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Size and concentration-dependent effects of polyethylene microplastics on soil chemistry in a microcosm study

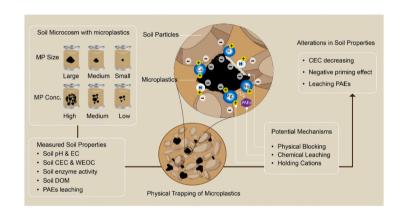
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HIGHLIGHTS

- Polyethylene MPs significantly altered soil CEC and DOM.
- Smaller polyethylene MPs caused more pronounced effects on soil CEC and DOM.
- Polyethylene MPs leached phthalate acid esters, raising concerns about soil health.
- Soil pH, EC, and enzyme activities showed no significant changes.
- No clear dose-response was observed for soil properties at field-relevant and elevated MP concentrations.

GRAPHICAL ABSTRACT



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ABSTRACT

The continuous use of plastics is expected to increase microplastic (MP) contamination in soils, raising concerns about impacts on soil ecosystems and crop productivity. This work investigated the effects of different sizes and concentrations of polyethylene microplastics (PE-MPs) on soil properties in a controlled microcosm experiment. Microplastics of three sizes (300–600, 600–2000, and 2000–5000 μm) were tested at three concentrations (0.02 %, 0.1 %, and 1 % by weight). Significant changes were observed in soil cation exchange capacity (CEC) and dissolved organic matter (DOM), accompanied by the leaching of phthalate acid esters (PAEs) from PE-MPs, with concentrations reaching up to 0.2 mg kg $^{-1}$. In contrast, soil pH, electrical conductivity (EC), water-extractable organic carbon (WEOC), and enzyme activity were not significantly affected. In particular, the smallest PE-MPs caused a 12.9 % reduction in soil CEC and a negative priming effect was observed in soil DOM. Although no clear dose–response relationship was observed, the findings suggest that MP-induced changes in soil chemistry are driven by both size-dependent surface interactions and complex soil matrix dynamics. These results demonstrate that PE-MPs can disrupt essential soil functions related to nutrient retention, organic matter

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dynamics, and pollutant transport, indicating broader impacts on soil health. As smaller MPs continue to accumulate, further research is needed to assess their long-term effects under varied environmental conditions and to inform effective mitigation strategies in agroecosystems.

Synopsis: Polyethylene microplastics altered key soil chemical properties, with smaller sizes and higher concentrations leading to greater changes in cation exchange capacity, dissolved organic matter, and diethyl phthalate release, raising concerns for long-term soil health.

1. Introduction

The agricultural industry uses plastic products for irrigation, protective housing, mulch film, seedling trays and containers. Over time, these plastics degrade and fragment into microplastics (MPs), leading to MP contamination in agricultural soils. Polyethylene (PE), including low-density polyethylene (LDPE), is the main plastic type used for drip irrigation pipes and plastic mulch films, which typically have only a oneyear lifespan. Studies show that farmlands using plastic mulch film systems contained, on average, more than twice the concentration of MPs compared to non-mulched farmlands in the agricultural region surrounding Hangzhou Bay, China. [1] Long-term use compounds the problem: in Xinjiang, China, under mulched drip-irrigated cotton farming, residual plastic film levels reached 121.85-352.38 kg ha⁻¹, depending on years of mulching (5-19 years). [2] These residues also migrate within the soil under drip irrigation, with PE-MPs detected up to 30 cm deep. Given the widespread use of PE in agriculture and its environmental persistence, understanding how PE-MPs influence soil health is critical for assessing risks to ecosystem functions and agricultural productivity.

