <i>v/v</i>)	29.4 ± 2.3 *	30.6 ± 1.5	22.9 ± 2.1 *	50.5 ± 3.3

2.2. Experimental Design

The experimental design involved four blocks, with different treatments and crops (Section 2.1). The present study focused on plots containing maize and wheat (Figure 1). A total of twelve soil samples, with three replicates per block, were taken for each treatment on the cropland site, with similar soil samples on the grassland site. Samples were collected on the cropland from a depth of 5–11 cm at various dates in 2018 and 2019 (Table 2). Soil samples from the grassland were collected on the same dates, excluding March 2018 and May 2018. The samples were placed in plastic bags and air-dried directly after collection. Plant materials and debris were removed from the soil. The date of fertilizer application is also given in Table 2.

Table 2. Date and quantity (kg ha^{-1}) of fertilizer application prior to the samplings on the cropland site and dates of sample collection. A: no fertilization, B: NPK+manure, C: NPK. NPK: Nitrogen, Phosphorus, Potassium. Manure is applied in each 4th year.

Application Date	Treatment	Maize			Wheat			Sampling
		N	P ₂ O ₅	K ₂ O	N	P ₂ O ₅	K ₂ O	Date
3 April 2018.	A	-	-	-	-	-	-	29 March 2018.
	B	-	-	-	50	-	-	23 May 2018.
	C	-	-	-	56	-	-	26 June 2018.
28 March 2019.	A	-	-	-	-	-	-	23 April 2019.
	B	25	-	-	25	-	-	4 June 2019.
	C	25	-	-	25	-	-	8 July 2019.
11 October 2019.	A	-	-	-	-	-	-	9 September 2019.
	B	100	45	50	50	45	50	14 October 2019.
	C	112	45	50	56	45	50	

2.3. Soil Organic Matter Extraction

The land use effect, including OM addition, may limit the possibility of using certain tools (e.g., a suction cup) to obtain soil solution directly from the field. However, such extraction methods could be a viable alternative, as there is no constant procedure for extraction [23]. Therefore, a modified water extraction method was used [24,25]. Briefly, 4 g of soil was suspended in 40 mL distilled water and shaken for 2 h. Then the suspensions were centrifuged at 4800 rpm for 15 min, followed by filtration through 0.45 μm glass fiber. The filtrates were then stored at 4 °C until analysis. DOC and total DN concentrations were measured using a TOC/TN analyzer (Shimadzu, Kyoto, Japan).

2.4. Spectroscopic Measurement of DOM Composition

Heterogeneity, complexity, and variations in the DOM concentration may all be factors in controlling DOM composition. Specific UV absorbance (SUVA) is an effective measure for predicting the chemical features of DOM, such as aromatic carbon content. SUVA₂₅₄ is the UV absorbance at 254 nm normalized for DOC concentration. An increase in SUVA₂₅₄ values indicates an increase in DOM aromaticity [26]. Aromaticity was determined using a UV2600i UV-ViS spectrophotometer (Shimadzu, Kyoto, Japan) and a TOC/TN analyzer (Shimadzu TOC-L/TN). Fluorescence intensity ratios (or fluorescence indices) were used to estimate the proportion of humified and microbial sources of DOM [27]. Three kinds of ratios were used: the biological index (BIX), which is based on the presence of fluorophore peptides and amino acids [28]; the fluorescence index (FI), based on that of high-molecular-weight humic acids (HA) with low aromatic content [29]; and the humification index (HIX), based on that of high-molecular-weight HA with high aromatic content [30]. Fluorescence spectra were measured using an RF-6000 spectrofluorophotometer (Shimadzu, Kyoto, Japan).

2.5. Soil Water Content, Air Temperature, and Precipitation Monitoring

In situ soil water content (SWC) was monitored at soil depths of 0–7 cm with TEROS-10 sensors and Em-50 data loggers (METER Group, Pullman, WA, USA). Air temperature and rainfall data were obtained from a nearby meteorological station. Each weather factor (mean air temperature, rainfall sum and average SWC) recorded during the 14-day period preceding sampling was treated as a separate environmental variable.

