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Interactive effects of microplastics and glyphosate on the dynamics of soil dissolved organic matter in a Chinese loess soil

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ABSTRACT

The increased use of plastic films and pesticides on agricultural soil leads to the accumulation of plastic debris and pesticide residues in soil. This accumulation has become a serious environmental issue, as it threatens life of earthworms, inhibits the enzyme activities and microbial diversity, and contributes to the loss of soil microbial carbon and nitrogen. However, little information is available regarding the effects of pesticides on soil dissolved organic matter (DOM). It is also unknown how plastic debris, especially small-sized particles called microplastics, influences the effects of pesticides on soil DOM. In this study, we performed a 30-day soil incubation experiment. Three levels of the common herbicide glyphosate were applied to soil: 0 (control, CK), 3.6 kg ha^{-1} (G1) and 7.2 kg ha^{-1} (G2). We also tested four levels of glyphosate and microplastics (homopolymer polypropylene powder) co-addition: 3.6 kg ha^{-1} + 7% (w/w) (M1G1), 3.6 kg ha^{-1} + 28% (w/w) (M2G1), 7.2 kg ha^{-1} + 7% (w/w) (M1G2), and 7.2 kg ha^{-1} + 28% (w/w) (M2G2). Glyphosate addition slightly increased soil fluorescein diacetate hydrolase (FDAse) and phenol oxidase (PO) activities. Although the glyphosate addition significantly promoted the accumulation of dissolved organic phosphorus (DOP) within the first 14 days, the M2 treatment decreased DOP at day 30. M2G1 and M2G2 increased soil FDAse activity and promoted the accumulation of DOC and DOP relative to G1 and G2 respectively while M1G1 and M1G2 benefited DON accumulation. Our results highlighted that the interaction between glyphosate and low microplastics content negatively affected DOC and DOP dynamics, leading to the loss of bioavailable C and P loss. The interaction between glyphosate and high content microplastics negatively affected DON compared with glyphosate addition, possibly decreasing DON.

1. Introduction

Dissolved organic matter (DOM) is a ubiquitous and heterogeneous mixture of aliphatic and aromatic organic compounds that range from simple organic molecules (e.g., carbohydrates, lipids, and proteins) to more complex organic molecules (e.g., humic and fulvic acids) in the soil matrix (McKnight et al., 2001). Protein-like fluorescence, the sum of tyrosine and tryptophan-like components, is a useful indicator of biodegradable DOC (Fellman et al., 2008). Humic acids with a high molecular weight are more readily degradable than fulvic acid, which has a lower molecular weight (Kisand et al., 2008; Rocker et al., 2012). DOM plays an important role in numerous chemical, physical and biological processes in soil, especially in the cycling of soil organic carbon (C), nitrogen (N) and phosphorus (P), and in the transformation and transportation of pollutant (Kalbitz et al., 2003; Kalbitz et al., 2000). DOM influences the transport budgets of total carbon, nitrogen, phosphorous, and others, from terrestrial ecosystems to aquatic environments, such as lakes, rivers, and estuaries. Hence, DOM is an important contributor to global elemental cycles (Battin et al., 2008; Stedmon and Markager, 2005; Tranvik et al., 2009).

In the last 50 years, the use of pesticides has greatly increased the quantity and quality of food (Arias-Estevez et al., 2008). However, >

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95% of samples collected from streams and almost 50% of samples collected from wells contained at least one pesticide, which could potentially pose a hazard to the environment and human health (Gilliom et al., 1999; Younes and Galal-Gorchev, 2000). Glyphosate ($C_3H_8NO_5P$, *N*-(phosphonomethyl) glycine) is a broad-spectrum, post-emergence, non-selective herbicide that is the most commonly used herbicide in the world (Wojtaszek et al., 2004). Glyphosate has been applied in China (mainly in orchards) for 30 years (Chen et al., 2015), and China has become the largest user of glyphosate in the world (Song et al., 2011). With repeated application, glyphosate and its main metabolite aminomethylphosphonic acid (AMPA) are among the pesticides that are most commonly found in surface/groundwater (Aparicio et al., 2013; Poiger et al., 2017; Ronco et al., 2016). Glyphosate and its metabolite remains primarily water-soluble after glyphosate is applied to soil (Cassigneul et al., 2016). Two actions of glyphosate in soil, glyphosate adsorption on and desorption from soil particles and glyphosate degradation, primarily control the transport of pesticides from the soil to water and alter the dynamics of soil DOM (Arias-Estevez et al., 2008; Linn et al., 1993). Glyphosate is used as a C and P source by microorganisms (Busse et al., 2001; Dick and Quinn, 1995; Schnurer et al., 2006), and is generally considered a toxicologically and environmentally safe herbicide (Busse et al., 2001; Duke and Powles, 2008; Lupwayi et al., 2007; Panettieri et al., 2013). However, negative effects of glyphosate on soil enzyme activity were also reported (Tejada, 2009; Tsui and Chu, 2003). The effect of glyphosate on soil microbial activity is dependent on soil characteristics, such as pH, texture and organic matter content (Albers et al., 2009; Tejada, 2009). The soil microbial biomass is considered a potentially important source of DOM, and microbial metabolites constitute a significant proportion of DOM (Kalbitz et al., 2000). Microorganisms also play a vital role in DOM decomposition, since up to 40% of released DOM is potentially biodegradable in solution in a period of days to a few months (Boyer and Groffman, 1996; Nelson et al., 1994). Thus, glyphosate in soil can alter DOM dynamics by influencing soil microorganisms and further affecting the cycling of C, N and P.

The plastic mulching technique plays an important role in Chinese agriculture to improve soil temperature, conserve soil moisture, and increase crop production (Wang et al., 2013b; Yan et al., 2010). The quantity of plastic film used in agriculture increased nearly four times—from 0.32 million to 1.25 million tons—from 1991 to 2011 (Yearbook, 2012). The large amount of plastic film residue causes serious environmental problems (Jambeck et al., 2015). The increasing accumulation of film residues significantly decreased soil microbial carbon and nitrogen, enzyme activities and microbial diversity (Farmer et al., 2017; Wang et al., 2013b; Wang et al., 2011). Ultraviolet (UV) light can render the plastic residues brittle in agricultural fields, which contributes to high amounts of microplastics (smaller than 5 mm) entering farmland soil (Barnes et al., 2009). Microplastics can be digested by earthworms (Huerta et al., 2016; Rillig et al., 2017), leading to microplastics leaching into deeper soil layers, which poses potential environmental risks. Small plastic particles are prone to absorbing pesticide and pollutants in the soil (Ivar do Sul and Costa, 2014; Ramos et al., 2015). However, plastic debris in soil enhanced microbial respiration, stimulated the activity of fluorescein diacetate hydrolase (FDAse), β-glucosidase, and phosphatase; and increased the nutrient content in soil dissolved organic matter (Liu et al., 2017; Yang et al., 2018). Small pieces of plastic debris can change soil physical properties such as porosity and air circulation (Rillig, 2012; Zhang et al., 2015), which might indirectly impact the glyphosate degradation process through soil properties and further affect dynamics of the soil DOM. However, no previous study has investigated the interaction between glyphosate and microplastics on soil DOM dynamics.