The impacts of MPs on soil chemical properties have been comprehensively evaluated in recent reviews. These includes three major categories of soil chemical parameters affected by MPs: (i) pH, (ii) soil nutrient content, and (iii) soil enzyme activities. [3] Additionally, micro- and nanoplastics can act as vectors for potentially harmful substances such as plastic additives, antibiotics, and heavy metals, which may further influence soil chemistry. Other key soil chemical parameters impacted by MPs include electrical conductivity (EC), soil organic carbon (SOC), nutrient availability, and heavy metal mobility. [4,5]

While these reviews consistently emphasize the multifaceted effects of MPs on soil chemistry, they differ in scope and methodological approaches. Some focus on a broad biochemical spectrum including enzyme activities, while others provide more detailed evaluation of physicochemical parameters and contaminant dynamics. [3–5] However, all reviews highlight methodological limitations such as heterogeneity in MP characterization (size, polymer type, and concentration), experimental conditions, and soil types assessed, which complicate direct comparisons and synthesis of findings. This underscores the need for standardized methodologies and more comprehensive assessments encompassing multiple soil chemical properties simultaneously.

Individual experimental studies often examine the effect of MPs on single soil chemical properties or a limited subset of parameters. For example, some studies have focused solely on MP-driven alterations of specific soil parameters, such as pH, often examining how factors like polymer type, particle shape, or exposure time influence these changes. [6] Others have investigated effects on nutrient cycling under controlled conditions or in particular soil types. [7] However, these studies typically do not assess comprehensive changes across multiple soil chemical properties simultaneously, limiting our understanding of the broader impacts of MPs on soil chemistry. Moreover, these studies vary widely in geographic context, polymer types, MP sizes, and concentrations used, limiting extrapolation to real-world field scenarios.

Mechanistically, the interactions between MPs and soil chemical properties are influenced by particle size, concentration, polymer type and the leaching behaviour of plastic additives and adsorbed contaminants. Smaller MP particles have higher specific surface areas, which can facilitate greater release of additives and bound pollutants during

rainfall or irrigation, potentially causing more pronounced soil chemical shifts than larger particles. [8–10] Similarly, higher MP concentrations exacerbate chemical perturbations. [11,12] Despite this understanding, empirical data explicitly linking MP size and concentration to a comprehensive suite of soil chemical properties, particularly for PE-MPs, remain limited. Laboratory and field studies have reported inconsistent or incomplete findings, and there is a notable scarcity of research conducted under realistic contamination scenarios that reflect agricultural field conditions.

In Australia, plastic mulching and irrigation practices are widespread in horticulture, with estimated annual PE usage ranging from 5000 to 10,000 Mg, and LDPE use around 5500 Mg. [3,13] Despite this extensive application, few studies have systematically evaluated the effects of PE-MPs on the chemical properties of Australian agricultural soils. This knowledge gap is significant considering the economic importance of horticulture in Australia and its reliance on plasticulture for optimizing yield and product quality. The sector encompasses high-value crops such as strawberries, tomatoes, and leafy vegetables, where soil chemical status critically influences crop performance.

This study aims to address these research gaps by investigating the effects of PE-MPs on multiple soil chemical properties under controlled microcosm conditions. We selected three particle size categories and three concentrations of PE-MPs, reflecting both current field exposure levels and worst-case contamination scenarios, based on empirical data collected from a strawberry farm in Queensland, Australia. [14] By simultaneously assessing a suite of soil chemical parameters—including pH, EC, cation exchange capacity (CEC), SOC, and organic matter—this work offers a holistic evaluation of MP-soil chemical interactions (Fig. 1). The results contribute mechanistic insights into how PE-MPs of varying sizes and concentrations alter soil chemistry and may inform sustainable soil management and plastic use policies. While the study is grounded in the Australian horticultural context, the findings have broader relevance for global agroecosystems where plasticulture is employed.

