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Variations in the quantity and chemical composition of soil dissolved organic matter along a chronosequence of wolfberry plantations in an arid area of Northwest China

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Abstract

Background Dissolved organic matter (DOM) is the most active component of soil organic matter (SOM), playing a major role in regulating soil fertility and carbon cycling. However, the effects of different wolfberry (*Lycium barbarum* L.) planting ages on the chemical diversity of DOM and its interaction with soil physicochemical properties have not been comprehensively studied. In this context, we collected soil samples (0–10 cm) from wolfberry orchards at different planting ages (1, 4, 6, 10, and 13 years) and from a corn field (0 years) in the arid region of Northwest Ningxia in China to assess the changes in soil DOM quantity and quality using ultraviolet–visible absorbance, fluorescence spectroscopy, and parallel factor analysis.

Results We found that the ages of the wolfberry plantation changed the contents of soil nutrients and SOM. In addition, significantly higher DOM concentrations were observed at wolfberry planting ages of 10 and 13 years than those in the control group (0 years) by 176.6 and 190.2%, respectively. The specific ultraviolet absorbance at 254 nm (SUVA_{254}) and 254 nm to 365 nm ultraviolet absorbance ratio (E2/E3) values were decreased and increased, respectively, after wolfberry planting, indicating low aromatic and molecular weight compounds of soil DOM. The biogenic index (BIX) and fluorescence index (FI) of soil DOM ranged from 0.6 to 0.7 and 1.42 to 1.93, respectively, suggesting a combination of allochthonous and autochthonous sources. The short- and long-term wolfberry cultivations of 1 and 4 years decreased and increased the humification degrees of soil DOM, respectively. The contribution rate of the protein-like (C1) fluorescence intensity decreased, while that of the fulvic acid-like component (C3) increased with increasing wolfberry planting age, suggesting a change in the structure of soil DOM from protein-like to fulvic acids. In this study, total nitrogen (TN) and exchangeable Ca^{2+} were the main factors affecting the quantity and quality of soil DOM in the wolfberry orchards with different planting ages.

Conclusions This study demonstrated that long-term wolfberry plantation enhances the accumulation of soil DOM and more complex compounds, thereby promoting soil organic carbon sequestration under different planting ages and land-use types in terrestrial ecosystems.

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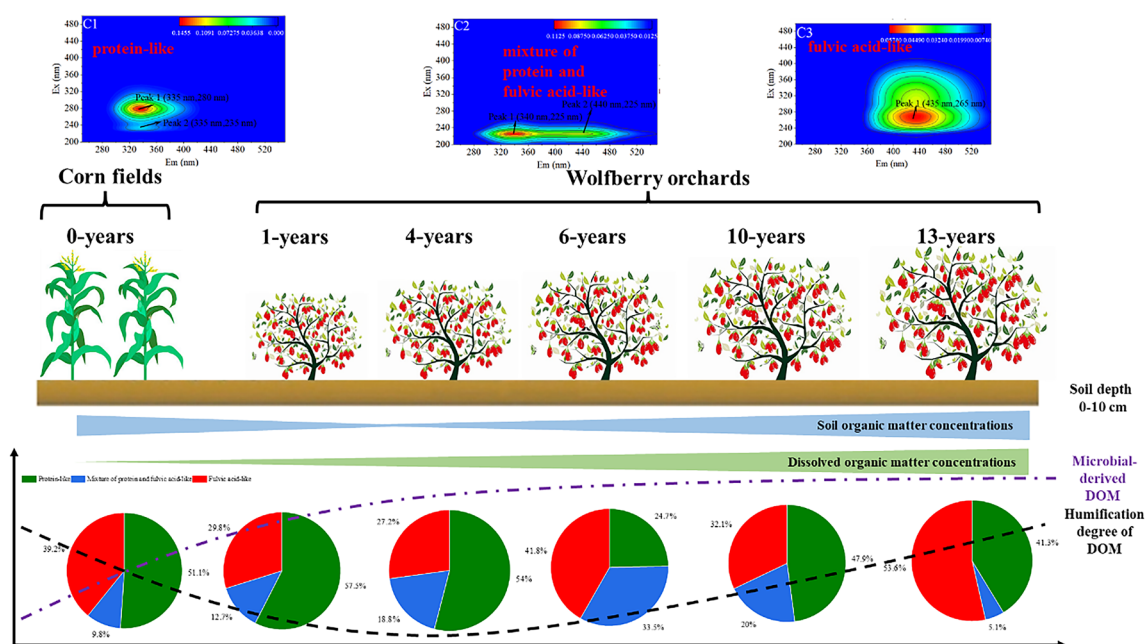
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Highlights

- Plantation age of wolfberry influenced the quantity and chemical composition of soil DOM.
- Long-term wolfberry plantation (more than 10 years) significantly enhanced the DOM content.
- The conversion of farmland to wolfberry forest decreased the aromaticity and molecular weight of soil DOM.
- The fulvic acid-like substances become the main fluorescent components of soil DOM over time since wolfberry plantations.
- TN and exchangeable Ca^{2+} were the main factors affecting the quality of wolfberry soil DOM.

Keywords Aridisols, Wolfberry, Planting age, Dissolved organic matter, Parallel factor analysis, Three-dimensional fluorescence spectrum

Graphical Abstract



Background

Dissolved organic matter (DOM) in soil environments is composed of soil organic matter (SOM) molecules [1] with relatively strong biological activity and high bioavailability. In fact, DOM plays a key role in the global carbon cycle and biogeochemical functions of ecosystems [2–5], including soil formation, mineral weathering, and pollutant transport [6]. Soil DOM is mainly derived from plant-based substances, soil humus, root exudates, and microbial decomposition of SOM [6–8].

The stability and chemical composition of DOM in soils are influenced by several factors, such as biological, abiotic, and anthropogenic factors [9]. These factors include soil physicochemical properties (e.g., pH, nutrients, and C/N) [1, 10–12], land use patterns, ecosystem

types and years of planting. Research shows that soil pH was the key factor affecting DOM composition in the upper soil layers during peatland succession [13]. In addition, available nutrients and magnesium (Mg) forms in soils were positively associated with labile carbon inputs, while available calcium (Ca) forms were positively associated with stable carbon inputs, demonstrating that the availability of soil nutrients and minerals are influential factors of the DOM carbon-containing groups [14, 15]. Land use patterns, ecosystem types and planting years also affect the content, nature and composition of soil DOM. Early works in various terrestrial ecosystems have found that the concentration of DOM in soil generally decreased from forestland > grassland > cropland [16–19]. Li et al. [20] showed that the transition from evergreen

broad-leaved forest to long-term chestnut plantation increased the aliphatic and aromatic compounds of soil DOM. Wang et al. [21] investigated the DOM characteristics of farmland and forestland in a significant urban fringe area in eastern China, and they found that the aromaticity of farmland soils was higher than that of forest. Li et al. [19] found that the DOM content of forestland increased with time since stand establishment, especially after 20 years. Therefore, it is essential to identify soil DOM storage and composition parameters, particularly under conditions of environmental changes.

Several modern techniques have been used to evaluate the physicochemical characteristics of soil DOM (e.g., molecular weight fractions and components), including ultraviolet–visible (UV–Vis) and three-dimensional (3D) fluorescence spectroscopy [22, 23]. Previous studies have shown that UV absorbance at 254 nm ($SUVA_{254}$) can reflect the aromaticity level of DOM, with higher absorbance indicating higher aromaticity [24–26]. Peng et al. [25] showed an increase in the $SUVA_{254}$ value of DOM in an intercropping soil, indicating high aromaticity and hydrophobicity of DOM under this cropping practice. In addition, the ultraviolet absorbance ratio of 254 to 365 nm (E2/E3) can reflect the proportionality of the relative estimated DOM molecular weight size to the DOM molecular weight [27, 28]. Zhang et al. [27] showed significant increases in the E2/E3 values by 21–25% following phosphate fertilizer applications, demonstrating the role of phosphate fertilization in increasing low molecular weight fractions of DOM. Several researchers have used fluorescence spectroscopy to comprehensively study DOM characteristics, trace DOM sources, and assess DOM quality in various environmental systems [17, 29]. The humification index (HIX), fluorescence index (FI), and biological index (BIX) were used in several studies to determine the humification degrees, main sources, and autogenic contributions of DOM, respectively [17, 29]. Many studies have assessed DOM characteristics by combining excitation–emission matrices (EEMs) with parallel factor analysis (PARAFAC) [13, 24, 30]. Zhang et al. [14] used the EEMs-PARAFAC approach and found that interplanting white clover in orchards can effectively improve the accumulation of macromolecular humic-like compounds of DOM, potentially promoting SOC storage. Moreover, Filep et al. [31] used the EEMs-PARAFAC approach to investigate high molecular weight compounds of DOM fractions that may be derived from lignin-like structures of the particulate organic matter fraction.