In this study, we combined PARAFAC modelling of fluorescence excitation-emission spectroscopy and specific UV absorbance to evaluate the changes in the chemical quality of soil DOM after the application of glyphosate and microplastics to soil. The main objective of this work was to evaluate the effect of glyphosate and the interaction between glyphosate and microplastics on various biochemical indicators of soil quality. We hypothesized that (1) glyphosate addition stimulates soil enzyme activities and promotes the accumulation of nutrients in DOM, and (2) microplastics addition promotes the positive effect of glyphosate on soil enzyme activity and further promotes the accumulation of nutrients in DOM.

2. Materials and methods

2.1. Experimental design

This experiment was performed in 2016 in a climate chamber (AGC-Doo3N, Hangzhou, China) at the Institute of Soil and Water Conservation, Chinese Academy of Sciences. The soil used in this experiment was collected from Ansai County (109°32′N, 36°87′W) in the Loess Plateau of China. The soil was a Huangmian soil (calcaric cambisols, FAO) developed on wind-deposited loessial parental material and characterized by the absence of bedding, loose silty texture, macroporosity, and wetness-induced collapsibility. The soil's initial properties are shown in Table S1. Two hundred grams (dry weight) of soil was incubated in a sealed 330 ml PVC pot. Two doses of glyphosate (3.6 kg ha^{-1} (G1) and 7.2 kg ha^{-1} (G2)) and two concentrations of microplastics (7% (M1) and 28% (M2), w/w) were used in this study. Our experiment contained 7 treatments: 1) CK: only soil; 2) G1: 3.6 kg ha^{-1} glyphosate; 3) G2: 7.2 kg ha^{-1} glyphosate; 4) G1M1: 7% microplastics and 3.6 kg ha^{-1} glyphosate; 5) G1M2: 28% microplastics and 3.6 kg ha^{-1} glyphosate; 6) G2M1: 7% microplastics and 7.2 kg ha^{-1} glyphosate; and 7) G2M2: 28% microplastics and 7.2 kg ha^{-1} glyphosate. The doses of glyphosate were determined according to local glyphosate application rates (Yang et al., 2015). The microplastics contents were determined based on the study of Huerta et al. (2016) that stimulated the hotspots of plastic debris in the field.

Glyphosate solutions were prepared by accurately dissolving glyphosate (98% purity, purchased from Dr. Ehrenstorfer, Germany) in distilled water. The final concentrations of the glyphosate solutions were 0.46 g l^{-1} and 0.92 g l^{-1}, corresponding to 3.6 kg ha^{-1} and 7.2 kg ha^{-1} and to a glyphosate concentration in pure dry soil of 11.5 μg g^{-1} and 23 μg g^{-1}, respectively. The glyphosate solutions were subsequently stored at 4 °C under non-sterile conditions until use. The microplastics source was analytical grade homopolymer polypropylene (materials for plastic film) powder (Youngling-TECH Company, Beijing, China), with a density of 0.91 g cm^{-3} and bending strength of 200 kg cm^{-2}. This material had a particle size < 250 μm, with 58.3% microplastics particles with sizes of 250–125 μm, 35.9% of 125–100 μm, 2.3% of 100–63 μm, 1.1% of 63–50 μm, and 0.6% of particle size < 50 μm.

The soil was slightly compacted using a small manual soil compactor to guarantee the same compaction in all samples. The soil moistures were maintained at 10% (approximately 60% of field capacity) throughout the experiment. The pots were incubated at 28 °C (relative humidity of 80%, 300 μ (photons) $m^{-2} s^{-1}$). The light was controlled automatically, with 16 h on and 8 h off. Because the soil was air-dried, pre-incubation was conducted for 1 week to re-establish microbial metabolism. Each treatment had three replicates, and this experiment contained 126 pots (7 treatments × 3 reps × 6 sampling points) in total. A sub-soil sample was sampled from each pot after 0, 1, 3, 7, 14, and 30 days after the microplastics and glyphosate were added to the soil, and finally 126 sub-samples were collected. Within 1 h of harvest, the soil samples were passed through a 2-mm sieve and hand-homogenized. One part of the soil was immediately used for fresh enzyme analysis. The other part of the soil was stored at 4 °C, and the extraction of soil DOM was completed within 1 day after the soil was stored at 4 °C.

2.2. Soil DOM concentration and composition analysis

The DOM solution was extracted by adding 120 mL distilled water to subsamples of 40 g homogenized soil (soil: solution, 1:3, w/w) according to Kalbitz et al. (2003). All soil extracts were centrifuged at 4000 rpm for 10 min and filtered through pre-rinsed 0.45-μm cellulose-acetate membranes (Schleicher & Schull). The filtered solutions were stored frozen and analysed within 1 week. In all samples, total dissolved N (TDN), DOC, NH_4^+, NO_3^-, total dissolved P (TDP), and PO_4^{3-} contents were measured using the standard soil test procedures of the Chinese Ecosystem Research Network (CERN Editorial Committee, 1996). DOC contents were determined using a TOC analyser (liquid TOC II, Elementar, Germany). TDN contents were determined using the alkaline persulfate digestion-UV spectrophotometric method (Doyle et al., 2004). TDP contents were measured using the ammonium molybdate spectrophotometric method (Galhardo and Masini, 2000). PO_4^{3-} contents were determined using the phospho-molybdenum blue method (Jarvie et al., 2002). NH_4^+ content was measured by an AA3 continuous flow autoanalyser (AutoAnalyzer3-aa3, Bran + Luebbe, Germany). NO_3^- content was determined by ultraviolet colourimetry with an ultraviolet spectrophotometer (UV2300, Shanghai, China). Dissolved organic N (DON) and dissolved organic P (DOP) contents were calculated as TDN- ($NH_4^+ + NO_3^-$) and TDP-PO_4^{3-}, respectively. EEM spectrograms of the subsamples were measured using an F-4600 fluorescence spectrometer (HITACHI, Japan). The detailed method for analysing EEM spectrograms was described in our previous study (Liu et al., 2017). The activities of two enzymes were measured: phenol oxidase (PO) and fluorescein diacetate hydrolase (FDAse). The detailed method for measuring the activities of these two enzymes was presented in our previous study (Liu et al., 2017). FDAse activity was measured using a method adapted from Daou et al. (2016) and Green et al. (2006). Briefly, 1 g of soil sample was added to 9 ml phosphate buffer (0.1 M, pH 7) and shaken for 30 min, after which 180 μl of soil suspension was mixed in 20 μl of 20 mM of fluorescein diacetate (FDA) solution (assay) dissolved in dimethyl sulfoxide (DMSO) or 20 μl of phosphate buffer (control) in a microplate well and incubated for 1 h at 37 °C. PO activity was determined spectrophotometrically in clear 96-well microplates using L-3,4-dihydroxyphenylalanine (L-DOPA) as a substrate as described by DeForest (2009).