2. Materials and methods

2.1. Soil collection and preparation

Soils in this study were collected from Haden, Queensland, Australia (27.22 °S, 151.89 °E). The average annual temperature at the site is approximately 17 $^{\circ}$ C, with summer maximums reaching 28–34 $^{\circ}$ C. The region receives an average annual precipitation of about 685 mm, mostly occurring between December and February. Soil samples were homogenized, air-dried and sieved by 2 mm pore size. The topsoil at a depth of 0-20 cm was collected from random locations using a shovel. The soil texture was classified as loamy sand, determined by the hydrometer method, with 23 % clay, 13 % silt, and 64 % sand. It had a reddish colour and supported light grazing for cattle. The soil pH was 6.24 and EC was $0.055~dS~m^{-1}$ determined using a 1:5 soil-to-water ratio. Microplastic contamination in soil samples was investigated by the total organic carbon-solid sample combustion unit (TOC-SSM) method. [15] The quantified MP concentrations in the collected soil samples were approximately $20.289-21.755 \text{ mg kg}^{-1}$ of soil (0.00203 -0.00205 %; w/w), with slight variations due to differences in estimated carbon content (83 - 89 %). These background levels are negligible compared to the target concentrations used in this study (0.02 %, 0.1 %, and 1 %; w/w) and are therefore not expected to affect the experimental outcomes

2.2. Microplastic treatment

Microplastics were prepared by milling black PE mulch film, which was purchased online (Heman Agriculture Weeding Black Mulch, 0.8 m width, thickness 0.01 mm). The film was ground into a powder using a coffee grinder and then sieved into three size categories: Large (2000 -5000 μ m), Medium (600 – 2000 μ m), and Small (300 – 600 μ m). The size selection was informed by our previous finding that most MP residues in soil samples were larger than 300 µm, and by reported environmentally relevant size ranges for soil MPs (200–1000 μm). [15,16] The powdered PE-MPs were inspected using Attenuated Total Reflectance - Fourier Transform Infrared Spectroscopy (ATR-FTIR) to compare degradation and Scanning Electron Microscopy (SEM) to observe any critical alterations that occurred. The details of the MPs' preparation and characterization can be found in our previous publication. [15] The concentrations of MPs added to the soil microcosm experiment were Low (0.02 %), Medium (0.1 %), and High (1 %) by weight. The 0.02 % concentration corresponds to the MP levels observed in agricultural fields using plastic mulch film in Queensland. [15] The Medium and High levels were selected to explore potential effects under increasing contamination. In particular, the High level (1%) represents a worst-case scenario intended to accelerate impact detection and reveal possible system thresholds within a short experimental timeframe.

2.3. Incubation setup and conditions

The soil microcosm experiment was conducted in 70 mL glass bottles fitted with aluminium lids containing two perforations in a temperature-controlled chamber ($29\pm1~^\circ\text{C}$) with continuous air flow. Each bottle contained 70 g of air-dried soil. Soil moisture was maintained at 60 % of water holding capacity (WHC, w/w), monitored daily, and adjusted with distilled water as necessary. The treatment included three different MP sizes (Small, Medium, and Large) and three different MP concentrations (Low, Medium, and High). The total experimental group was ten and each group had five replicates: control (soil without MPs), SL (Small Low), SM (Small Medium), SH (Small High), ML (Medium Low), MM (Medium Medium), MH (Medium High), LL (Large Low), LM (Large Medium) and LH (Large High). The incubation experiment was

conducted for 90 days.

2.4. pH and EC of soils

Soil pH and EC were measured at 0, 30, 60, and 90 days after incubation using a pH meter and EC meter. The measurement was taken from a 1:5 soil-to-water suspension, equilibrated for 24 h using a mechanical shaker set to 120 revolutions per minute (rpm). The initial soil pH was 6.24, and EC was 0.055 dS $\rm m^{-1}$.

2.5. Cation exchange capacity measurement

Soil CEC was measured at 0, 60 and 90 days after incubation using 1 M NH₄Cl extraction solution. Briefly, 100 mL of 1 M NH₄Cl solution was added to 5 g of soil and adjusted to pH 7 with NH₄OH. The mixture was equilibrated on a mechanical shaker at 120 rpm for 1 h, filtered, and analysed by atomic absorption spectroscopy (AAS). Data collected at the 30-day sampling point were excluded due to instrument maintenance and sensitivity changes affecting data quality. The initial CEC was 11.72 cmol(+) kg. The concentrations of exchangeable cations were: Ca $^{2+}$ at $8.04\pm0.31~\text{cmol}(+)~\text{kg}^{-1},~\text{Mg}^{2+}$ at $1.74\pm0.05~\text{cmol}(+)~\text{kg}^{-1},~\text{Na}^{+}$ at $1.14\pm0.03~\text{cmol}(+)~\text{kg}^{-1},$ and K $^{+}$ at $0.80\pm0.00~\text{cmol}(+)~\text{kg}^{-1}.$