2.6. Statistical Analysis

The data were presented as the mean \pm standard error (SE) of twelve replicates for each fertilizer treatment, crop cover and sampling date, giving a total of $n = 648$. The Mann-Whitney U test was used to study fluctuations in DOM parameters across sampling dates [31]. As the data were not normally distributed, outliers were identified and removed from the data within a range of 3 to -3 based on the Z score (Equation (1)).

$$Z_i = \frac{X_i - \mu}{\sigma} \quad (1)$$

where X_i was the value measured for variable i , μ was the mean, and σ the standard deviation. This method was previously used on similar study areas [32,33]. The Z score for each variable was then used to calculate the principal component (PC). Principle component analysis (PCA) was used to reduce the parameter dimensions based on scree plots [34,35]. PC scores were classified on the basis of a loading factor, for further interpretation, only values >0.6 were considered (Table 2).

The validity of selected variables in PCA was examined using the Kaiser-Meyer-Olkin (KMO) and Bartlett tests. Neither HIX nor the C/N ratio were included in PCA because they were correlated with other variables (e.g., the C/N ratio was negatively correlated with DN), thus lowering the KMO value (<0.5) and reducing the validity of PCA [36]. SPSS statistical software (version 28, IBM Corp., Armonk, NY, USA) was used for PCA. Each classified component was correlated with environmental factors (rainfall, soil water content, and air temperature) using Spearman's correlation coefficient. When significant correlation coefficients (ρ -value ≥ 0.5) were obtained, further regression analysis was performed to determine the most influential factors that explained data variability across sampling dates. For this purpose, a correlation plot was compiled using the R statistical software (R Core Team, Vienna, Austria) package (version 4.1).

3. Results and Discussion

3.1. DOC Contents and DOM Composition across Sampling Dates

DOC concentration and total DN fluctuated seasonally (Figures 2 and S1) without any clear temporal trend. Previous studies also found that a slightly higher DOC concentration between May and November showed the importance of climatic factors under low rainfall-high-temperature conditions [37], whereas Wickland et al. [38] found no seasonal variations in the biodegradability and chemical properties of DOC in soil pore water in boreal regions, except that poorly drained sites had the lowest biodegradable DOC.

Soil properties vary spatially and temporally, depending on local soil conditions, thus affecting DOM contents. In incubation experiments in the boreal areas, temperature controlled DOC production [17,39], while soil moisture and air drying controlled soil DOM extraction [40]. The hydraulic conductivity and bulk density in the surface and subsurface (15 cm depth) soil were found to exhibit temporal variability [41], but in soil with a high infiltration rate, no soil moisture levels were found to affect DOM leaching or runoff [42]. These findings proved the importance of spatial variations, procedures, and soil conditions in DOM studies. This also demonstrated the varied effects of environmental conditions on DOM. Weather factors may increase biomass production, providing more food for soil biota; rainfall, for instance, may ensure optimum soil moisture, which will affect the SOM quantity [43]. If a dataset including microbial activity was available, the temporal changes

observed in this study might be clearer, and the soil moisture content at sampling time might be found to trigger the microbial contribution to DOM.