Fig. 1 shows the fluorescent components and the proportional distribution of the components of soil DOM. Two protein-like fluorescence peaks were observed in component 1 (C1), which are centred at excitation/emission (Ex/Em) wavelength pairs of 215/280 nm and 260/280 nm. This component was associated with tryptophan-like substances that indicated more degraded peptide material (Coble et al. 1998, Parlanti et al. 2000). The Ex/Em wavelength pair of component 2 (C2) is centred at 225/450 nm, and this component is identified as UVC humic-like components, which are related to high molecular weight and aromatic humic (Murphy et al., 2006; Stedmon and Markager, 2005). The humic-like fluorescence peak, which is centred at the Ex/Em wavelength pair at 235/420 nm, is identified from component 3 (C3). This component is associated with high-molecular-weight humic material (Murphy et al., 2006; Stedmon et al., 2003) and is more labile than C2. In addition, the Ex/Em wavelength pair of component 4 (C4) is centred at 220/440 nm. This component originates from an UVA humic-like substance, which is associated with fluorescence resembling fulvic acid (Cory and McKnight, 2005; Stedmon and Markager, 2005).

2.3. Data analysis

The differences in soil enzyme activities, nutrient contents and fluorescence-specific components in DOM solutions on different sampling days ($P < 0.05$) were determined using one-way analysis of variance (ANOVA) with Duncan's multiple-range test. One-way ANOVA with Duncan's multiple-range test was used to compare the significant differences among treatments sampled on the same day ($P < 0.05$). All statistical analyses were performed using SPSS 21.0. The EEM data were analysed using MATLAB 2010a (MathWorks Inc., USA). PARAFAC modelling of the fluorescence EEMs was conducted with MATLAB using the DOMFluor toolbox (Stedmon and Bro, 2008) following the procedures described by Stedmon and Bro (2008). The figures were drawn using SigmaPlot 10.0 software.

3. Results

3.1. Dynamics of soil dissolved organic matter (DOM)

The dynamics of DOC, DON, NH_4^+, NO_3^-, DOP and PO_4^{3-} were observed during the incubation period (Tables 1, 2a, 2b, 2c, 3a, and 3b). G2 significantly increased the DOC content relative to CK (Table 1). M2G1 and M2G2 significantly increased the DOC content relative to CK, G1 and G2, especially between day 3 and 30. Compared to CK, the DOC content in M2G1 and M2G2 increased 92% at day 30, while it decreased 54.9% in M1G1.

G1 significantly increased the DON content relative to CK (Table 2a). A higher DON content was observed in M1G2 relative to CK, G1 and G2, especially at day 30. The DON content in M1G2 increased 92.1% relative to CK at day 30, while it decreased 60.5% in M2G1. The glyphosate addition significantly decreased NO_3^- in soil DOM solution, while no significant differences in NO_3^- were observed between co-addition of microplastics and glyphosate and CK treatments (Table 2b). No significant differences in NH_4^+ were observed between G1, G2, and CK treatments between day 3 and 30 (Table 2c). The co-addition of microplastics and glyphosate showed no significant effect on NH_4^+ relative to glyphosate addition and CK treatments between day 3 and 30.

The DOP and PO_4^{3-} contents in G2 were significantly higher than CK and G1 (Tables 3a and 3b). Compared to CK, the M1G1, M2G1 and M2G2 treatments increased DOP content 77.8%, 81.6% and 209.4%, respectively, at day 14 (Table 3a). The PO_4^{3-} content was the highest in the M2G2 treatment during day 3 to 14 (Table 3b). Similar to DOC, higher DOP and PO_4^{3-} contents were observed in M2G1 and M2G2 relative to G1 and G2, while DOP and PO_4^{3-} contents in M1G1 and M1G2 were lower than G1 and G2 (Tables 3a and 3b).

3.2. FDAse and phenol oxidase activities

The PO activity ranged from 4.8 to 8.0 μmol h^{-1} g^{-1}. G2 increased PO activity relative to CK at day 14, while G1 increased PO activity at day 7 (Table 4a). The co-addition of glyphosate and microplastics decreased phenol oxidase activities relative to CK at day 1, 3 and 30, but it significantly increased phenol oxidase activities at day 7. G1 and G2 dramatically promoted FDAse activity relative to CK (Table 4b). Compared to CK, FDAse activities in M2G1 and M2G2 increased 166.7% at day 30 while in M1G1 and M1G2, it increased by 80.0% and 93.3%, respectively.

3.3. Changes in the fluorescence excitation-emission matrices (EEMs) of DOM

During the incubation period, significant changes were observed in fluorescence-specific components after glyphosate and microplastics addition G2 significantly increased the C1 content relative to CK and G1 (Table 5a). The glyphosate addition had no significant effect on the contents of C2, C3 and C4 ($P < 0.05$) (Tables 5b, 5c, and 5d). The co-addition of glyphosate and microplastics significantly increased the C1 content relative to CK at day 30 (Table 5a). At day 14, the co-addition of glyphosate and microplastics dramatically decreased the C2 content (Table 5b). M1G1 and M1G2 dramatically decreased the C3 content between day 14 and 30, while significantly higher C3 contents in M2G1 and M2G2 relative to CK were observed at day 30 (Table 5c). M2G1 and M2G2 increased the C4 content between day 0 and 14, but M1G1 and