2.6. Water-extractable organic carbon measurement

Soil water-extractable organic carbon (WEOC) was measured at 0, 60, and 90 days after incubation (day 30 excluded as above) using a 1:5 soil-to-water extraction, shaken for 24 h at 120 rpm, centrifuged and filtered by a 0.45 μm nylon filter. The filtrate was diluted four times before analysis using a total organic carbon analyser (Shimadzu, TOC-V CSH). The initial WEOC was 28.27 mg L^{-1} .

2.7. Enzyme activity

Enzyme activity of the soils was evaluated at 0, 30, 60, and 90 days after incubation by measuring the activity of fluorescein diacetate (FDA) hydrolysis. Enzyme activity was quantified using substrate-based assays, with absorbance readings taken at 490 nm. [17] A fluorescein ($C_{20}H_{12}O_5$) standard stock solution (1 mg mL $^{-1}$ in acetone) was used for calibration. Initial enzyme activity was 111.96 µg g $^{-1}$ h $^{-1}$.

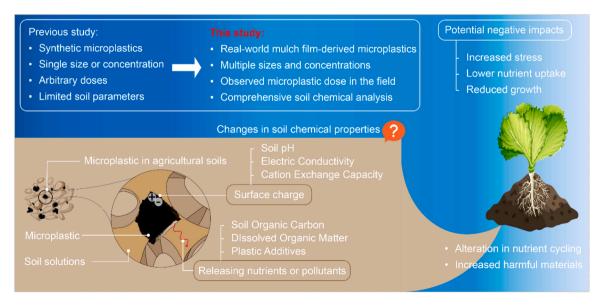


Fig. 1. Conceptual framework illustrating the hypothesized pathways by which microplastic inputs may alter soil chemical properties in agricultural systems. This schematic proposes that MPs could influence soil parameters via mechanisms such as altered surface charge and leaching of chemical additives, thereby changing soil chemical properties. These changes have the potential to affect soil function and, ultimately, crop performance.

2.8. Soil dissolved organic matter and fluorescence spectroscopy

At the end of the 90-day incubation, soil dissolved organic matter (DOM) was extracted at a soil-water ratio of 1:5. The extraction was performed by continuously shaking at $150\,r\,\text{min}^{-1}$ for 24 h in the dark at room temperature. The extracts were centrifuged (3000 rpm, 10 min), and the supernatants were immediately filtered through a 0.45- μ m nylon membrane.

Excitation–emission matrices (EEMs) of fluorescence were measured using a spectrofluorometer (RF-6000, Shimadzu) over excitation wavelengths of 200–500 nm and emission wavelengths of 250–550 nm, both at 2-nm intervals, in a 1-cm quartz cell at room temperature. [18] To eliminate inner filter effects, samples were diluted to ensure absorbance at 254 nm was below 0.1 before EEM measurements. [19] Water Raman and Rayleigh scatters in sample spectra were eliminated by subtracting blank spectra recorded under identical conditions. [20] The EEM data were subsequently normalized to Raman units (R.U.) using the integrated intensities of the Raman signal of the blank at an excitation wavelength of 350 nm.

The fluorescence data were processed in MATLAB R2024b using the drEEM toolbox for Parallel Factor Analysis (PARAFAC). [20] Models with two to six components were tested with non-negativity constraints and residual analysis, followed by split-half analysis to determine the optimal number of fluorescent components. Based on the results of these analyses, a four-component model was selected for further investigation. The model was validated through core consistency analysis and split-half validation. The relative contribution of individual components was calculated by determining the maximum fluorescence intensities (F_{max}) for each identified component and estimating their relative abundance in the samples. [21,22]