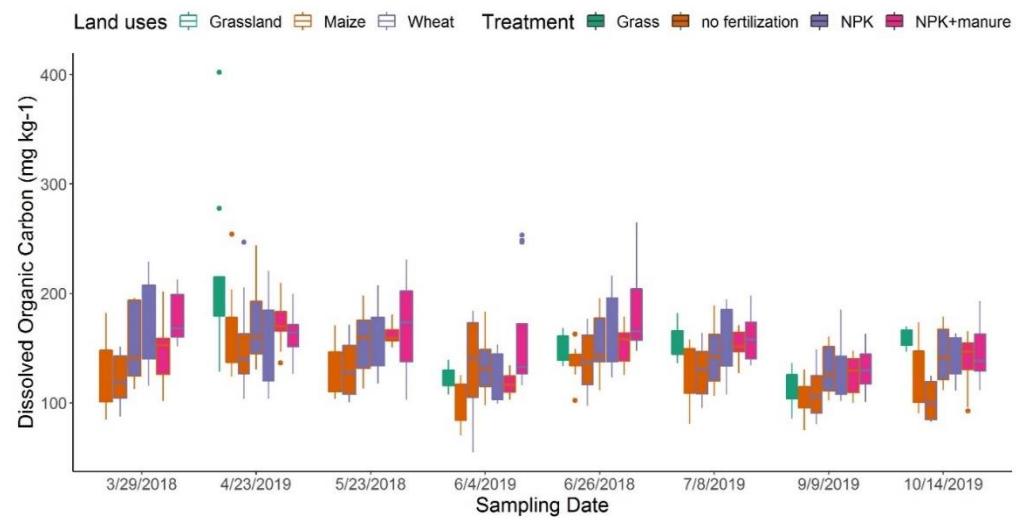


Figure 2. Dissolved organic carbon (DOC) concentrations in cropland and grassland plots under different treatments and across crop cover. Total sample size was $n = 648$, with $n = 12$ for each treatment and crop cover. Dots indicate outliers. NPK: nitrogen, phosphorus, potassium.

There were two indirect proofs of the importance of microbial activity, one of which was the C/N ratio, which has been reported to vary between 5 and 8 in the case of microbial biomass [44], between 12 and 30 for terrestrial humus [45,46] and between 20 and 200 for plant litter and plant tissues [46]. The C/N in the present work varied from 1 to 13. The lowest (<5) C/N ratios were found after fertilization, indicating the influence of N fertilizers on the cropland. Therefore C/N was only interpreted for the grassland, where it was higher in springtime indicating humus-related components, but lower, reflecting to microbial biomass during the growing season (Figure 3). The C/N ratio in April significantly differed from that detected at other sampling dates (Mann-Whitney, Asymp. Sig. (2-tailed) < 0.05 , supplementary data). The highest value was observed in April (10.4 ± 0.84) and the lowest in July (5.3 ± 0.79). Although the soil C/N ratio was previously reported to be approximately 10–12 [47], lower values were found during the summer and fall seasons, showing the role of temperature and soil moisture in affecting microbiota and controlling OM degradation [48]. The present results confirm findings obtained on different soils, indicating that the C/N ratio was a significant indicator of SOM contents and losses from the soil system [49–51].

The other indirect proof of the importance of microbial activity was BIX, which is based on detecting the presence of fluorophore amino acids and the peptides which contain them. The BIX values varied over a narrow range (0.42–0.60). On the grassland plots, lower values (representing complex, transformed organic matter) were characteristically recorded in spring, with higher values during the growing season. In accordance with the C/N ratio results, the BIX index in April was significantly lower than that at other sampling dates (Mann-Whitney, Asymp. Sig. (2-tailed) < 0.05 , supplementary data) on the grassland. Similar temporal dynamics were also observed for the cropland plots, but the differences were not significant (Figure 4).