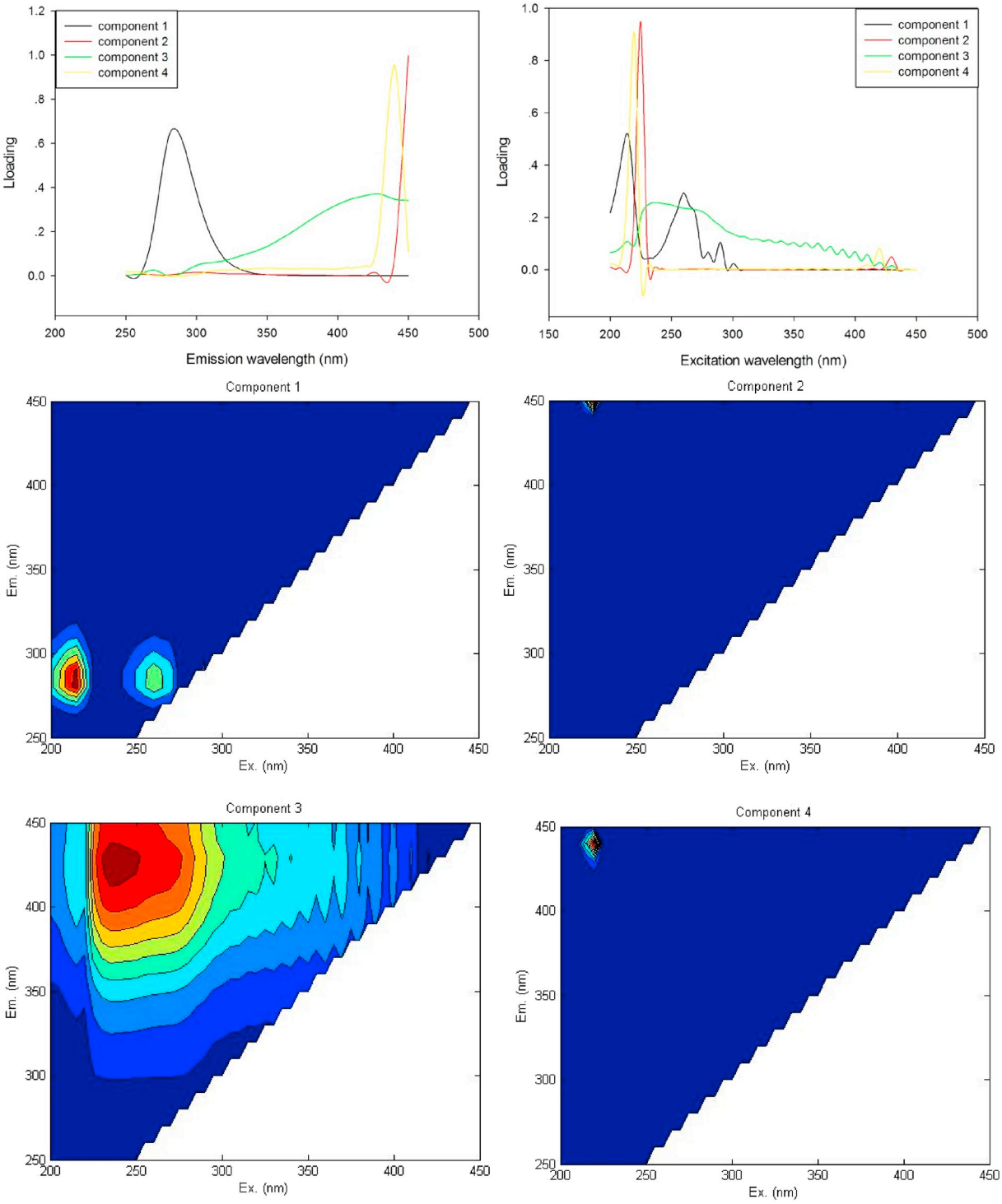

Fig. 1. The EEM spectra of the four component identified by PARAFAC analysis.

M1G2 dramatically decreased it at day 14 (Table 5d).

4. Discussion

4.1. Impacts of glyphosate addition on C, N and P pools in soil DOM and soil enzyme activities

Glyphosate is thought to be the herbicide with the strongest stimulating effects on soil biochemical properties compared with 2,4-dicholorophenoxyacetic acid and metsulfuron-methyl (Zabaloy et al., 2008). A previous study reported an evident and long-lasting stimulatory effect of glyphosate on soil enzyme activities (Panettieri et al., 2013). However, most studies have shown that glyphosate has no immediate direct effects or short-term effects on soil microbial activity and bacterial community structure (Gomez et al., 2009; Lupwayi et al., 2007; Ratcliff et al., 2006). In this study, both the G1 and G2 levels of glyphosate addition significantly stimulated soil FDAse activity. FDAse activity can represent general metabolic activity, and it is a good indicator of soil life intensity and microbial activity (Perucci, 1992). This result indicates that both G1 and G2 levels of glyphosate addition can

Table 1
Effects of treatments on soil dissolved organic carbon during the 30 incubation days.

Treatments	Soil dissolved organic carbon (mg kg^{-1})					
	D0	D1	D3	D7	D14	D30
CK	55.61 ± 9.03abB	44.61 ± 2.36bE	62.40 ± 7.23aC	49.54 ± 5.29abCD	45.42 ± 3.82bC	51.35 ± 3.75abB
G1	41.44 ± 11.11bBC	61.90 ± 11.29aCD	53.93 ± 7.06abCD	51.02 ± 2.86abC	48.32 ± 7.65abC	47.07 ± 1.30bB
G2	48.19 ± 11.97bBC	102.19 ± 4.33aB	54.50 ± 7.79bCD	51.79 ± 3.34bC	64.51 ± 10.86bBC	41.05 ± 12.86bBC
M1G1	21.66 ± 3.05bD	46.66 ± 12.12aDE	60.95 ± 8.52aCD	22.87 ± 4.26bE	22.51 ± 7.78bD	22.94 ± 6.30bC
M1G2	28.10 ± 10.03dCD	73.78 ± 8.44aC	51.00 ± 1.57bD	46.17 ± 1.57cD	30.95 ± 17.65cdCD	35.42 ± 13.10cdBC
M2G1	57.44 ± 7.54bB	101.04 ± 32.49aABC	85.24 ± 5.59aB	93.77 ± 4.47aB	83.27 ± 8.30B	96.55 ± 12.59aA
M2G2	128.15 ± 16.35bA	125.92 ± 15.90bA	116.77 ± 4.91bA	161.71 ± 14.85aA	156.40 ± 34.18abA	101.04 ± 6.32cA

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

Table 2a
Effects of treatments on soil dissolved organic nitrogen during the 30 incubation days.

Treatments	Soil dissolved organic nitrogen (mg kg^{-1})					
	D0	D1	D3	D7	D14	D30
CK	8.77 ± 2.66bC	6.89 ± 2.34bC	14.53 ± 2.3aB	5.98 ± 1.85bcC	3.03 ± 1.39cC	7.65 ± 2.33bB
G1	4.85 ± 1.67bD	13.49 ± 2.18aB	13.62 ± 2.31aB	13.23 ± 4.07aAB	14.71 ± 4.54aAB	4.65 ± 1.16bBC
G2	4.02 ± 0.94cE	13.57 ± 4.32abB	9.68 ± 3.57bBC	9.29 ± 3.21bBC	19.26 ± 2.86aA	7.30 ± 2.21bB
M1G1	16.14 ± 1.77aB	7.53 ± 1.40cC	7.41 ± 0.69cC	12.60 ± 1.64bAB	14.92 ± 4.17aAB	9.97 ± 1.75bcAB
M1G2	21.88 ± 3.30aA	20.72 ± 4.33abAB	20.60 ± 3.21abA	17.99 ± 4.31abA	13.83 ± 3.03bAB	14.56 ± 3.33bA
M2G1	6.58 ± 0.17cD	8.96 ± 1.12bBC	13.70 ± 1.39aB	10.77 ± 2.02abB	9.28 ± 1.72bB	3.04 ± 1.16dC
M2G2	10.01 ± 2.58cC	25.18 ± 3.37aA	18.67 ± 4.50abAB	12.34 ± 4.66bcAB	17.30 ± 2.43bA	4.25 ± 2.01dBC

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

Table 2b
Effects of treatments on soil dissolved nitrate nitrogen during the 30 incubation days.

Treatments	Soil dissolved nitrate nitrogen (mg kg^{-1})					
	D0	D1	D3	D7	D14	D30
CK	28.86 ± 3.69abA	32.35 ± 0.95aA	24.21 ± 1.33bA	18.09 ± 3.53cAB	17.81 ± 4.46cB	16.39 ± 3.79cB
G1	19.24 ± 1.15aB	19.62 ± 3.19aBC	18.51 ± 2.51aBC	15.07 ± 3.33abB	11.10 ± 3.25bC	19.58 ± 2.66aAB
G2	17.29 ± 4.40abB	19.70 ± 3.54abBC	20.60 ± 1.97aB	19.70 ± 2.93abAB	8.60 ± 1.33cC	16.37 ± 0.68bB
M1G1	7.61 ± 0.87cC	23.00 ± 3.89aB	23.12 ± 2.34aAB	18.82 ± 2.77aAB	19.26 ± 3.28aB	14.76 ± 0.77bB
M1G2	16.84 ± 1.90cB	17.48 ± 0.75cC	21.52 ± 1.44bAB	20.84 ± 1.27bA	24.00 ± 0.75aA	11.49 ± 2.04dC
M2G1	19.85 ± 1.93aB	20.18 ± 2.32aBC	16.97 ± 1.51aC	17.09 ± 2.30aB	16.73 ± 2.35aB	16.44 ± 2.94aB
M2G2	23.60 ± 4.32aAB	22.77 ± 3.28aB	20.22 ± 3.63aBC	16.68 ± 2.10bB	11.17 ± 1.71cC	20.92 ± 1.47aA

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

Table 2c
Effects of treatments on soil dissolved ammonium nitrogen during the 30 incubation days.