Wolfberry is a deciduous woody perennial plant belonging to the *Solanaceae* family, known for its liver and kidney-nourishing, lung-moistening, and sight-enhancing functions [32, 33]. In addition, wolfberry is a

salt-tolerant plant species that can effectively reduce soil salinity [34], explaining its widespread cultivation in arid and semi-arid areas of Northwest China [35]. Ningxia is an autonomous region of the People's Republic of China that has become an important agricultural region in China due to its typical geographical and climatic conditions. Indeed, wolfberry cultivation has become an important means of increasing economic income and reclaiming saline-alkali lands in the Ningxia region. Studies have shown that changes in the growth status of above-ground plants will correspondingly affect changes in underground microbial communities [36]. Therefore, different planting years and soil microbial community changes have become important factors for the sustainable development of wolfberry industry [37]. The results showed that the content of soil organic matter in 0–30 cm wolfberry fields of Ningxia increased with the increase of tree age, which affected the content and properties of DOM [38]. However, other studies have shown that long-term mono-cultivation of wolfberry will affect the input quality of litter, and secondary salinization will occur in soil, thus inhibiting the root activity of wolfberry, inducing oxidative damage of root cells [39], reducing the diversity of soil microbial community [40], affecting the accumulation and decomposition of soil organic matter, and thus affecting DOM. So far, few studies have investigated the effect of planting years on soil DOM structure complexity in wolfberry orchard. Therefore, it is necessary to further study the response degree of soil DOM and the change mechanism of chemical composition in different planting years of wolfberry orchard soil. Zhang et al. [24] found that cultivation and maintenance of clover in the orchard promoted the accumulation of DOM and the humic-like content, increasing the potential for soil C sequestration, this gives us a good insight. Based on this insight, the present study aims (a) to analyze the changes in the DOM compositions under wolfberry plantations with different planting years; (b) to explore the main factors influencing soil DOM. We hypothesized that increases in plantation ages of wolfberry increase the contents of soil DOM and its molecular compounds by affecting soil physicochemical properties.

Materials and methods

Site description

This study was conducted at the wolfberry planting base (37°29'N, 105°38'E, 1348 m a.s.l.) in Zhongning County, Zhongwei City, in the NingxiaHui autonomous region (NHAR), China (Fig. 1). This region is located on the south bank of the Yellow River, belonging to the northern temperate continental monsoon climate zone, with average annual precipitation, average annual temperature, and average annual evaporation of 201 mm, 9.5 °C, and

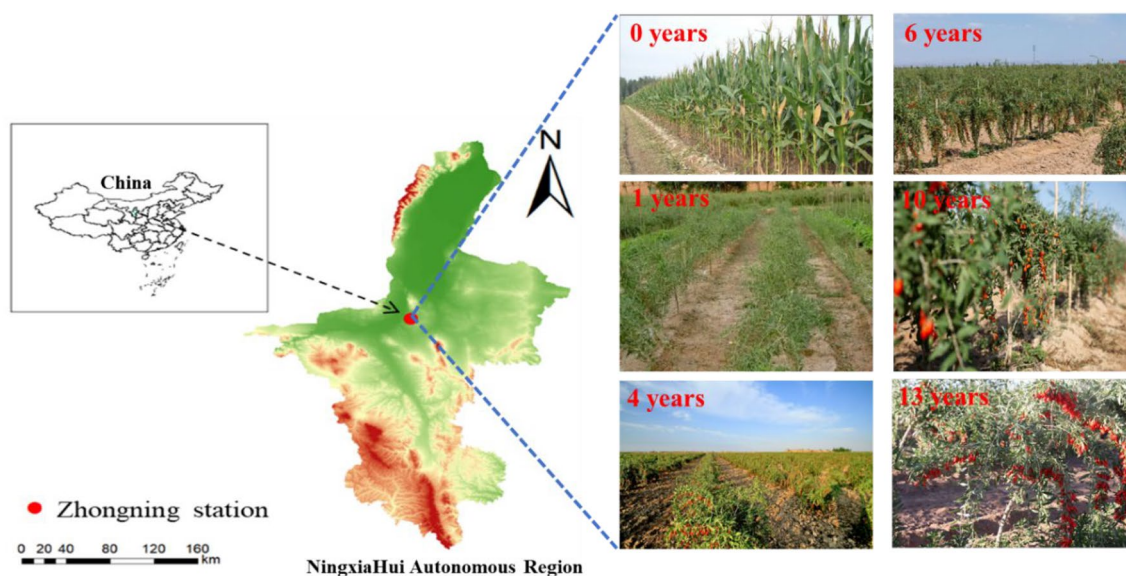


Fig. 1 Geographic location of the study area and image of corn field and wolfberry orchards with different planting ages

1947 mm, respectively. Approximately 60% of the precipitation amount occurs in the growing season from June to August. The soil classification is Aridisols (IUSS Working Group WRB, 2007) by widespread irrigated silty loam soils. In this study, wolfberry orchards were selected from an agricultural cooperative with similar agricultural management practices. The wolfberry planting density in these orchards was 220 plants·ha⁻¹, with row and plant spacing of 1.7 and 1.1 m, respectively. An organic fertilizer was used as a base fertilizer at a rate of 9000 kg/ha. This fertilizer was spread and incorporated at the beginning of April each year. In addition, mineral fertilizers were also applied twice annually in April and August. The total amounts of N, P₂O₅, and K₂O applied were 500, 240, and 50 kg/ha, respectively. In the traditional agricultural area of the Yellow River Oasis in Zhongning County, corn is the main crop. After planting corn, the humification degree of soil DOM increases and the DOM component is mainly humic acid [41]. But in order to promote regional economic development, most of the corn fields have been converted to wolfberry cultivation. Therefore, in order to investigate the changes of DOM content and composition after the conversion from farmland to wolfberry planting, corn fields with the same soil texture and type as wolfberry were selected as the control.

Sample collection and processing

In this study, specimens from five wolfberry plantations of different ages (1, 4, 6, 10, and 13 years) were collected in July 2022. Surrounding corn fields were used as a blank control (0 years). Six plots were selected for each stand

age (36 plots), and 20×20 m subplots were randomly set in each plot as replicates. Five 'S'-shaped points were randomly excavated in each subplot to collect undisturbed soil samples from the 0–10 cm soil layer after cutting the above-ground herbaceous layer and removing litter. In total, 36 soil composite samples of approximately 1000 g were collected (6 plots×6 replicates×1 soil layer). The collected soil samples were first air-dried in the laboratory and removed from gravel, plants, and other debris, then ground and sieved into two parts. The first part was analyzed for soil physicochemical properties, while the second one was analyzed for soil DOM.

Methods of soil analysis

Determination of soil physical and chemical properties and extraction of DOM

Soil total nitrogen (TN) was determined using the Kjeldahl method [42]. Available phosphorus (AP) was extracted by 0.5 mol/L NaHCO₃ and determined using the molybdate colorimetric method [43]. Soil organic matter (SOM) was determined using the external heating H₂SO₄–K₂Cr₂O₇ oxidation method [44]. The exchangeable Na⁺, K⁺, Mg²⁺, Ca²⁺ cations and the total exchangeable base (TEB) were determined by the method of ammonium acetate replacement method. The exchangeable cations in the leachate were determined using atomic absorption spectrometer [45]. Soil pH was measured with an S20K pH meter (Mettler Toledo, Switzerland) at a soil/water ratio of 1:2.5 [46].