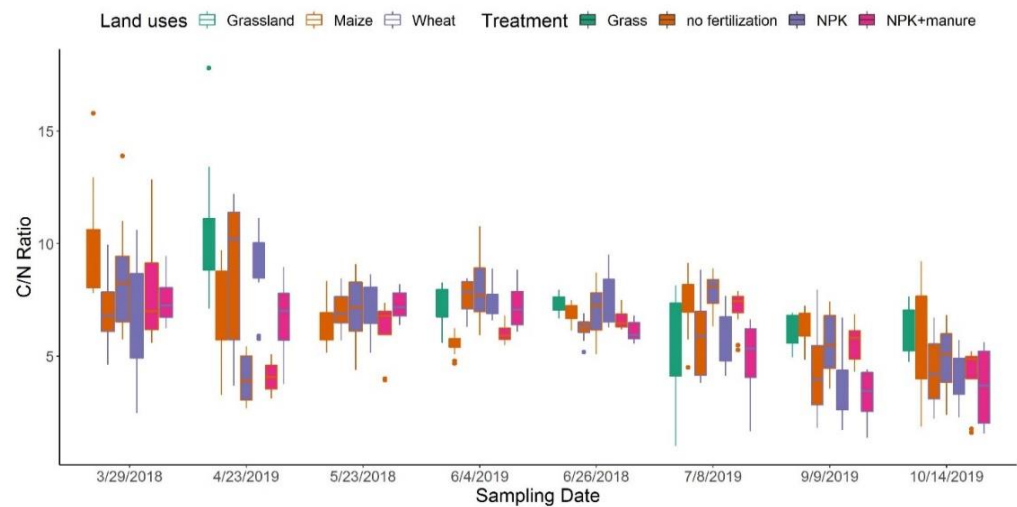


Figure 3. Stoichiometric carbon to nitrogen (C/N) ratio in cropland and grassland plots under different treatments and across crop cover. Sample size was $n = 12$, total sample size was $n = 648$; dots indicate outliers. NPK: nitrogen, phosphorus, potassium fertilization.

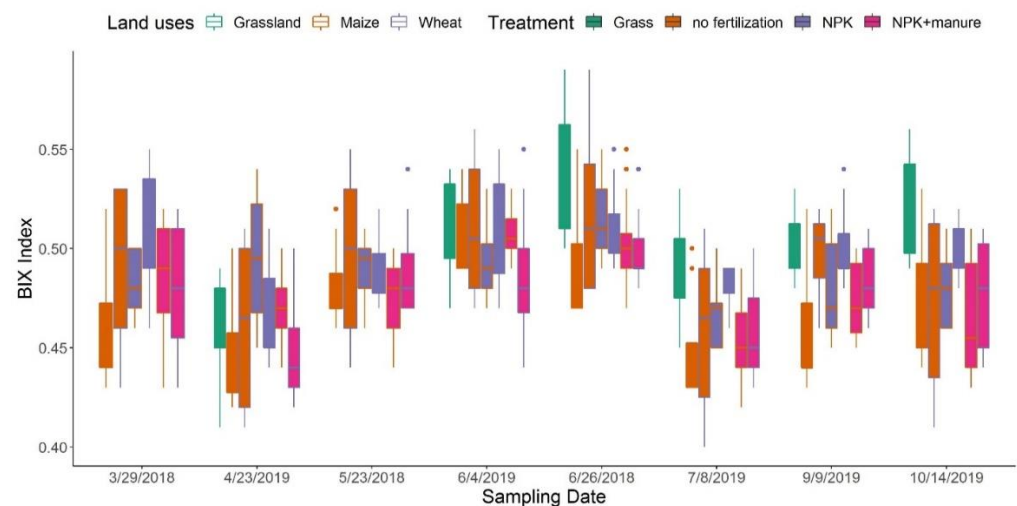


Figure 4. Biological index (BIX) values on cropland and grassland sites under different treatments and across crop cover. Sample size was $n = 12$ for each treatment and crop cover, total sample size was $n = 648$; dots indicate outliers. NPK: nitrogen, phosphorus, potassium fertilization.

The presence of humified compounds in DOM was revealed by the $SUVA_{254}$, HIX and FI values. The $SUVA_{254}$ values ranged from <1 to 8 (Figure S2), which was close to previous findings [52]. Both $SUVA_{254}$ and FI values varied with sampling time but without trends. Minimal variance among the treatments was recorded for some sampling dates and high variability for others (Figures S2 and S3). The HIX values were close to 0.9 (Figure S4), indicating a high degree of humification and lower biological activity [53]. On the other hand, none of the treatments showed seasonal variations based on the HIX values. In April, however, all samples from all treatments had values >0.9 , suggesting possible seasonal effects on DOM composition. The highest hydrophobic aromatic composition on the organic forest floor was observed in summer, while hydrophilic content was noted in winter and spring [7,8]. The complexity of riverine DOM was reduced by a period of soil dryness [19]. Lower FI and BIX indices with a higher HIX value may suggest the effect of weather factors on microbiome conditions, which may regulate microbiological activity and make the DOM content more humified.