Treatments	Soil dissolved ammonium nitrogen (mg kg^{-1})					
	D0	D1	D3	D7	D14	D30
CK	0.55 ± 0.03aB	0.44 ± 0.03bB	0.37 ± 0.06bcA	0.31 ± 0.11bcB	0.28 ± 0.09cBC	0.24 ± 0.04cA
G1	0.40 ± 0.15abBC	0.54 ± 0.12aAB	0.35 ± 0.09abA	0.28 ± 0.01cB	0.24 ± 0.12bcBC	0.28 ± 0.04bcA
G2	0.41 ± 0.01aC	0.54 ± 0.04aAB	0.29 ± 0.04bA	0.48 ± 0.16aAB	0.56 ± 0.14aA	0.23 ± 0.07bA
M1G1	0.41 ± 0.04bC	0.71 ± 0.19aA	0.31 ± 0.04cA	0.16 ± 0.04dC	0.21 ± 0.01dC	0.22 ± 0.02dA
M1G2	0.54 ± 0.12aBC	0.46 ± 0.19abAB	0.35 ± 0.03bA	0.26 ± 0.08bcBC	0.18 ± 0.03cC	0.25 ± 0.10bcA
M2G1	0.59 ± 0.07aB	0.46 ± 0.07aB	0.33 ± 0.05bA	0.50 ± 0.12aAB	0.31 ± 0.03bB	0.23 ± 0.11bA
M2G2	0.71 ± 0.03aA	0.68 ± 0.09abA	0.32 ± 0.11bA	0.67 ± 0.21abA	0.43 ± 0.17bAB	0.43 ± 0.25bA

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

dramatically promote general microbial metabolic activity. Glyphosate addition also temporarily stimulated PO activity, which is involved in the degradation of recalcitrant (phenolic) compounds by breaking C bonds in complex structures, such as tannin and lignin (Keuskamp et al., 2015). Glyphosate degradation involves enzymatic reactions that break either the C—P bond or the C—N bond by C—P lyase or glyphosate oxidoreductase and glyoxylic acid, respectively. This effect leads to the formation of sarcosine or amino-methyl phosphonic acid (AMPA) (Ternan et al., 1998). These compounds can act as a source of C, N and P for microorganisms to increase their activity and stimulate soil

Table 3a
Effects of treatments on soil dissolved organic phosphorus during the 30 incubation days.

Treatments	Soil dissolved organic phosphorus (mg kg^{-1})					
	D0	D1	D3	D7	D14	D30
CK	0.22 ± 0.03bE	0.18 ± 0.03bD	0.36 ± 0.10aE	0.40 ± 0.07aF	0.38 ± 0.04aE	0.23 ± 0.04bD
G1	0.82 ± 0.17bC	1.03 ± 0.19abC	1.13 ± 0.22abD	0.54 ± 0.05cE	0.93 ± 0.12bD	1.22 ± 0.14aA
G2	2.33 ± 0.63aB	2.05 ± 0.29aB	2.11 ± 0.25aC	2.36 ± 0.27aC	3.86 ± 0.69aB	0.07 ± 0.01bE
M1G1	0.44 ± 0.12cD	0.87 ± 0.20bC	1.44 ± 0.25aD	0.25 ± 0.05dG	0.41 ± 0.04cE	0.06 ± 0.01eE
M1G2	0.85 ± 0.15cC	0.94 ± 0.14cC	2.46 ± 0.36aBC	1.69 ± 0.25bD	0.88 ± 0.06cD	0.41 ± 0.50cBCD
M2G1	0.67 ± 0.07cC	1.29 ± 0.24bC	2.88 ± 0.11aB	3.11 ± 0.54aB	1.54 ± 0.40bC	0.42 ± 0.07dC
M2G2	3.92 ± 0.26cA	5.37 ± 0.42bA	5.69 ± 0.42bA	5.86 ± 0.68abA	6.62 ± 0.36aA	0.71 ± 0.05 dB

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

Table 3b
Effects of treatments on soil dissolved inorganic phosphorus during the 30 incubation days.

Treatments	Soil dissolved inorganic phosphorus (mg kg^{-1})					
	D0	D1	D3	D7	D14	D30
CK	0.13 ± 0.004aA	0.10 ± 0.003aB	0.09 ± 0.04abCD	0.13 ± 0.05aBC	0.15 ± 0.05aCD	0.08 ± 0.01bA
G1	0.07 ± 0.02bB	0.19 ± 0.06aA	0.14 ± 0.02aC	0.19 ± 0.04aB	0.17 ± 0.08aBCD	0.07 ± 0.03bA
G2	0.12 ± 0.03bAB	0.06 ± 0.01cC	0.08 ± 0.03bcD	0.11 ± 0.03bBC	0.29 ± 0.07aB	0.07 ± 0.04bcA
M1G1	0.08 ± 0.01aB	0.07 ± 0.01aC	0.08 ± 0.02aD	0.07 ± 0.01aC	0.07 ± 0.01aE	0.07 ± 0.01aA
M1G2	0.08 ± 0.002bB	0.34 ± 0.19aA	0.09 ± 0.03bCD	0.09 ± 0.02bC	0.06 ± 0.06bDE	0.11 ± 0.06abA
M2G1	0.26 ± 0.13abA	0.19 ± 0.08abA	0.25 ± 0.09aB	0.21 ± 0.09abB	0.27 ± 0.07aB	0.11 ± 0.05bA
M2G2	0.23 ± 0.08bA	0.15 ± 0.04bcA	0.45 ± 0.09aA	0.54 ± 0.22aA	0.71 ± 0.21aA	0.10 ± 0.05cA

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

enzyme activity (Krzysko-Lupicka and Orlik, 1997; Panettieri et al., 2013; Ternan et al., 1998).