Soil DOM was extracted using the soil–water oscillation method [47]. In fact, 5 g of the air-dried soil samples

(0.25 mm) were first placed in centrifuge tubes and mixed with deionized water at a soil/water ratio of 1:5, then agitated for 30 min and centrifuged for 10 min at 3000 r/min. The supernatant was filtered through a 0.45- μ m filter membrane to obtain the DOM solutions. The solutions were stored in a refrigerator at 4 °C for subsequent DOC and spectral analyses. Soil DOM concentration was represented by DOC and determined using a TOC analyzer (TOC-V CSH/CPN, Shimadzu, Japan).

Spectrum determination and parameter calculation

The UV–Vis spectra of the DOM samples in a 1-cm quartz cuvette were determined using a TU-1900 spectrometer (Beijing Purkinje General Instrument Co. Ltd., Beijing, China) with a scanning wavelength range of 200–800 nm. Deionized water was used as a measurement blank for the UV–Vis spectral analysis. In this study, the UV–Vis spectra relevant parameters included UV_{254} , E2/E3, E3/E4, and $SUVA_{254}$. Among them, UV_{254} reflects the DOM concentration and aromaticity [48], while $SUVA_{254}$ refers to the UV_{254} to DOC concentration ratio, representing the DOM aromaticity [49]. Whereas E2/E3 and E3/E4 refer to the 254 to 365 nm and 300 to 400 nm ultra-violet ratios, representing the DOM molecular weight/size and humification/aromatic degree, respectively [50].

The EEMs of the DOM solution samples were measured using a F7000 fluorescence spectrometer (Hitachi, Japan), with excitation (Ex) and emission (Em) wavelength ranges at 5 nm steps and 5 nm increment of 200–500 nm and 250–550 nm, respectively. The scanning speed was fixed at 1200 nm min⁻¹, while Raman scattering-derived artifacts were eliminated by subtracting the reference measurement of the water blank. In this study, PARAFAC modeling was performed to evaluate the DOM fluorescence components and to identify the source of characteristic variables using the DOMFluor v.1.7 toolboxes in Matlab 2021. The FI was determined by calculating the Em intensity ratio at 450 to 500 nm at an Ex wavelength of 370 nm to reveal the sources of the humic-like substances in soil DOM. Indeed, low and high FIs of about 1.4 and 1.9 suggest terrestrial and microbial sources, respectively, of the humic-like substances [51]. On the other hand, the BIX refers to the Em fluorescence intensity ratio at 380 to 430 nm at an Ex wavelength of 310 nm, reflecting the intensity of biological features of DOM. BIX values greater and lower than 1 imply high and low production of DOM components from biological sources, respectively. The higher the BIX value, the higher the DOM degradation degree and the higher the autotrophic product concentration [52, 53]. The HIX can be used to reflect the humification degree of soil DOM. It was determined in this study by calculating the Em spectra

peak area at the 435–480 nm wavelength range to the sum of the peak areas at the 300–345 nm and 435–480 nm wavelength ranges at an Ex of 254 nm. HIX greater than 6 indicates high humification and terrigenous contribution, while HIX lower than 4 indicates that the humification degree of soil DOM is dominated by autogenetic DOM [54].

Data processing and analysis

Excel 2021 was used to organize the collected data. One-way analysis of variance (ANOVA) was performed using SPSS. 26.0 (IBM, USA) to determine whether the differences in the soil physicochemical properties and DOM spectral characteristics between the different wolfberry planting ages were statistically significant at the $P < 0.05$ level. The relationships between the DOM components, spectral parameters, and soil physicochemical properties were assessed using principal components analysis (PCA) and Pearson correlation analysis. All figures were generated using Origin 2021.

Results

Soil physicochemical properties and DOM contents at different wolfberry plantation ages

The results showed significant increases in the SOM, TN, and AP contents with increasing wolfberry planting ages ($P < 0.05$). Compared with the control group (0 years), the AP content was significantly increased by 24.70% at a planting age of 10 years, while the TN and SOM contents were increased by 27.74 and 26.52%, respectively, at a planting age of 13 years. On the other hand, the total exchangeable base ions (TEB) content in the soil exhibited a significant decrease with increasing wolfberry planting age ($P < 0.05$). Indeed, compared with the control group (0-yr), the TEB, K⁺, Ca²⁺, Na⁺, and Mg²⁺ contents decreased by 27.22, 27.69, 8.88, 33.33, and 17.21%, respectively, with increasing planting age. However, there were no significant differences in the pH and C/N values between the different planting ages ($P > 0.05$) (Table 1).

The results revealed an increase in the DOM content with increasing planting age (Fig. 2). In fact, the DOM contents at wolfberry planting ages of 10 and 13 years were significantly higher than those observed at the other planting ages ($P < 0.05$). Compared with the control group, the DOM content increased significantly by 176.58 and 190.20% at planting ages of 10 and 13 years, respectively. The above results showed that DOM content, SOM, TN and AP contents in soil significantly increased with the increasing of plantation ages of wolfberry ($P < 0.05$), the results are consistent with our hypothesis.

Table 1 Effects of the wolfberry planting age on the soil physicochemical properties

Parameters	Planting age (y)					
	0	1	4	6	10	13
SOM (g·kg ⁻¹)	23.54±1.72 bc	21.45±4.01 bc	21.15±1.15 c	21.54±1.94 bc	25.48±1.51 b	30.07±0.93 a
pH	8.11±0.03 a	8.16±0.03 a	8.12±0.06 a	8.18±0.01 a	8.16±0.03 a	8.22±0.03 a
AP (mg·kg ⁻¹)	118.6±22.41 bc	100.9±8.32 bc	98.23±4.70 c	121.3±2.40 bc	147.9±12.70 a	123.57±12.43b
TN (g·kg ⁻¹)	1.32±0.13 b	1.17±0.23 b	1.19±0.09 b	1.23±0.15 b	1.43±0.06 b	1.67±0.03 a
C/N	10.34±0.33 a	10.62±0.20 a	10.32±0.19 a	10.21±0.36 a	10.35±0.22 a	10.46±0.15 a
TEB (coml·m ⁻²)	27.22±0.47 a	25.91±0.47 b	25.7±0.85 bc	25.27±0.12 bc	25.26±0.17 bc	24.99±0.37 c
K ⁺ (coml·m ⁻²)	0.65±0.05 a	0.57±0.08 ab	0.53±0.07 b	0.5±0.04 b	0.51±0.05 b	0.47±0.05 b
Ca ²⁺ (coml·m ⁻²)	23.08±0.41 a	21.73±0.24 b	21.58±0.09 b	21.43±0.63 b	21.14±0.24 b	21.03±0.33 b
Na ⁺ (coml·m ⁻²)	1.11±0.20 a	0.94±0.08 ab	0.89±0.03 ab	0.84±0.22 ab	0.83±0.15 b	0.74±0.03 b
Mg ²⁺ (coml·m ⁻²)	2.86±0.03 a	2.84±0.07 a	2.68±0.01 b	2.49±0.05 c	2.45±0.06 c	2.44±0.06 c

Different lower-case letters indicate significant differences between the wolfberry planting age ($P < 0.05$)

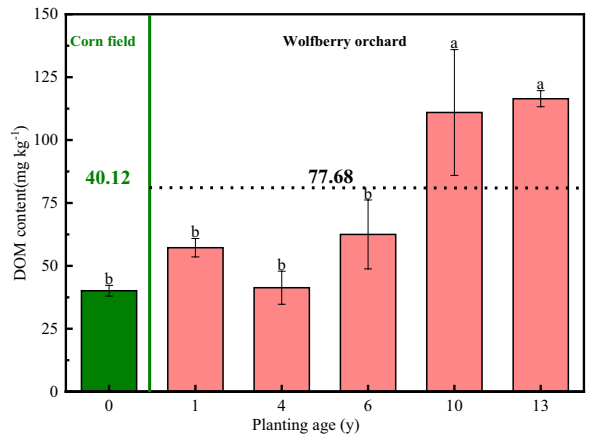


Fig. 2 DOM contents under the corn field and five wolfberry plantations with different ages. Different lower-case letters indicate significant differences in the DOM contents between the wolfberry plantation ages ($P < 0.05$). The dotted line indicates the mean value calculated from the DOM data of the five wolfberry plantations with different ages

Spectral properties of soil DOM at the different wolfberry planting ages

UV-Vis spectral features

The UV₂₅₄, SUVA₂₅₄, E₂/E₃, and E₃/E₄ indices were used in this study to assess the chemical characteristics of soil DOM released at the different wolfberry planting ages using UV-visible spectrophotometry (Fig. 3). Compared with the results obtained in the corn field (0-yr), substantial decreases in the UV₂₅₄ and SUVA₂₅₄ values of soil DOM with increasing planting age were observed (Fig. 3A, B). In fact, the UV₂₅₄ and SUVA₂₅₄ of soil DOM initially decreased with increasing planting age. The UV₂₅₄ and SUVA₂₅₄ values at the wolfberry planting age of 6 years were considerably higher than

those observed at the remaining planting ages (Fig. 3A, B), indicating comparatively higher DOM aromaticity at this age. The E₂/E₃ and E₃/E₄ showed gradual increases with increasing wolfberry planting age, indicating lower aromatics and molecular weight of soil DOM (Fig. 3C, D). The results showed that the aromatic property of farmland was greater than that of wolfberry forest land, but with the increase of planting years, the aromatic property of soil DOM increased first and then decreased, and the molecular weight decreased.