3.2. Effects of Land Use and Fertilization on DOM

The results showed no differences in DOM content between sampling dates, treatments, and crop covers. In contrast to the original hypothesis, the DOM content was neither a sensitive indicator for land use, including fertilizer and crop cover, nor for the sampling time. Lower FI values were detected on grassland, with values around 1.2 to 1.5 for all soil samples. As a general trend, HIX varied between sites rather than over time. Values higher than 0.9 were found for the untreated cropland and grassland than for fertilized soil (0.8). Consequently, BIX and FI indices were found to explain most of the data variation in the first component (Table 3), with a total variance of 39.3%. The BIX and FI indices acted in the same direction, indicating their role in explaining changes in microbiological activity as a whole [28]. PC2 contained both DOC and DN, with lower variance (24.6%). The second component described how the DOM concentration (DOC or N content) changed as a function of microbiological activity. Accordingly, the results showed a total explained variance of 63.9% for the two components. Neither PC revealed any apparent effect of land use on the DOM contents, suggesting that the soil microbiome and microbial activity might be more relevant in studying DOM contents over time.

Table 3. Rotated component matrix showing the structure loading matrix correlation between the studied variables and the principal components (PCs).

Variables	Principal Components	
	PC1	PC2
Dissolved nitrogen (DN)	0.038	0.744
Dissolved organic carbon (DOC)	−0.530	0.658
Specific ultraviolet absorbance at 254 nm (SUVA ₂₅₄)	−0.396	−0.499
Biological index (BIX)	0.894	0.023
Fluorescent index (FI)	0.848	0.038

Extraction Method: Principal Component Analysis. Rotation Method: Varimax with Kaiser Normalization.

As a rough trend, there was no differentiation between sampling dates or land uses, suggesting that the microbiological activity was influenced to a greater extent by environmental (meteorological) variables than by the treatments, crops, or sampling dates (Figures 5, S5 and S6). These findings should not be generalized to suggest that DOM was an unresponsive index for any soil management practice. Instead, they highlighted the fact that soil management practices in conjunction with soil microbiome conditions might reduce the effects of OM addition on the DOM content at certain study sites.

Soil management practices on cropland may decrease the effect of OM addition. Compared with the grassland site, soil samples from the cropland were collected from a tilled layer, which may have accelerated organic matter decomposition, offsetting any fertilizer effects between the study sites. While fertilizer inputs should be a sustainable practice [54] to mitigate agricultural soil degradation and climate change, priming effects could increase microbial activity and consequently soil respiration after OM addition [55]. This may explain why no differences in DOM content were detected between the study sites, although fertilizers were added on the cropland, which was expected to increase the DOM content. Another factor was the tillage effect, which may have caused a tradeoff between OM addition and soil structure to enhance crop growth. Previous studies indicated that tillage systems influenced DOM but did not reduce the yield quantity [56,57]. As soil management practices affect organic matter quality by affecting SOM content and aggregate stability, which control the activity of soil organisms [58], decomposition processes may dramatically change, affecting DOM properties [13]. This also explained why the conversion of 5% of grassland to cropland leads to a C loss of 300 Mt CO₂ eq. over a 50-year timescale, offsetting any alternate management practices such as fertilization [54].