The results suggested that the positive effect of glyphosate addition on the soil DOC, DON and NH_4^+ contents was transient. Glyphosate itself is a water soluble and widely available carbon, and the application of glyphosate can slightly increase the DOC content (Panettieri et al., 2013). DOM dynamics depend on the imbalance between production and in situ mineralization (Gogo et al., 2014; Schimel and Weintraub, 2003). A previous study showed that the measured dynamics of extractable ^{14}C-DOC essentially paralleled that of pesticides in soil (Pagel et al., 2016). Thus, glyphosate degradation plays a vital role in altering DOM dynamics. Glyphosate and its metabolite remained mainly water-soluble after glyphosate was applied to soil, and a lower proportion of the herbicide became non-extractable in bare soil over time (Cassigneul et al., 2016), leading to increased DON. Finally, these organic nitrogen compounds are decomposed into CO_2 and NH_4^+ (Borggaard and Gimsing, 2008), contributing to the accumulation of NH_4^+ in soil. The soil glyphosate degradation period occurs over a relatively short time (Tejada, 2009; Veiga et al., 2001). A previous study revealed that the degradation process of glyphosate followed a single first-order kinetic model, and the average half-life of glyphosate in this experiment was 32.8 ± 2.6 days (Yang et al., 2018), which is distinct from that in clay loam soil (3.5 days), in sand (16.9 days) and clay (110 days) top soil (Al-Rajab and Schiavon, 2010; Bergstrom et al., 2011). The fast microbial glyphosate degradation process resulted in the beneficial effect of glyphosate on soil DOC, DON and NH_4^+ being temporary.

The G2 level of glyphosate addition significantly increased soil DOP and PO_4^{3-} contents. The positive effect of glyphosate on DOP was stronger and lasted longer than on DOC, DON, NH_4^+ and PO_4^{3-}. The accumulation of soil DOP after glyphosate addition occurs because the glyphosate itself is a P-containing amino acid, and its decomposition by-product, AMPA, is also an organic P compound. However, the DOP content in G2 treatment sharply decreased between day 14 and 30, indicating that excessive glyphosate addition has a risk of causing DOP deficiency after a certain time. Kashem et al. (2004a) reported that the content of soil extractable P significantly decreased from 1 week to 32 weeks after P addition, and soil extractable P content decreased relative to control soil after 16 weeks. P extractability depends not only on the total amount of added P, but also on the characteristics of the P

Table 4a
Effects of treatments on soil phenol oxidase activities during the 30 incubation days.

Treatments	Soil phenol oxidase activities (μmol h^{-1} g^{-1} soil)					
	D0	D1	D3	D7	D14	D30
CK	5.15 ± 1.00cC	6.88 ± 0.44bcAB	7.28 ± 0.27bAB	5.19 ± 0.78cB	5.94 ± 0.54cB	7.91 ± 0.14aA
G1	6.27 ± 1.01bcAB	5.00 ± 0.96cD	7.35 ± 0.25aA	6.66 ± 0.64bA	5.96 ± 0.75bcB	6.78 ± 0.28bB
G2	5.46 ± 0.56bC	7.30 ± 0.11aA	7.60 ± 0.25aA	4.78 ± 0.31bB	7.99 ± 0.33aA	6.91 ± 0.96abAB
M1G1	6.43 ± 0.20aA	6.25 ± 0.07ab	6.39 ± 0.18aC	6.03 ± 0.16bAB	6.23 ± 0.20abB	6.06 ± 0.12bC
M1G2	6.17 ± 0.19aAB	6.51 ± 0.28aBC	6.20 ± 0.14aC	6.07 ± 0.13AB	6.24 ± 0.41aB	5.74 ± 0.09bD
M2G1	6.11 ± 0.07abB	6.17 ± 0.10abC	6.44 ± 0.33aBC	6.09 ± 0.07abA	6.09 ± 0.07abB	5.99 ± 0.09bC
M2G2	6.43 ± 0.15aA	6.60 ± 0.16aB	6.82 ± 0.20aB	6.36 ± 0.06aA	7.08 ± 1.14aAB	6.16 ± 0.20aC

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

Table 4b
Effects of treatments on soil phenol oxidase activities during the 30 incubation days.

Treatments	Soil FDAse activities (mg Kg^{-1} soil h^{-1})					
	D0	D1	D3	D7	D14	D30
CK	10.93 ± 2.83aBC	8.75 ± 0.49aB	6.06 ± 1.85bD	8.94 ± 2.24abBC	8.54 ± 1.00aC	6.52 ± 0.94bD
G1	16.23 ± 2.38aA	13.71 ± 3.17abA	9.49 ± 2.22bCD	10.74 ± 1.60bBC	6.60 ± 0.32cD	9.45 ± 2.01bBC
G2	8.04 ± 1.75aC	6.97 ± 1.27abC	9.35 ± 2.27aCD	9.14 ± 2.03aBC	6.02 ± 0.43bD	9.15 ± 0.19aC
M1G1	15.58 ± 0.42aA	10.29 ± 0.98bAB	12.12 ± 1.35bB	12.19 ± 1.02bAB	14.94 ± 0.30aA	10.37 ± 0.59bB
M1G2	15.10 ± 0.65aA	9.76 ± 0.77 dB	10.16 ± 0.51dC	11.37 ± 0.30cB	12.90 ± 0.80bB	11.18 ± 0.44cB
M2G1	14.90 ± 1.24aAB	9.30 ± 0.51bB	15.10 ± 1.10aA	13.26 ± 0.54aA	12.95 ± 0.47aB	15.31 ± 2.02aA
M2G2	15.64 ± 0.64aA	8.77 ± 1.27cBC	16.50 ± 0.44aA	8.73 ± 0.81cC	12.04 ± 1.40bB	16.21 ± 0.85aA

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

source and soil type (Kashem et al., 2004a; Kashem et al., 2004b). P is an essential nutrient in soil, and P addition stimulated soil enzyme activities, including phosphatases and dehydrogenases, which might lead to the promotion of the net mineralization of P (Hu et al., 2014; Wang et al., 2013a; Zhang et al., 2016). In this study, glyphosate addition increased microbial hydrolytic activity (Table 4b) and stimulated the mineralization of DOP, resulting in the deficiency of DOP at day 30. Additionally, the positive effect of glyphosate addition on PO_4^{3-} was considerably weaker than on DOP, and this effect was transient and primarily attributed to glyphosate degradation that formed phosphate.

The G2 level of glyphosate addition significantly promoted the accumulation of tryptophan-like material. Tryptophan-like fluorescence indicates more highly degraded peptides (Mayer et al., 1999; Yamashita and Tanoue, 2003; Yamashita and Tanoue, 2004), and tryptophan-like fluorescence is considered as a useful indicator of biodegradable DOC (Fellman et al., 2008). Although glyphosate is thought to inhibit protein synthesis via the shikimic acid pathway in bacteria and fungi (Bentley, 1990), no loss of amino acid content in soil DOM was observed after glyphosate addition. Moreover, the positive effects of glyphosate addition on high-molecular-weight humic-like material and fulvic acid are temporary. Humic substances (HS), which are recalcitrant to biological degradation, are the major constitutes of soil organic matter. According to difference in solubility, HS is classified into three groups: humic acid, fulvic acid (FA), and non-soluble humin. Humic acid and FA provide an important source of macro- and micronutrients for plants and microorganisms (Schulten and Schnitzer, 1997; van Hees et al., 2005), play an important role in the acid–base buffering capacity of soils (Schnitzer, 2000), contribute largely to the retention and release, biological availability, and mobility of metal ions (Hayes and Malcom, 2001; Plaza et al., 2002), affect soil biological activity (Garcia and Hernandez, 1997), and can bind mineral particles together promoting a good soil structure, thereby improving aeration and moisture retention (Hayes and Clapp, 2001; Piccolo and Mbagwu, 1994). Additionally, the FA content of soil affects the transport and bioavailability of environmental contaminants, acting as carrying agents and complexing media (Chirenje et al., 2002; Plaza et al., 2005). Therefore, glyphosate addition had little detrimental effect on soil quality and environmental pollution.