3D-EEM spectral features

According to the classification of the five regions established by [47], three fluorescence peaks were observed in the soil DOM three-dimensional fluorescence spectra at the different wolfberry planting ages (Fig. 4). Fluorescence peaks 1, 2, and 3 belonged to soluble microbial metabolites, humic acids, and tryptophanoid, respectively. These peaks were observed at wolfberry planting ages of 1, 6, and 13 years. Compared with the results observed in the corn control group (0 years), the fluorescence intensity of Peak 1 showed a decreasing-increasing trend with increasing wolfberry planting age. The lowest and highest fluorescence intensities at the wolfberry planting ages of 4 years were attributed to Peak 1 and Peak 2, respectively. This finding indicates a decrease and increase in the contents of soluble microbial metabolites and humic acids, respectively, suggesting transformations of these two compounds. Peak 2 showed a blue shift with increasing wolfberry planting age. Whereas Peak 3 only appeared at wolfberry planting ages of 1, 6, and 13 years, showing gradual temporal increases, indicating a gradual transformation of humic acids into tryptophan and, consequently, decreasing the aromatic and molecular weight of SOM.

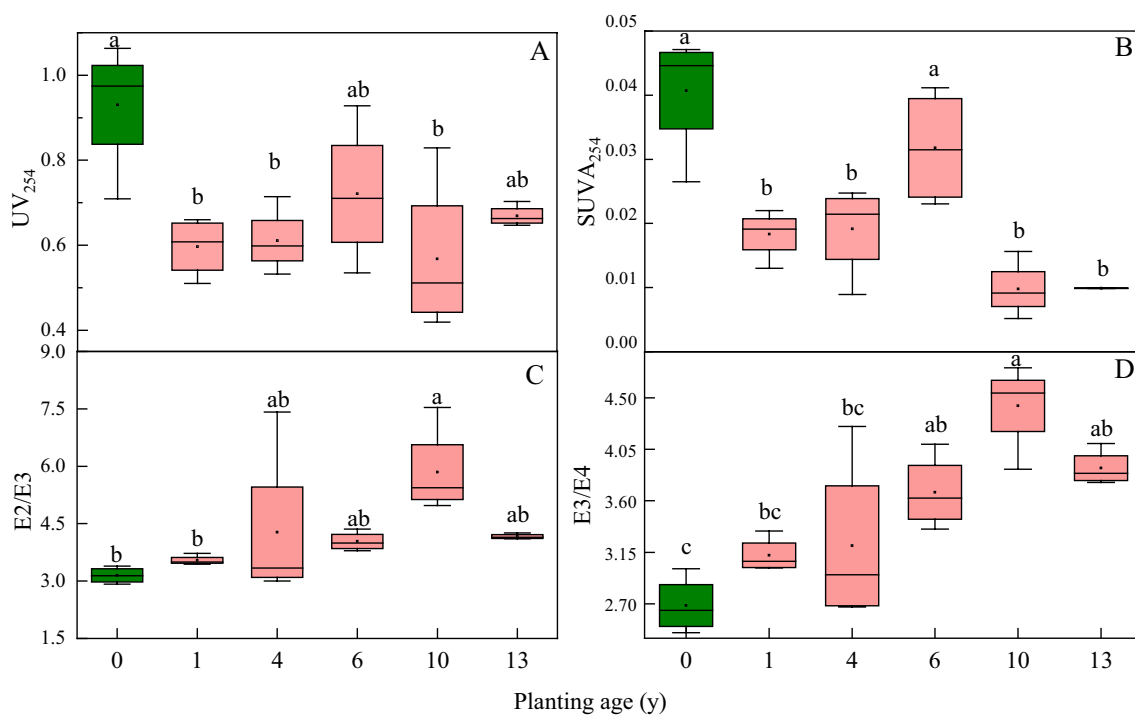


Fig. 3 UV₂₅₄ (A), SUVA₂₅₄ (B), E2/E3 (C), and E3/E4 (D) of soil DOM under the corn field and five wolfberry plantations with different ages. Different lower-case letters indicate significant differences between the wolfberry planting ages ($P < 0.05$)

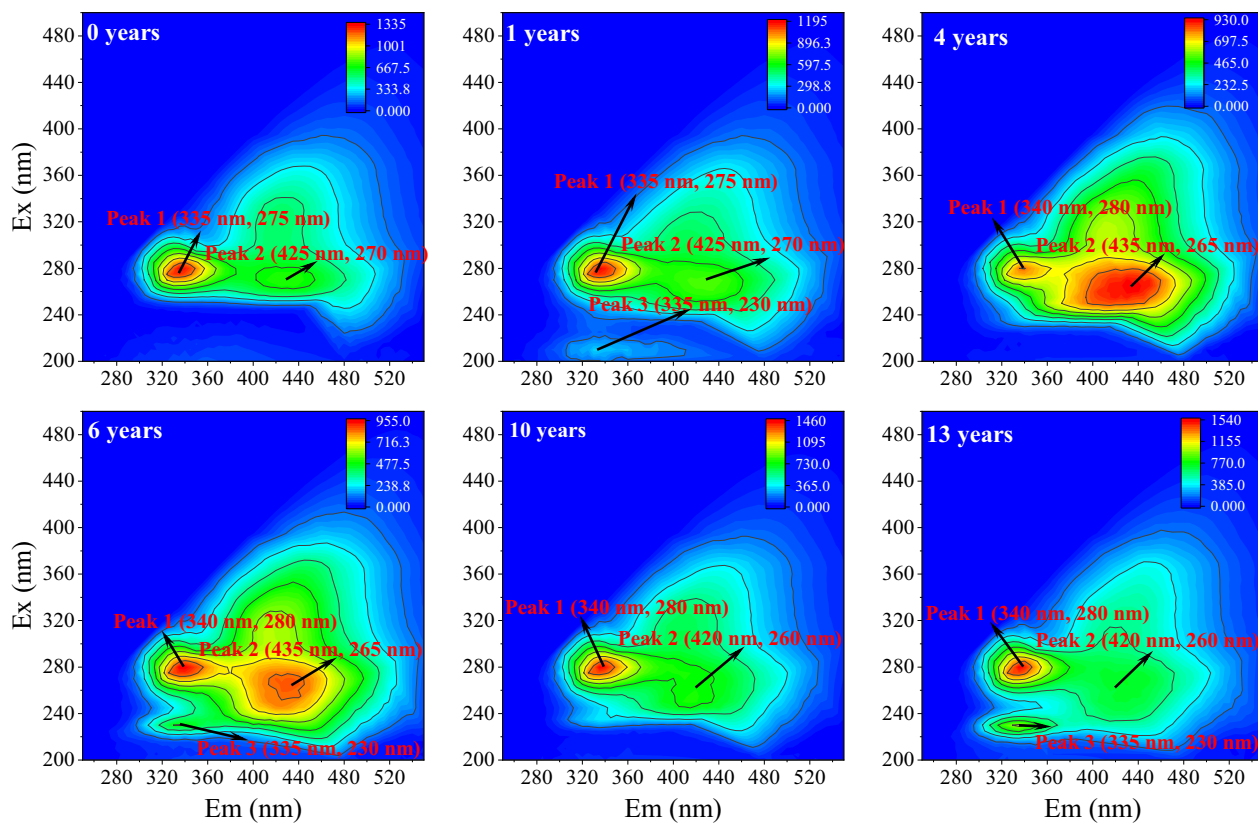


Fig. 4 Three-dimensional fluorescence spectral characteristics of soil DOM under the corn field and five wolfberry plantations with different ages