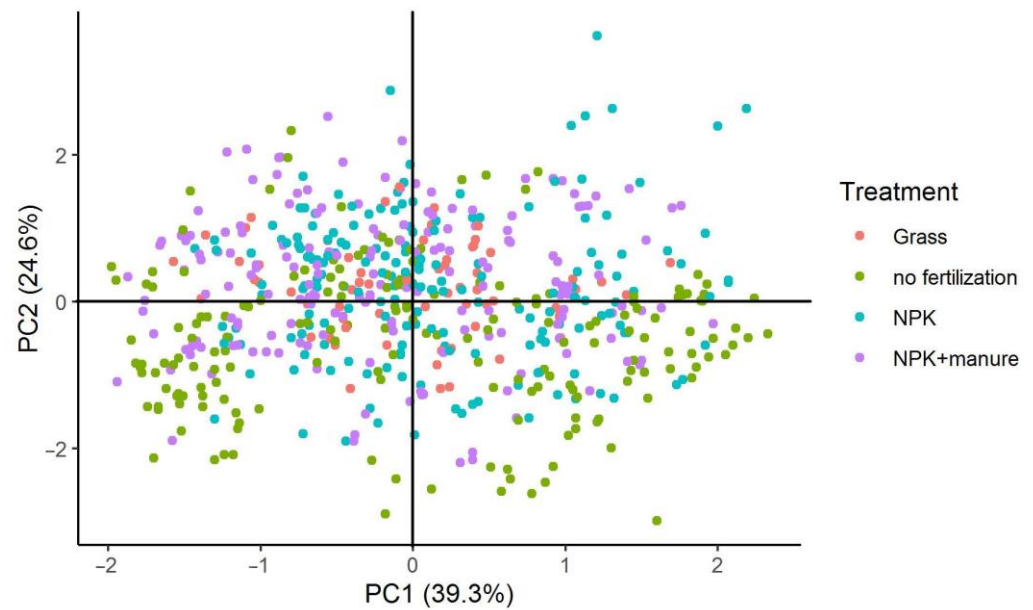


Figure 5. Principle component analysis (PCA) scores for components (PC) 1 and 2. The explained variance is in brackets. Colors highlight the land-use and nutrient-management effects. NPK: nitrogen, phosphorus, potassium fertilization.

3.3. Effect of Weather Parameters on DOM

There was a weak but significant positive association between PC1 and both the rainfall sum and average SWC during the 14 days before sampling (Spearman's correlation coefficients p -value < 0.05, Figure 6). At the same time, PC2 was negatively associated with these two weather factors. Neither PC had any significant association with the air temperature. The highest correlation coefficient, $\rho = 0.4$, was found between PC1 and the rainfall amount in the preceding 14 days. Due to this weak correlation between the PC results and weather factors, no further regression analysis was performed on these data. The lack of significant correlation could be due to the nature of the soil as a chaotic and heterogeneous system, thus stressing the importance of considering microbiome conditions and regularly including microbial data as best practices when studying seasonal changes in the DOM content.

When the results showed no apparent seasonal effect or land-use effect (e.g., OM addition) on DOM variations, the OM addition may have been adsorbed onto a mineral phase or mineralized by the microbiome, thus limiting the effect of crops or OM addition on DOM. These results are in partial agreement with those of Angers et al. [10], who found that temporal variations had a more decisive effect on soil DOM than manure effects. Based on the material applied, amendments could increase the molecular weight of DOM and its sorption onto soil solids, possibly contributing to organic C pools, as stated previously by Ohno et al. [59]. However, the present results contradicted those of previous studies on the effect of the weather on DOM variation at sites ranging from forest floor leachates [7] to peaty gley soils [9]. Differences in DOC concentrations were found to be caused by site and climatic factors (rainfall) [37]. An increase in DOC concentration was also found on a forest floor after a summer rainstorm following a dry period [8]. DOC production in a Gelisol in Alaska increased by 20–135% under warmer conditions than at a colder temperature [4]. Embacher et al. [12] found seasonal differences in the DOM concentration when studying different soil groups with a spatial variation between sites (>600 km). Therefore, microbial activity may cause variations in DOM irrespective of the origin of the soil samples (sites), the crop cover and the land use (treatments).

