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# Response of soil dissolved organic matter to microplastic addition in Chinese loess soil



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## HIGHLIGHTS

• Microplastic addition stimulated soil activity of fluorescein diacetate hydrolase (FDAse) in soil.

• The lower level of microplastic addition had a negligible effect on the nutrient contents in DOM solution at day 30.

• The higher level of microplastic addition significantly increased the nutrient contents in DOM solution.

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## ABSTRACT

Plastic debris is accumulating in agricultural land due to the increased use of plastic mulches, which is causing serious environmental problems, especially for biochemical and physical properties of the soil. Dissolved organic matter (DOM) plays a central role in driving soil biogeochemistry, but little information is available on the effects of plastic residues, especially microplastic, on soil DOM. We conducted a soil-incubation experiment in a climate-controlled chamber with three levels of microplastic added to loess soil collected from the Loess Plateau in China: 0% (control, CK), 7% (M1) and 28% (M2) (w/w). We analysed the soil contents of dissolved organic carbon (DOC), dissolved organic nitrogen (DON), NH4,  $NO_{\overline{3}}$ , dissolved organic phosphorus (DOP), and  $PO_{\overline{4}}^{-}$  and the activities of fluorescein diacetate hydrolase (FDAse) and phenol oxidase. The higher level of microplastic addition significantly increased the nutrient contents of the DOM solution. The lower level of addition had no significant effect on the DOM solution during the first seven days, but the rate of DOM decomposition decreased in M1 between days 7 and 30, which increased the nutrient contents. The microplastic facilitated the accumulation of high-molecularweight humic-like material between days 7 and 30. The DOM solutions were mainly comprised of highmolecular-weight humic-like material in CK and M1 and of high-molecular-weight humic-like material and tyrosine-like material in M2. The Microplastic stimulated the activities of both enzymes. Microplastic addition thus stimulated enzymatic activity, activated pools of organic C, N, and P, and was beneficial for the accumulation of dissolved organic C, N and P.

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## 1. Introduction

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<sup>1</sup> The equal author contribution.

http://dx.doi.org/10.1016/j.chemosphere.2017.07.064 0045-6535/© 2017 Elsevier Ltd. All rights reserved. The increasing use of plastic has increased the amount of plastic debris in the environment, especially in the oceans. More than 240 million tonnes of plastic are used every year (Thompson et al., 2009), contributing to 11.7–25% of the municipal waste in Europe and the USA. Only 1–2% of this plastic is recycled (Shent et al., 1999;



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Panda et al., 2010). Plastic debris is accumulating in the environment because of its durability and the limitation of recycling technology (Barnes et al., 2009; Rillig, 2012). Microplastic, particles <5 mm in size (Claessens et al., 2011), have been studied in the marine environment and on shorelines, but few studies have focused on the microplastic in soil and terrestrial systems (Rillig, 2012: do Sul and Costa. 2014: Van Cauwenberghe et al., 2015). Microplastic can enter the soil either as primary microplastic from industrial abrasives and cosmetic products by sludge application (Cole et al., 2011) or secondary microplastic from the environmental degradation of plastic mulch. Agricultural sites and landfills in Europe contain 1000-4000 microplastic particles per kg of sludge dry mass (Zubris and Richards, 2005; Barnes et al., 2009). The use of plastic films as agricultural mulches increased in China, especially in northern regions, nearly four-fold from 1991 to 2011, from 0.32 to 1.25 million tonnes which has contributed to the accumulation of microplastic in farmland (Yearbook, 2012; Wang et al., 2013; Liu et al., 2014). Microplastic can be ingested by fauna due to their small size and can thus accumulate in the food chain (Besseling et al., 2013; Huerta Lwanga et al., 2016b). Some studies have identified interactions between microplastic and the chemical pollutants they absorb on their surfaces (Ivar do Sul and Costa, 2014). Microplastic can also alter the physical properties of soil and can accumulate in soil, reaching levels that can affect soil function and biodiversity (Rillig, 2012).

Dissolved organic matter (DOM) plays an important role in numerous physical, chemical, and biological processes in soil (Kalbitz et al., 2000), including the cycling of soil organic carbon (C) and the transport of nutrients such as nitrogen and phosphorus (Kalbitz et al., 2003b). DOM represents <0.25% of the total soil organic matter but facilitates the solubility and mobility of metals and organic compounds (Kalbitz et al., 1997; Temminghoff et al., 1997) and thus increases the transport of pollutants (Kalbitz et al., 2000). DOM is a complex mixture of various labile and recalcitrant organic substances (Michel et al., 2006) and is a substrate and the most important C source for microorganisms (Marschner and Kalbitz, 2003; DeForest et al., 2004a, 2004b). Soil dissolved organic C (DOC) is a more sensitive indicator of changes in soil quality changes than total organic C (TOC), because short-term changes in TOC are not easily detected (Purakayastha et al., 2008; Gong et al., 2009; Li et al., 2016).