The FI is negatively related to aromatic contents and is typically employed to identify the sources of DOM [55]. The FI of soil DOM at the different wolfberry planting ages ranged from 1.42 to 1.93 (Fig. 5A), indicating that both microorganisms and plant residues were the main sources of the soil DOM pool. The FI value of soil DOM at the wolfberry planting age of 1 year was 1.42, indicating that plant root, stem, and leaf residues were the main sources of soil DOM at this planting age (Fig. 5A). However, the average FI value at the wolfberry planting age of 6 years was greater than 1.8 (Fig. 5A), which is closer to the endogenous characteristic value of 1.9. The source of soil DOM at this planting age was relatively dominated by microbial metabolism and degradation products. The results showed that soil DOM changed from terrestrial source to biological source with increasing wolfberry planting age, decreasing the contents of aromatic compounds. The BIX value of soil DOM ranged from 0.6 to 0.7 (Fig. 5B), indicating that mixed terrigenous and microbial substances were the main sources of soil DOM. The HIX is an indicator of the condensation and conjugation degrees of aromatic structures and unsaturated aliphatic chains, respectively, in soil DOM [55]. Compared with the control group (0 years), significant decreases in the HIX value of soil DOM were observed after planting wolfberry. The lowest HIX value was observed at the wolfberry planting age of 1 year (Fig. 5C). Therefore, the conversion of corn fields to wolfberry orchards decreased the humification degree of soil DOM. On the other hand, the HIX value showed a gradual increase with increasing wolfberry planting age (Fig. 5B), indicating an increase in the humification degree of soil DOM and, consequently, increasing the contents of aromatic compounds (e.g., humus).

The fluorescence EEM spectra of three DOM components are shown in Fig. 6. C1 (Ex/Em = 235, 280/335 nm) refers to a protein-like DOM component rich in carboxyl functional groups and aromatic cyclic amino acids, mainly produced by microbial and heterotrophic organisms. C2 (Ex/Em = 225/340, 440nm) refers to a mixture of protein and fulvic acid-like components, while C3 (Ex / Em = 265/435) indicates fulvic acid-like components. These results indicate that soil DOM was relatively stable, with high aromaticity degrees.

The fluorescence intensities of the EEM-PARAFAC components are shown in Fig. 7. According to the obtained results, C1 and C3 exhibited the highest contributions to the fluorescence intensities. In addition, the contribution rate of fluorescence intensity of C1 was the largest at 0-yr, and that of C3 was the largest at 13 years. At the same time, after changing to long-term cultivation mode of wolfberry, C1 decreased and C3 increased with the increase of planting years. On the other hand,

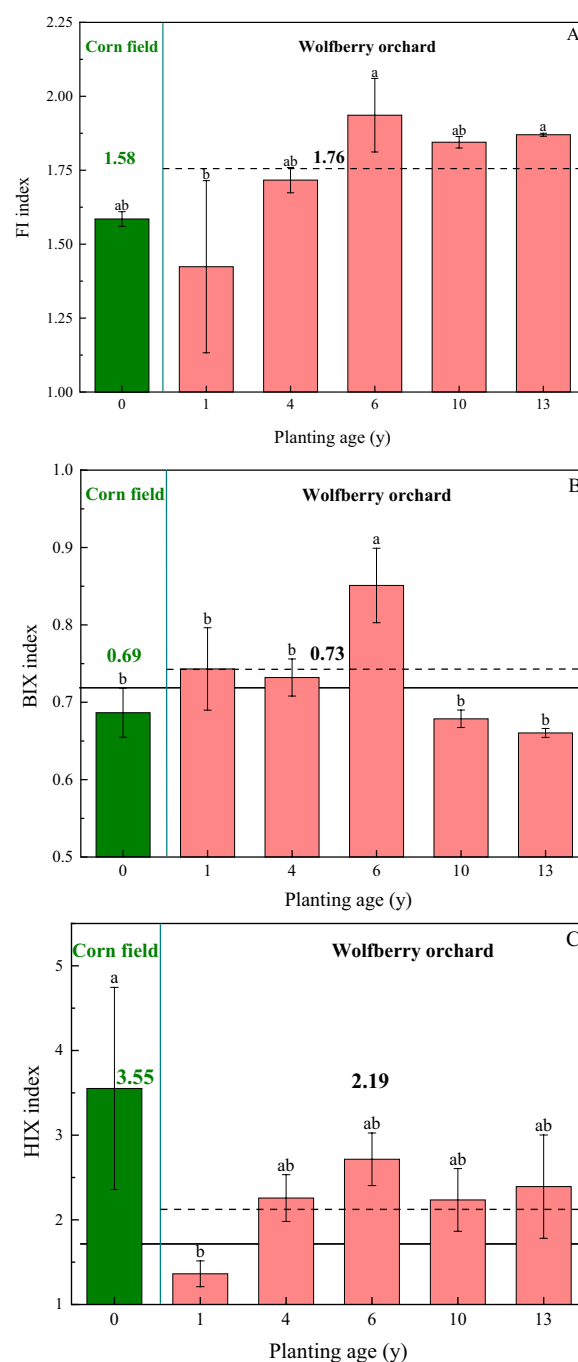


Fig. 5 FI (A), BIX (B), and HIX (C) values of soil DOM under the corn field and five wolfberry plantations with different ages. Different lower-case letters indicate significant differences between the wolfberry planting ages ($P < 0.05$). The dotted line indicates the mean value calculated using the data of the five wolfberry orchards with different planting ages

the contribution of C2 (mixture of protein-like and fulvic acid-like) to the fluorescence intensity showed an increasing–decreasing trend with increasing wolfberry