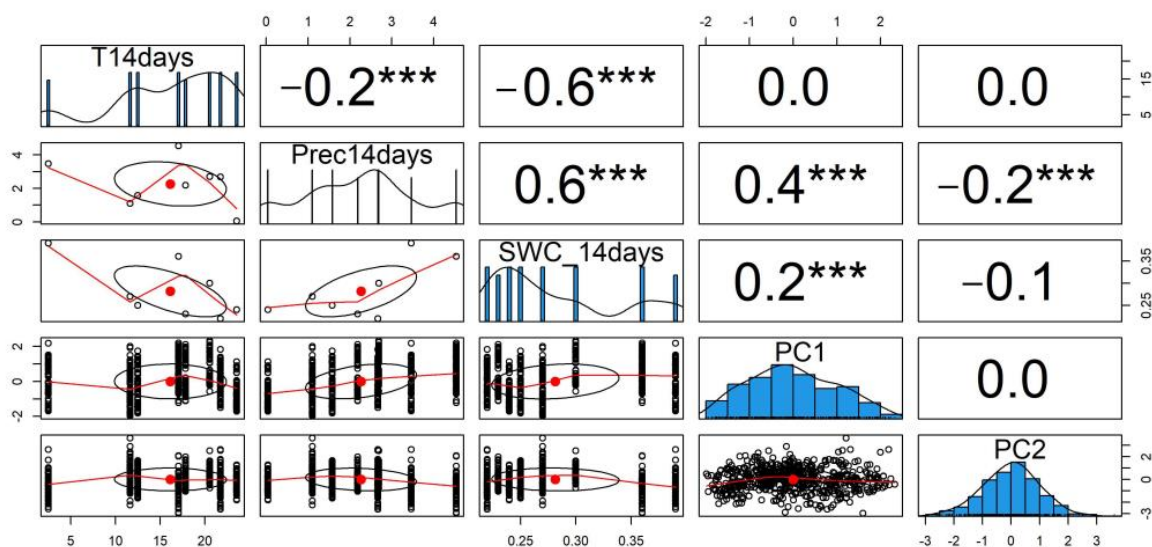


Figure 6. Correlation plot showing the data frame of correlations between the studied variables, classified within each component (PC1 and PC2). Significance was calculated on the basis of Spearman's correlation coefficients (ρ) (***) significant at the 1% level). Weather factors: T14days: mean air temperature 14 days before each sampling date, SWC_14days: average soil water content 14 days before each sampling date, Prec14days: rainfall sum 14 days before each sampling date.

4. Conclusions

During a period of nearly two years, some DOM parameters showed fluctuations, whereas others did not reveal a temporal pattern. Temporal variations were detected only for the permanent grassland with higher DOM concentration in spring. This increased DOM content mainly originated from the humified, solid phase associated, recalcitrant OM. This suggests the relevant role of sampling time under quasi natural ecosystems. In contrast, there were no differences among fertilization treatments and sampling dates under cropland conditions. Cultivation practices may trigger a spatially and temporally heterogeneous complex system, which might be sensitive to even small environmental changes, as indicated by the high variance of most of the investigated DOM related parameters. For the same reason, climatic conditions were not proven as a general ruler of DOM properties. Therefore, momentary DOM alone is not necessarily the direct property of soil organic matter on croplands. The application of this measure needs further details of sampling conditions to achieve adequate comparability.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/agronomy12112797/s1>, Figure S1: The total dissolved nitrogen (DN) concentrations in cropland and grassland plots under different treatments and across crop cover. The total sample size ($n = 648$) and dots indicate outliers; Figure S2: The specific absorbance ($SUVA_{254}$) in cropland and grassland plots under different treatments and across crop cover. The total sample size ($n = 648$) and dots indicate outliers; Figure S3: The fluorescence index (FI) in cropland and grassland plots under different treatments and across crop cover. The total sample size ($n = 648$) and dots indicate outliers. Each treatment and crop cover had a sample size ($n = 12$), except that it had ($n = 11$) under wheat cover for NPK and NPK+manure treatments in March 2018 and NPK+manure under Maize cover in May 2018.; Figure S4: The humification index (HIX) in cropland and grassland sites was under different treatments and across crop cover. The total sample size ($n = 648$) and dots indicate outliers. Each treatment and crop cover had a sample size ($n = 12$), except that it had ($n = 11$) under wheat cover for NPK and NPK+manure treatments in March 2018 and NPK+manure under Maize cover in May 2018; Figure S5: Principle component analysis (PCA) scores for component 1 and 2. Colors highlighted the sampling date effects; Figure S6: Principle component analysis (PCA) scores for component 1 and 2. Colors highlighted the land-uses (Crop cover) effects; and Detailed Statistical Data.

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