2.9. Phthalate acid ester analysis

Phthalate acid esters (PAEs) were extracted from soils following previously described methods with minor modifications. [23,24] Air-dried soil (2 g) was placed in a glass bottle, and 20 mL of n-hexane was added. Only glassware was used to avoid contamination with PAEs or other impurities. All glassware was rinsed sequentially with distilled-deionized water, acetone and hexane. Potential sources of contamination and interferences were identified by performing procedural blanks throughout all steps of the analysis. Samples were shaken for 24 h at 150 rpm on an orbital shaker to ensure thorough extraction. After the extraction, the sample was filtered through glass fibre filters to remove soil particles and obtain a clear extract. The extract was evaporated under a gentle flow of nitrogen to a final volume of 1 mL. An internal standard (benzyl benzoate) was then added to the sample to account for any variability in extraction and instrument response. The samples were analysed using a Gas Chromatography-Mass Spectrometry (GC-MS) system, Shimadzu GC-MS-QP2010 Ultra equipped with an AOC-20i autoinjector. Operating conditions were: 250 °C injection temperature, split-less injection mode, 1 µL injection volume and helium carrier gas. The temperature program began at 60 °C (1 min hold), ramped at 10 °C min⁻¹ to 280 °C and maintained for 6 min. [23] The Limit of Detection (LOD) and Limit of Quantification (LOQ) were set at a signal-to-noise ratio (S/N) of 3:1 and 10:1, respectively. Automated Mass Spectral Deconvolution and Identification System (AMDIS 2.73) software was used for deconvolution, and the National Institute of Standards and Technology mass spectra library (NIST 2.73) was utilized for compound identification. [25]

Targeted PAEs included five compounds from the EPA Phthalate Esters Mix (Sigma-Aldrich, Australia): dimethyl phthalate (DMP), diethyl phthalate (DEP), dibutyl phthalate (DBP), butyl benzyl phthalate (BBP), and bis (2-ethylhexyl) phthalate (DEHP). These compounds were calibrated with three different concentrations for further quantification of PAE analysis (Supplementary Table S1). Quantification was based on the ratio between the internal standard concentration to the detected

area, and the target compound concentration to the detected area for the detected PAEs.

3. Statistical analysis

Statistical analyses were performed using SPSS statistical software (version 28.0.1.0, IBM Corporation, Armonk, NY, USA). A multivariate analysis of variance (MANOVA) was conducted to assess the effects of different factors on multiple dependent variables. Post-hoc comparisons were conducted for significant factors to identify specific group differences. All statistical tests were performed at a significant level of p < 0.05. Parallel Factor Analysis (PARAFAC) was performed using MATLAB R2024b, following the method outlined by Murphy, et al. [20] All plots were generated using MATLAB R2024b to visualize the results.

4. Results

4.1. pH and EC of soils with MP treatment

The soil pH in the microcosms decreased steadily over the first 30 days and then fluctuated within a lower range compared to the initial pH. The MP-treated groups exhibited a similar trend across all groups throughout the incubation period. However, after 90 days, all MP-treated groups exhibited a higher pH compared to the control group (Figs. 2a and 2b). Both MP concentration (p = 0.037) and MP size (p = 0.042) had significant effects on soil pH. Higher MP concentrations increased soil pH by approximately 0.3 units compared to the control. When comparing MP size groups, no significant difference was observed between the S, M, and L size groups (Fig. 2a). However, the Medium and High concentration groups showed higher pH than the Low concentration group (Fig. 2b).

Soil EC slightly increased over the incubation period in all groups (Figs. 2c and 2d). At 90 days, statistically significant differences in EC were observed between the control and some MP-treated groups—specifically for large-sized MPs (p = 0.031) and medium MP concentrations (p = 0.006), as shown in Supplementary Table S1. The overall change in soil EC was approximately 0.2 dS m $^{-1}$. Soil EC generally remained more stable in MP-treated groups compared to the control, though fluctuations occurred, particularly at 60 days, when the high concentration group exhibited a higher EC than the control (Fig. 2d). However, no clear trend was observed regarding the impact of MP size and concentration on soil EC.