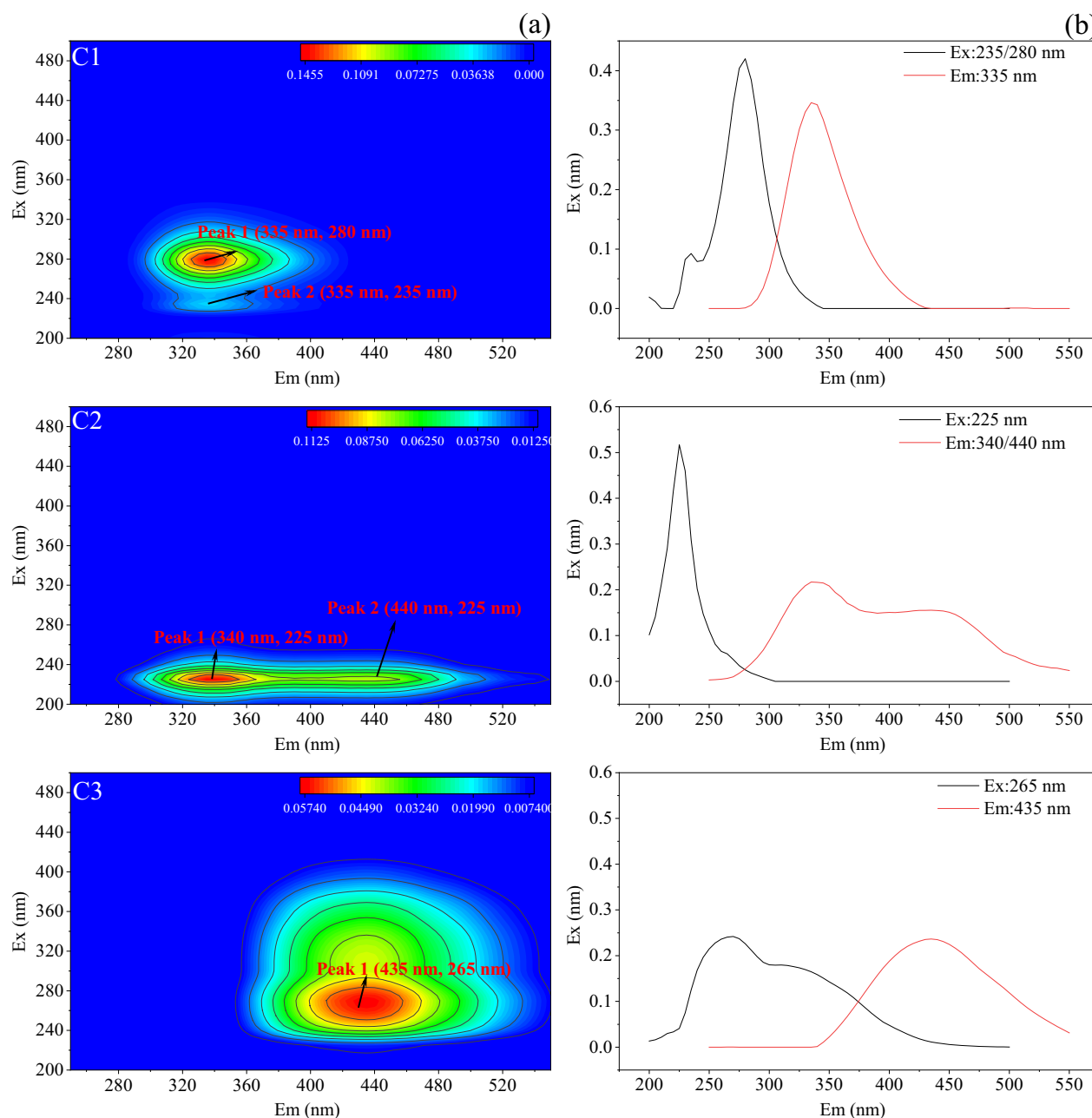


Fig. 6 Fluorescence spectra (a) and loadings (b) of different soil DOM components obtained using EEM spectroscopy and PARAFAC. C1 protein-like component, C2 mixture of protein and fulvic acid-like components, C3 fulvic acid-like component

planting age. The above results showed that after the conversion of farmland to wolfberry cultivation, with the increase of planting years, the main components of soil DOM were microbial metabolites and tryptophan (Fig. 4), the source of DOM moved from terrestrial to biological source (Fig. 5A), the degree of humification first decreased and then increased (Fig. 5C), and the fluorescence components of DOM gradually changed from

protein-like substances to fulvic acid-like substances (Fig. 7), this is consistent with our expected assumptions.

Relationships between the DOM compositions, spectral indices, and soil properties

In this study, Pearson correlation analysis was performed to evaluate the relationships between the soil physico-chemical properties and soil DOM (Fig. 8). The results

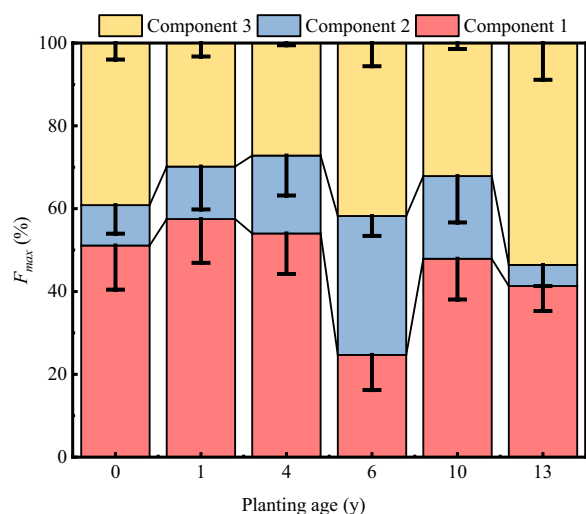


Fig. 7 Contribution rates of the three soil DOM components to the fluorescence intensities under the corn field and five wolfberry plantations with different ages. C1 protein-like component, C2 mixture of protein and fulvic acid-like components, C3 fulvic acid-like component

showed significant positive correlations of soil DOM with SOM, TN, and E3/E4 ($P < 0.05$), with correlation coefficients of 0.84, 0.83, and 0.87, respectively. The UV_{254} and $SUVA_{254}$ values were significantly and positively correlated with the soil Ca^{2+} contents ($P < 0.05$), with correlation coefficients of 0.83 and 0.84, respectively. On the other hand, E3/E4 exhibited significant negative correlations with Ca^{2+} , Mg^{2+} , Na^+ , and TEB ($P < 0.05$); whereas, the FI showed a significant negative correlation coefficient with soil Mg^{2+} of -0.93 ($P < 0.05$).

In this study, PCA was performed to evaluate the relationships between the soil physicochemical properties and DOM optical properties (Fig. 9). Principal component 1 (PC1) and principal component 2 (PC2) explained 54.7% of the total variance of the DOM fluorescence properties. PC1 was positively correlated with K^+ , Ca^{2+} , Na^+ , Mg^{2+} , and TEB, while PC2 exhibited strong positive and negative correlations with C/N and C2, respectively. The DOM components were correlated with the soil physicochemical parameters to varying degrees (Fig. 8). C1 (protein-like component) was positively correlated with the soil C/N, K^+ , Ca^{2+} , Na^+ , Mg^{2+} , and TEB

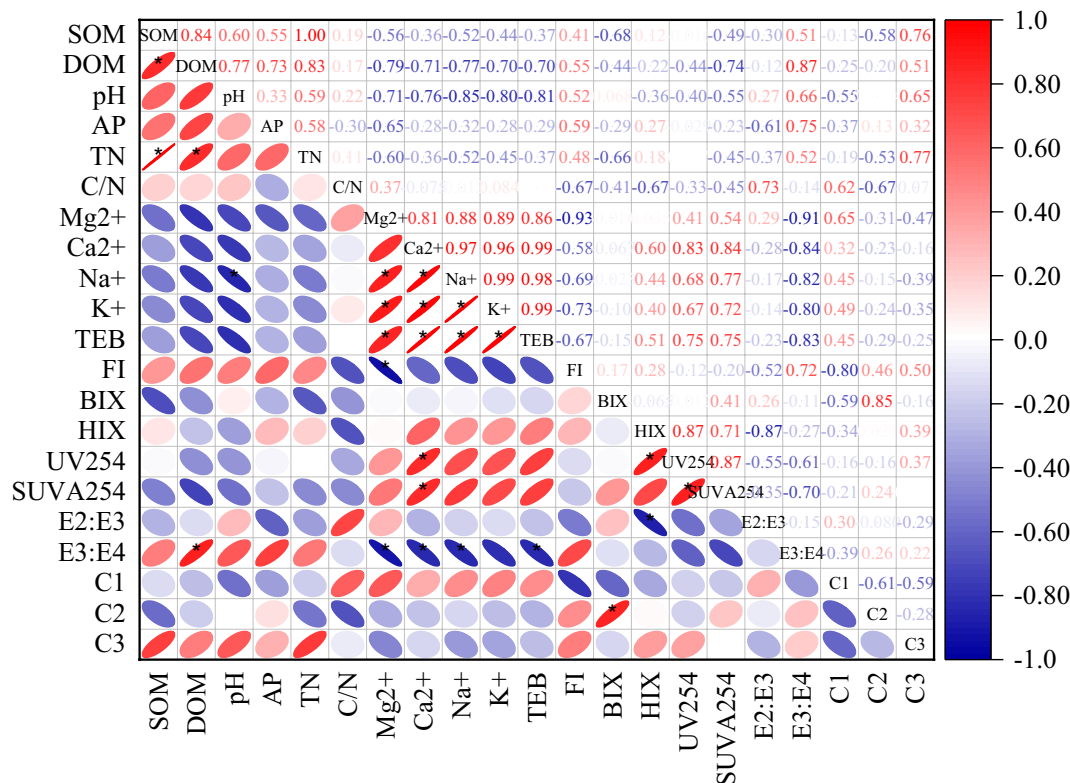


Fig. 8 Heatmap of the correlation between soil properties, fluorescence components, and indices. SOM soil organic matter, DOM dissolved organic matter, AP available phosphorus, TN total nitrogen, C/N carbon/nitrogen ratio, ExK exchangeable potassium; ExNa exchangeable sodium; ExCa exchangeable calcium, ExMg exchangeable magnesium, TEB soil total exchangeable bases, FI fluorescence index, BIX autogenetic index; HIX: humification index, C1 protein-like component, C2 mixture of protein and fulvic acid-like components, C3 fulvic acid-like component. * indicates significant correlations at the 0.05 level