4.2. Influence of microplastics addition on glyphosate effect on C, N and P pools in soil DOM and soil enzyme activities

Previous studies found that plastic mulching film residues reduced soil C and N biomass and FDAse activity (Wang et al., 2013b). This finding was primarily attributed to the multiple heavy metals and organic pollutants released by plastic mulching film residues (He et al., 2015; Moreno and Moreno, 2008; Teng et al., 2014; Wang et al., 2016). In this study, the positive effect of glyphosate and microplastics co-addition on FDAse activity was considerably stronger and longer lasting than that of glyphosate addition. Zhang et al. (2015) suggested that microplastics affect porosity and air circulation. Porosity and specific surface area are considered to be positively correlated with soil microbial activities (Arthur et al., 2012; Girvan et al., 2003; Naveed et al., 2016). Previous studies showed that microplastics addition significantly stimulated FDAse activity and microbial respiration (Liu et al., 2017; Yang et al., 2018). In comparison with our previous study (Liu et al., 2017), no significant differences were observed between FDAse activity after glyphosate and microplastics co-addition and microplastics addition. Therefore, microplastics plays a major role in the positive effect after glyphosate and microplastics co-addition. Small plastic particles are prone to absorbing and accumulating pesticides and other biocides in the soil (Barnes et al., 2009; Ramos et al., 2015), and may influence the behaviours of existing compounds in the soil matrix (Hueffer and Hofmann, 2016). Nevertheless, microplastics cannot absorb glyphosate, and glyphosate and microplastics only minimally interact with each other (Yang et al., 2018), so the interaction between glyphosate and microplastics has a negligible effect on FDAse activity. Additionally, FDAse activities in M2G1 and M2G2 at day 30 were significantly higher than in M1G1 and M1G2 at earlier timepoints, showing that the accumulation of microplastics in soil promotes microbial metabolic activity

Table 5a
Effects of treatments on fluorescent intensity of component 1 during the 30 incubation days.

Treatments	Fluorescent intensity of component 1 (RU)					
	D0	D1	D3	D7	D14	D30
CK	74.3 ± 13.2c	100.9 ± 33.1bcC	139.5 ± 1.6bD	277.1 ± 28.7aV	151.5 ± 35.1bA	25.3 ± 5.5dF
G1	498.0 ± 47.4bB	168.6 ± 33.1eB	237.3 ± 30.6 dB	307.9 ± 39.6cB	154.8 ± 13.8eA	1544.0 ± 48.0aA
G2	1368.2 ± 164.2aA	159.8 ± 15.6eB	463.7 ± 24.9cA	192.0 ± 3.4dC	163.4 ± 7.4eA	1024.7 ± 87.3bB
M1G1	162.4 ± 15.3bC	42.6 ± 10.3cD	410.2 ± 37.4aA	478.1 ± 51.8aA	22.5 ± 15.1cC	124.5 ± 31.7bE
M1G2	33.5 ± 4.5eD	215.8 ± 11.6cA	174.5 ± 13.2dC	293.8 ± 18.8bB	188.0 ± 41.0cdA	374.5 ± 15.7aC
M2G1	133.9 ± 16.0cC	71.2 ± 10.6dC	157.3 ± 2.8bC	74.4 ± 8.9dD	120.5 ± 27.5cA	208.2 ± 16.1aD
M2G2	139.8 ± 11.4cC	177.1 ± 6.2bB	118.2 ± 16.4cdE	94.9 ± 12.2dD	88.6 ± 8.9 dB	889.6 ± 89.8aB

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

Table 5b
Effects of treatments on fluorescent intensity of component 2 during the 30 incubation days.

Treatments	Fluorescent intensity of component 2 (RU)					
	D0	D1	D3	D7	D14	D30
CK	115.0 ± 13.6eC	1430.9 ± 27.9bC	1253.1 ± 153.6cG	615.3 ± 74.7dD	1641.2 ± 98.4aA	0
G1	474.3 ± 100.2 dB	462.9 ± 33.7dE	2439.8 ± 45.4aC	1333.6 ± 121.6bB	867.9 ± 106.7cCD	0
G2	506.9 ± 88.3 dB	604.9 ± 31.8dD	2884.8 ± 87.1aA	1106.9 ± 11.3cC	1469.7 ± 36.1bB	0
M1G1	0.5 ± 0.8dD	98.4 ± 1.8cF	2036.2 ± 86.1aF	235.5 ± 61.5bE	157.3 ± 41.4bE	0
M1G2	0	0	876.3 ± 70.5H	2599.2 ± 259.3A	0	0
M2G1	543.0 ± 70.5 dB	1884.8 ± 62.0bB	2225.7 ± 35.5aD	283.8 ± 30.3fE	748.8 ± 42.3cD	0
M2G2	2413.5 ± 50.5bA	2189.6 ± 82.1cA	2671.0 ± 108.7aB	597.7 ± 31.3eD	900.4 ± 59.7dC	119.1 ± 17.5f

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

and hydrolytic activity.

Compared with glyphosate addition, interaction between glyphosate and microplastics at the M2 level significantly promoted the accumulation of DOC. The rise in FDAse after the co-addition of microplastics and glyphosate indicates an enhanced hydrolytic activity on soil organic matter or DOM. An increase in soil organic matter hydrolysis can lead to an increased DOC, while a rise in DOM hydrolysis changes the DOC content, as dissolved compounds remained dissolved after hydrolysis (Delarue et al., 2014). However, the positive effect of co-addition of glyphosate and microplastics at the M2 level on DON content was lower than the positive effect of glyphosate addition and that of microplastics addition (Liu et al., 2017). This beneficial effect lasted < 30 days. Moreover, the co-addition of glyphosate and microplastics at the M2 level increased inorganic nitrogen, especially in M2G2. The increase in FDAse activity after the co-addition of glyphosate and high content microplastics enhanced microbial metabolic activity that resulted in increased mineralization of DON and accumulation of inorganic nitrogen. The co-addition of glyphosate and microplastics decreased the PO activity at day 30, leading to less recalcitrant organic matter (e.g., cellulose and lignin) being decomposed into dissolved organic matter. The interaction between glyphosate and the M2 level of microplastics accelerates soil P cycling and is beneficial for P accumulation, as the contents of DOP and PO_4^{3-} in treatments of glyphosate and the M2 level of microplastics co-addition were significantly higher than that of the glyphosate addition and microplastics addition (result published in Liu et al. (2017)). A previous study revealed that microplastics only minimally interact with glyphosate, and microplastics addition does not affect glyphosate degradation (Yang et al., 2018). Glyphosate can occur as an organophosphate compound that can bind to the soil with ligand exchange through the phosphonic acid moiety (Al-Rajab et al., 2008). Microplastics, such as the polypropylene powder used in this study, are resistant to degradation by almost all organic solvents and strong oxidants (Rillig, 2012; Yang et al., 2018). However, the significantly increased FDAse and PO activities in M2G1 and M2G2 within the first 14 days indicated an enhanced hydrolytic activity and decomposition of a recalcitrant substance, leading to the decomposition of more high-molecular-weight insoluble substances' into soluble substances.