The accumulation of microplastic in soil can affect microbial activity, attributed to absorbed harmful contaminants (Rillig, 2012), earthworm activity (Huerta Lwanga et al., 2016b, 2017) and soil physical properties, such as soil porosity and aggregate structure (Ladd et al., 1993; Rillig, 2012; Zhang et al., 2015). The activities of soil enzymes represent microbial activities and the availability of substrates for microorganism uptake. Soil enzymes with high catalytic capacities are produced by soil microorganisms and are the main medium controlling the cycling of soil nutrients such as C, N, and P (Dick et al., 1994; Allison and Jastrow, 2006; Trasar-Cepeda et al., 2008). Phenol oxidase (PO) is involved in the degradation of phenolic compounds which are recalcitrant DOMs and decreases the DOM biodegradability (Marschner and Kalbitz, 2003). PO activity is negatively correlated with soil humification (Li et al., 2015). Fluorescein diacetate hydrolase (FDAse) can represent overall microbial metabolic activity and is an effective indicator of short-term changes of soil quality (Muscolo et al., 2014, 2015).

So far the effect of microplastic on soil fertility and microbial activity were still not clear, although researches demonstrated that plastic-film residues can decrease soil porosity, air circulation, microbial biomass and microbial activity and can probably affect soil fertility (Moreno and Moreno, 2008; Kasirajan and Ngouajio, 2012). A detailed study of the effects of microplastic on the dynamics of soil DOM and enzymatic activities is needed for

minimizing environmental risks and determining the sustainability of farming practices. We used three-dimensional excitation-emission matrix (EEM) fluorescence spectroscopy, a rapid, sensitive, selective, and reagent-free technique for fingerprinting organic matter in terrestrial ecosystems, to study the composition of soil DOM (Chen et al., 2003; Matilainen et al., 2011).

Our specific aims were to: (1) determine the effect of microplastic on the quantities and composition of soil DOM, (2) determine the effect of microplastic on the dynamics of soil enzymatic activity, and (3) clarify the relationships between soil DOM and enzymatic activity.

#### 2. Materials and methods

#### 2.1. Experimental design

This experiment was conducted in the climate-controlled chamber (AGC-Doo3N, Hangzhou, China) at the Institute of Soil and Water Conservation, Chinese Academy of Sciences, Yangling, China. The soil used in this experiment was collected in Ansai county on the Loess Plateau, and soil properties can be seen in Table 1. The soil is mainly composed of Cultivated loessial soils (calcaric cambisols, FAO) developed on wind-deposited loess parental material and is characterised by the parental material, the absence of bedding, a loose silty texture, macroporosity, and wetness-induced collapsibility. Two hundred grams were incubated in PVC pots 10 cm in diameter and 9 cm in height. Our experiment contained three treatments: 1) CK. no microplastic added to the soil: 2) M1.14 g of microplastic added to the soil (7% w/ w); 3) M2, 56 g microplastic added to the soil (28% w/w). The microplastic used in this experiment is made of polypropylene (Youngling-TECH company, Beijing, China). The density of Microplastic is 0.91 g/cm<sup>3</sup>, and its bending strength is 200 kg/cm<sup>2</sup>. The particle size of microplastic is below 180 µm. The soils were slightly compacted using a small manual soil compactor. The soil compactor was set to fall 10 times by gravity at the pot height to guarantee the same compaction for all samples. The microplastic contents were chosen based on the research of Huerta Lwanga et al. (2016a) but simulating the hotspots of plastic debris in the field. Soil moisture was maintained at 60% of field capacity throughout the experiment. Each treatment had three replicates. The pots were incubated in the light at 28 °C (relative humidity of 80%, 300  $\mu$ (photons)  $m^{-2} s^{-1}$ ). The soil was sampled from each pot after 0, 1, 3, 7, 14, and 30 days, so the experiment had a total of 54 pots: 3 treatments  $\times$  3 replicates  $\times$  6 days. The soil samples were passed through a 2 mm sieve, and then one subsample was stored at -80 °C for analysing soil enzymatic activities, and another subsample was stored at 4 °C for measuring DOM chemical properties.

#### 2.2. Analysis of soil DOM concentration and composition

DOM was extracted by adding 120 mL of distilled water to 40 g subsamples of homogenised soil (1:3 soil:water, w/w) as described by Kalbitz et al. (2003a, 2013b). All extracts were centrifuged at 4000 rpm for 10 min, and the supernatants were filtered through pre-rinsed 0.45 µm cellulose-acetate membranes (Schleicher & Schuell). The filtered solutions were stored frozen until analysis. Total dissolved N (TDN), DOC,  $NH_4^+$ ,  $NO_3^-$ , total dissolved P (TDP), and PO<sub>4</sub><sup>3-</sup> contents were measured in all samples using 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 alkaline persulfate digestion-UV spectrophotometric method (Doyle et al., 2004). TDP contents molybdate were measured using the ammonium

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