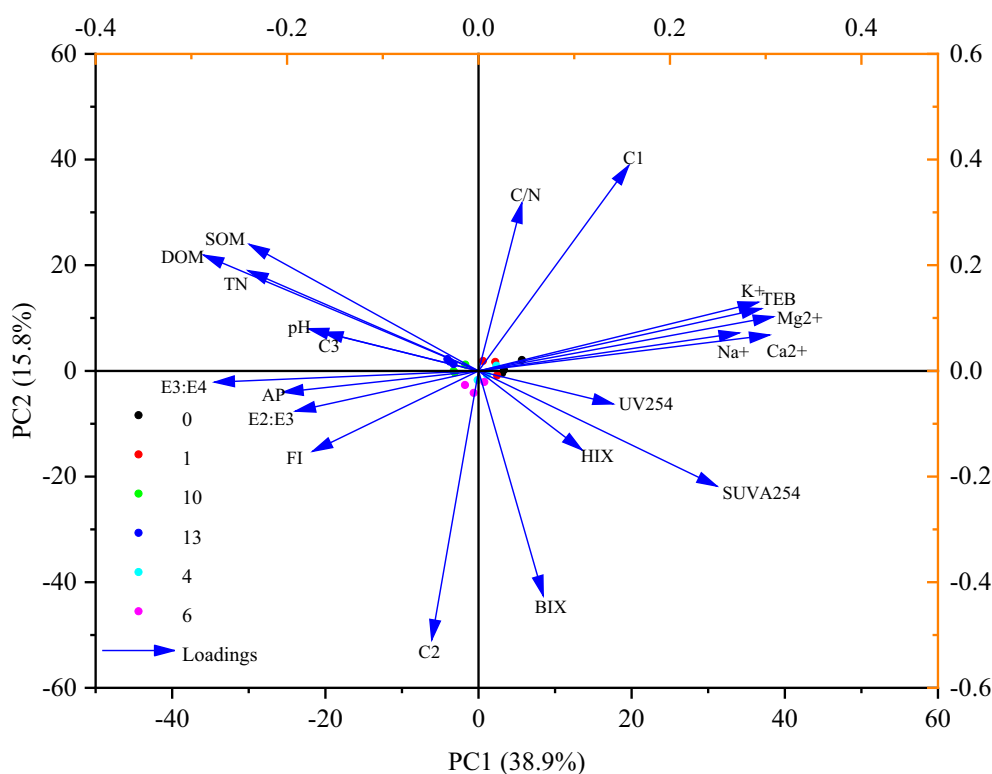


Fig. 9 Principal component analysis of the 3D DOM fluorescence properties and soil physicochemical properties under the corn field and five wolfberry plantations with different ages. SOM soil organic carbon, DOM dissolved organic matter, AP available phosphorus, TN total nitrogen, C/N carbon/nitrogen ratio, ExK exchangeable potassium, ExNa exchangeable sodium, ExCa exchangeable calcium, ExMg exchangeable magnesium, TEB soil total exchangeable bases, FI fluorescence index, BIX autogenetic index, HIX humification index, C1 protein-like component; C2 mixture of protein and fulvic acid-like components, C3 fulvic acid-like component

contents ($P < 0.05$), while C3 (fulvic acid-like component) was positively correlated with the soil pH, TN, and SOM ($P < 0.05$). The soil pH values were negatively correlated with the HIX and UV₂₅₄ values ($P < 0.05$), as well as positively correlated with the FI ($P < 0.05$). The results of PCA showed that the first principal component and the second principal component explained 54.7% of the fluorescence properties of DOM (Fig. 9), and the main factors affecting the quantity and quality of DOM in forest soil with different planting years were SOM, TN and Ca²⁺ (Fig. 8). The correlation between soil properties and DOM is exactly consistent with our hypothesis.

Discussion

Effects of the different wolfberry planting ages on the soil physicochemical properties and DOM contents

After the corn field is converted to wolfberry cultivation, the results showed increases in the SOM and soil TN contents with increasing planting age. These observations are consistent with those reported by Li et al., Xu et al., Zhao et al. [56–58]. The SOM and soil TN contents at the planting age of 1, 4 and 6 years were lower

than blank control, but at 13 years were significantly higher than those observed at the other planting ages (Table 1). On the one hand, this finding might be due to the farmland conversion into wolfberry orchards, resulting in less human interference (e.g., tillage), coupled with the increase of litter, residual roots and root secretions caused by wolfberry growth, resulted in the accumulation of SOM, so the SOM content in wolfberry orchards was higher than that in farmland ecosystems [59, 60]. This observation is consistent with the reported by Lu et al. [61]. On the other hand, long-term cultivation of wolfberry in semi-arid and arid regions can increase organic residue inputs, such as fine root biomass, litter biomass, above-ground biomass, and subsurface biomass, due to good soil moisture conditions, contributing greatly to the soil C pool, which is consistent with the results of Shi et al. [62]. Total N (TN) is an essential element for biological growth, improving plant productivity, and increasing SOM content [63]. In this study, long-term cultivation of wolfberry and the continuous application of litters and fertilizers/organic fertilizers continuous application of fertilizer/organic fertilizer in the wolfberry orchards

affected the quantity and quality of litter and root biomass [64], thus promoting the accumulation of soil TN content and affecting the SOM contents. These are consistent with the results of Zhang et al. [65] and Xue et al. [66] studies on the effects of different tea planting years on the physical and chemical properties of soil in tea gardens. Exchangeable base ions in soils (Ca^{2+} , Mg^{2+} , K^+ , and Na^+) are important indicators of soil quality, playing a crucial role in maintaining soil nutrients and buffering capacity [67]. The results of this study showed a decrease in the exchangeable base ion (Ca^{2+} , Mg^{2+} , K^+ , and Na^+) content with increasing wolfberry planting years (Table 1). First, this finding might be due to the requirement of high soil K^+ , Ca^{2+} , Na^+ , and Mg^{2+} amounts for the wolfberry growth process [68]. Second, soil K^+ , Ca^{2+} , Na^+ , and Mg^{2+} are the main elements utilized by wolfberry fruits [69]. In addition, the decrease in the exchangeable base ion contents might also be due to the effects of soil microorganisms or the environment, providing optimal conditions for the occurrence of leaching processes of these cations and, consequently, changing the soil nutrients of wolfberry orchard. This suggestion is consistent with the results reported by Wang et al. [69]. At the same time, other studies have found that under continuous planting and different land use types [70], soil exchangeable alkali ion content decreases, which may be due to the influence of leaching and soil properties [71], these are consistent with the results of this study.

The results of the chronosequence approach used in this study revealed increases in the DOM content in the 0–10 cm soil layer with increasing wolfberry planting years. Compared with the control group, there were significant increases in the DOM contents by 176.58 and 190.20% at the wolfberry planting ages of 10 and 13 years, respectively (Fig. 2), but the DOM content at the wolfberry planting ages of 4 years, respectively, no difference with the control group. This is consistent with the results of Li et al. [72, 73]. This finding suggests that long-term wolfberry planting led to a continuous increase in the DOM content over the entire study period. It might be due to the fact that long-term planting of wolfberry promoted increases in the plant, litter, and root biomass, which can undergo continuous microbial decomposition and, consequently, gradually increase soil DOM concentrations [19, 74]. At the same time, as perennial plants such as wolfberry remain on the soil surface, anaerobic microorganisms may increase and affect soil humidity and temperature, thus affecting the decomposition of plant residues and promoting DOM accumulation [75, 76]. The use of organic and chemical fertilizers is beneficial for the growth of wolfberry trees, providing more mineral nutrients and organic residues to the soils and potentially increasing the SOM and DOM

inputs. In our study, the DOM concentration was significantly and positively correlated with the SOM and TN contents, with correlation coefficients of 0.84 and 0.83, respectively, (Fig. 8, $P < 0.05$), indicated that high SOC and TN encourage plant development, and increasing litter and photosynthetic C input from plants boosts microbial growth and metabolism [77, 78], consequently affects the DOM concentration, this observation is consistent with the reported by Kalbitz et al. [6], further confirming that SOM and TN are important factors in soil DOM regulation. These observations are consistent with those reported by Zhang et al. [24], demonstrating the major importance of microbial biomass N in soil DOM variations in apple orchards on the Loess Plateau in China. The microbial community structure was changed with increasing wolfberry planting ages from bacteria to fungi [79–81], mainly increasing the community density and colonization ability of soil fungi [82]. Some previous studies have also highlighted the contributions of high above and below-ground biomass inputs to the increase in the fungal spore and mycelium densities through root secretions, promoting DOM accumulation in long-term wolfberry cultivations [83, 84]. Additionally, litter coverage in wolfberry orchards and microclimate changes can reduce SOC and nutrient losses caused by solar radiation [85], promoting DOM accumulation. This suggestion is consistent with the results of Wu et al. [86].