Unlike the co-addition of glyphosate and the M2 level of microplastics, M1G1 significantly decreased the DOC content relative to CK at day 30. M1G1 and M1G2 decreased the PO_4^{3-} content relative to CK between day 7 and 14, and M1G1 decreased the DOP content relative to CK at day 30. These results suggest that interaction between glyphosate and the M1 level of microplastics decreased the contents of DOC, DOP and PO_4^{3-} relative to the glyphosate addition, with the risk of causing DOC deficiency. On the one hand, significantly lower FDAse and PO activities in M1G1 and M1G2 were observed relative to those in M2G1 and M2G2, resulting in fewer high-molecular-weight insoluble substances being decomposed into soluble substances. On the other hand, the observed DOM concentration is the net result of processes that release DOM, such as desorption from the solid phase, and processes that remove DOM, such as adsorption. The surface area of minerals is a key factor influencing the adsorption capacity of soil DOM (Gu et al., 1994; Mayer 1994a, b). Microplastics can alter soil physical properties, and influence porosity and air circulation (Rillig, 2012; Zhang et al., 2015), which might promote the sorption process of DOM and decrease the DOC and DOP contents.

In comparison to glyphosate addition, the co-addition of glyphosate and the M2 level of microplastics accelerated the decomposition of high-molecular-weight humic-like material and promoted the accumulation of low-molecular-weight humic-like material. Moreover, co-addition of glyphosate and the M2 level of microplastics increased the fulvic acid content compared with the glyphosate addition and microplastics addition (result published in Liu et al. (2017)). Therefore, there is no detrimental effect of the interaction between glyphosate and the M2 level of microplastics on soil structure and nutrient availability, as fulvic acid and humic-like material are vital to soil structure, stability, water-holding capacity, and nutrient availability (Garcia and Hernandez, 1997; Hayes and Clapp, 2001; Schnitzer, 2000; van Hees et al., 2005). However, co-addition of glyphosate and M2 level of

Table 5c
Effects of treatments on fluorescent intensity of component 3 during the 30 incubation days.

Treatments	Fluorescent intensity of component 3 (RU)					
	D0	D1	D3	D7	D14	D30
CK	173.5 ± 9.9aA	159.2 ± 18.2abAB	141.8 ± 21.5bA	62.5 ± 13.7dBC	94.1 ± 16.9cA	96.6 ± 12.3cDE
G1	144.4 ± 18.0bAB	172.5 ± 18.2aA	105.5 ± 6.9cB	89.4 ± 6.9dA	79.1 ± 4.9dAB	116.6 ± 8.6cD
G2	104.7 ± 25.1cB	164.5 ± 5.7aA	83.7 ± 1.7dC	82.8 ± 3.0dA	86.6 ± 6.6dA	133.5 ± 2.4bC
M1G1	24.9 ± 0.5cC	112.5 ± 3.1aC	109.6 ± 5.5aB	56.0 ± 13.7bC	53.9 ± 20.6bB	54.4 ± 8.9bF
M1G2	89.2 ± 12.8bB	101.9 ± 6.6abD	108.2 ± 4.4aB	89.6 ± 19.2bAB	65.3 ± 3.3cB	71.7 ± 7.8cE
M2G1	192.4 ± 43.8aA	121.3 ± 12.2bC	67.0 ± 16.4cC	56.2 ± 9.2dC	70.3 ± 4.2cB	160.5 ± 17.6aB
M2G2	187.3 ± 19.6bA	149.7 ± 5.8cB	132.5 ± 8.4dA	90.2 ± 7.6eA	94.0 ± 13.1eA	236.6 ± 26.8aA

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

Table 5d
Effects of treatments on fluorescent intensity of component 4 during the 30 incubation days.

Treatments	Fluorescent intensity of component 4 (RU)					
	D0	D1	D3	D7	D14	D30
CK	12.3 ± 10.8dD	220.6 ± 26.7aC	155.6 ± 6.6bE	108.7 ± 11.7cD	214.3 ± 16.7aD	0
G1	17.0 ± 6.1cD	15.9 ± 5.2cE	572.5 ± 31.8aD	593.3 ± 33.3aB	356.8 ± 48.3bC	0
G2	211.1 ± 26.0cB	43.3 ± 3.1eD	1196.5 ± 21.5aB	162.1 ± 17.1dC	548.6 ± 50.2bA	0
M1G1	0	0	1025.3 ± 63.0aC	39.2 ± 15.9bE	12.6 ± 21.8bE	0
M1G2	0	0	150.7 ± 31.2E	720.9 ± 21.4A	0	0
M2G1	71.5 ± 10.7eC	834.2 ± 24.3bB	1252.4 ± 79.9aB	116.1 ± 22.7dD	473.2 ± 6.1cB	0
M2G2	839.7 ± 37.0bA	1519.2 ± 96.2aA	1959.6 ± 395.3aA	188.1 ± 7.8dC	423.2 ± 40.5cC	71.6 ± 11.3e

Different lowercase letters within the same row mean significant differences in each individual treatment during the incubation days; different capital letters within the same column mean significant differences among treatments in each incubation day ($p < 0.05$).

microplastic decreased the content of tryptophan-like material relative to glyphosate addition. This finding implied that a high microplastics content has a risk of environmental pollution and decreasing biodegradable DOC, since tryptophan-like fluorescence is a useful predictor of biodegradable DOC (Fellman et al., 2008). Additionally, the co-addition of glyphosate and the M1 level of microplastics significantly decreased the content of humic-like material and fulvic acid, and this effect was transient, which might affect soil structure and nutrient availability.

5. Conclusions

This study characterized the changes in the quantity and quality of soil DOM and the activities of soil enzymes at two levels of glyphosate addition and four levels of glyphosate and microplastics addition. Glyphosate application temporarily stimulated soil FDAse and PO activities, since its decomposition products provide sources of C, N and P for microorganisms. The stimulated enzyme activity increased DOC, DON, NH_4^+, DOP, and PO_4^{3-} within the first 14 days. However, high glyphosate application has a risk of decreasing DOP at day 30. The interaction between glyphosate and a high content of microplastics increased the soil enzyme activity and contents of DOC, DOP, tryptophan-like material, low-molecular-weight humic-like material and fulvic acid, but it negatively affected DON. Compared with glyphosate addition, the co-addition of glyphosate and a low content of microplastics negatively affected DOC, DOP, PO_4^{3-}, humic-like material and fulvic acid, which has a risk of bioavailable C and P loss, but it benefits soil N cycling and DON accumulation. Overall, more positive effects of the glyphosate addition and the interaction between glyphosate and microplastics on soil microbial activity and nutrient availability in DOM were observed than negative effects. These results provide a preliminary understanding of the effect of glyphosate on soil enzyme activity and DOM dynamics, as well as the effect of interactions between glyphosate and microplastics on soil nutrient availability. Further research is warranted to determine the effect of microplastics on glyphosate kinetic degradation and nutrient dynamics in soil-plant systems.

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