Effects of the different wolfberry planting ages on soil DOM quality

The low SUVA_{254} and high E_2/E_3 indicated low aromatic compound contents and DOM molecular weight, respectively. The SUVA_{254} values at the different wolfberry planting ages were lower than those observed in the control group. This observation is consistent with the reported by Wang et al. [21]. On the other hand, the E_2/E_3 values at the wolfberry planting ages of 1 and 4 years were higher than those in the control group (Fig. 3), indicating low soil DOM aromatic compounds and molecular weights under short-term wolfberry cultivations. This was probably due to the deep root systems of wolfberry trees, resulting in the gradual accumulation of the hydrophilic components with high aromaticity and molecular weight in deeper soil layers [87, 88]. However, the 0–10 cm soil layer in the farmland contained mainly maize roots. These materials provided sufficient energy for microbial growth, thereby enhancing the decomposition of these materials and the formation of humic substances [89]. This result was confirmed by the identified soil DOM components using the EEM-PARAFAC analysis. Compared with the control group, significant increase and decrease in the contributions of C1 and C3, respectively, to the DOM fluorescence intensity

were observed after short-term wolfberry cultivation (1 and 4 years) (Fig. 7). The decrease in the C3 components demonstrated that the conversion of farmland to wolfberry orchards decreases the contents of the fulvic acid-like component in soils. Previous studies have also found higher proportions of DOM-related humic-like substances in cropland soils than those in forest soils [90, 91]. In addition, Zhang et al. [92] detected more fulvic- and humic-like compounds following the conversion of forest land to farmland. This might be due to the perennial characteristics of wolfberry, leading to fewer plant residues and fast SOM decomposition under short-term wolfberry cultivation [86, 93]. In this study, we found significantly lower HIX values of soil DOM under wolfberry cultivation than those in the control group, especially in the first year of wolfberry plantation (Fig. 5C), indicating that short-term wolfberry plantation can decrease the humification degree of soil DOM. Soil DOM in the cropland and wolfberry orchards had relatively high and low humification degrees, respectively. Previous studies have demonstrated the capacity of maize and soybeans to enhance the accumulation of SOM and increase the humification degree [90, 94]. In this study, the FI and BIX of DOM in short-term planting wolfberry soils (1 and 4 years) ranged from 1.4 to 1.75 and 0.6 to 0.7, respectively (Fig. 5A and C), and there was no significant difference between farmland and short-term wolfberry orchards. It shows that plant source contributes more to DOM [95].

At the wolfberry planting ages of 6 years and 13 years, the C1 component contents decreased through biodegradation and mineralization, making C3 the main fluorescence component of soil DOM (Fig. 7), but the results were opposite at 10 years. This result was confirmed by the identified soil DOM components using the EEM-PARAFAC analysis. The results indicated that the humus degree increased during the long-term cultivation of wolfberry, which may be due to the fact that the number of litter and humus on the ground of wolfberry greatly increased during the planting years, thus promoting the production of lignin-derived aromatics, humus and condensation substances in DOM [96, 97]. This observation is consistent with the reported by Zhang et al. [98]. At the same time, Zhang et al. [24] reported increases and decreases in the relative abundances of humic-like and protein-like compounds, respectively, with increasing apple planting ages in the Loess Plateau in China, which also is consistent with our results. Long-term wolfberry planting can change the quality of litter inputs into soils [86, 99], which in turn promotes plant residue inputs and root exudates, providing additional carbon and energy sources for microbial activity and turnover. It has been proven that microorganisms in soil easily decompose proteins

used by microorganisms into monomers, which form humus in soil through polymerization [100]. Overall, after 6 years of wolfberries planting, FI values ranged from 1.76 to 1.93 (Fig. 5A), and DOM weights of plant residues on soil surface continued to decline, indicating that the source of DOM gradually shifted from plant stubble to related microorganisms and their metabolites [101]. This finding was in line with that reported by Li et al. [13], the results indicated that the litters of wolfberry orchard had lower lignin content and were more bioavailable to microorganisms [102], this finding was in line with that reported by Zhang et al. [98]. The HIX is closely related to soil microbial activities, reflecting humus contents or SOM humification degrees. The HIX values were less than 4 in our study, indicating that the lower degree of humification is dominated by autogenetic DOM [103]. On the other hand, the long-term wolfberry cultivation increased the HIX values, indicating the continuous supply of C sources (leaf litter and root exudates) with organic substances in the soil, effectively enhancing soil microbial activities and promoting macromolecular substance decomposition, thus forming more stable C components in soil DOM. The temporal increase in the HIX also suggested that DOM was depleted of labile-C components [12]. In addition, the obtained results in this study showed significant positive correlations between HIX and UV_{254} in DOM ($r=0.87$, $P<0.05$) (Fig. 8), suggesting the major role of DOM-derived sources served to soil microbial growth, enhancing the decomposition and humification of SOM [104, 105].

Wolfberry planting ages not only directly affect the chemical composition of soil DOM but also regulate it by mediating soil physiochemical properties. This study showed that there was a significant positive correlation between exchangeable Ca^{2+} content and DOM content, this finding was in line with that reported by Xiao et al. [106]. In addition, we found a significant positive correlation between the exchangeable Ca^{2+} contents and $SUVA_{254}$ values, suggesting an obvious Ca stabilizing effect on aromatic-C compounds. This finding explains the selective Ca^{2+} binding with specific organic components, promoting organo-Ca complex formation in arable soils [107, 108]. The variation in the DOM chemical composition was most strongly attributed to the basic soil properties, as well as to the Ca and Mg forms, demonstrating the major influences of these factors on soil physiochemical properties. Zhang et al. [14] found that stable C-containing groups were positively related to available Ca and Mg contents. This finding is, indeed, in line with that revealed in our study, showing that exchangeable cations are responsible for the microbial utilization and transformation of stable groups [109].

Conclusions

Our study demonstrated that the chronosequence of wolfberry plantations in the arid areas of Northwest China changed the quantity and chemical composition of soil DOM. Long-term planting of wolfberry increased the contents of SOM and DOM in soil, and decreased the content of aromatic compounds and molecular weight of DOM. The fluorescence components of DOM were mainly fulvic acids and the exchangeable Ca^{2+} contents enhanced the enrichment of stable components in soil DOM. In summary, this study has better understood the changes of soil DOM and its influencing factors under different land use types or planting years of wolfberry. Long-term planting of wolfberry can promote soil nutrient accumulation, increase the degree of humification and stability of DOM, and thus increase soil carbon sequestration potential. However, future studies on the relationship between DOM contents, DOM components, and microbial activities under wolfberry planting are required.

Acknowledgements

We thank MogoEdit (<https://www.mogoedit.com/>) for its linguistic assistance during the preparation of this manuscript.

Author contributions

RW: writing—original draft, methodology and formal analysis. BX: funding acquisition and project administration. BL and RG: data curation, investigation, software. GMN and MK: modify and make suggestions. YW and LF: conceptualization and supervision. KM, LD and HA: writing—review and editing. All authors have read and agreed to the published version of the manuscript.

Funding

We would like to acknowledge the Ningxia Key Research and Development Program (Grant numbers 2021BEB04076) for financing this research and Natural Science Foundation of Ningxia Province (Grant numbers 2022AAC05021) and National Natural Science Foundation of People's Republic of China (Grant numbers 42307439).

Availability of data and materials

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Ethics approval and consent to participate

Not applicable.

Competing interests

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 paper.

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Received: 28 November 2023 Accepted: 5 March 2024

Published online: 18 March 2024

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