4.2. CEC of soils with MP treatment

The CEC of soils decreased more in MP-treated groups than in the control (Figs. 3a and 3b). After 60 days, the control group generally maintained its CEC level, whereas the MP-treated groups exhibited a continuous decline. Soil CEC was calculated as the sum of exchangeable cations: calcium (Ca²⁺), sodium (Na⁺), magnesium (Mg²⁺), and potassium (K+). Based on measured concentrations, Ca2+ contributed over 80 % of total CEC, followed by Na * (11 %), Mg 2* (4 %), and K * (< 1 %). Among these, Ca2+ showed the most distinct response to PE-MP treatments, exhibiting a more pronounced decline across all MP-treated groups compared to the control. Although relatively stable over time in the MPtreated soils, Na⁺ levels were consistently lower than those in the control group by day 90 (Fig. 3a). Magnesium and K⁺ displayed trends similar to the control. The influence of MP size was significant for soil CEC (p = 0.031). Importantly, the smallest MP size group showed the greatest decrease in CEC (over 3 cmol(+) kg⁻¹), which was largely attributed to the pronounced decline in Ca^{2+} concentration in this group (Fig. 3a). However, different MP concentrations did not significantly influence soil CEC or individual cations across all size categories; the medium and high concentration groups tended to show a more pronounced decline in both CEC and Ca²⁺ levels (Fig. 3b).

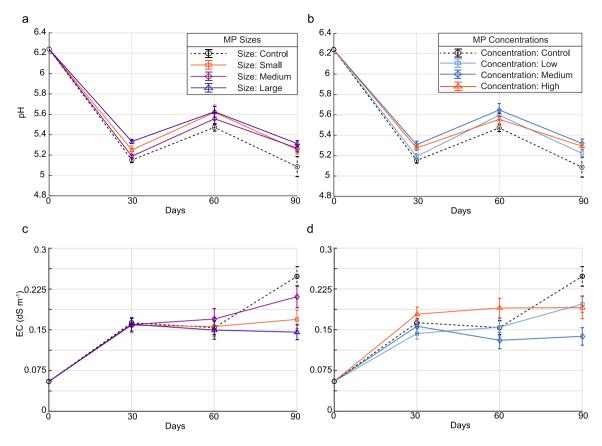


Fig. 2. pH and electrical Conductivity (EC) changes of soils after mixing with MPs over soil microcosm incubation periods. a: pH of soils over times treated by MPs with various sizes across all categories of MP concentrations, b: pH of soils over times treated by MPs with the various concentrations across all categories of MP sizes, c: Soil EC over time treated with MPs of various sizes across all categories of MP concentrations, and d: Soil EC over time treated with MPs of different concentrations across all categories of MP sizes. Note: Error bars represent the standard error of the mean.

4.3. Enzyme activity and WEOC of soils

All groups showed a similar trend in enzyme activity (measured by FDA) of soils over time, with an initial decrease followed by an increase (Figs. 4a and 4b). At the end of the 90-day incubation period, higher FDA were observed in the small and medium-sized MP groups compared to the control and large-sized MP groups.

The WEOC of soils showed a decreasing trend over the incubation period, with a steeper decline over time in all of the MP-treated groups compared to the control (Figs. 4c and 4d). A significant difference was observed between the MP concentration groups at 60 days, with the high-concentration MP group showing the fastest decline in soil WEOC. Between the 60- and 90-day incubation period, soil enzyme activity shifted to an increasing trend, while WEOC exhibited a steeper decline.

4.4. Dissolved organic matter

Fluorescence spectroscopy of EEM, analyzed using the PARAFAC model, revealed four fluorescent components in soil DOM after 90 days of microcosm incubation (Fig. 5a). Each component was classified based on findings from previous studies (Table 1). The C1 and C3 components were identified as fulvic-like substances while C2 and C4 were classified as humic-like substances. Although C2 and C4 are closely related, C2 is associated with lower molecular weight and less altered structures that could reflect more natural (unmodified) organic matter. [26] In contrast, C4 corresponds to humic-like substances but is characterized by a higher aromatic content, which may indicate microbial decomposition or another form of organic matter degradation. [27]

Most of the MP-treated groups showed a decrease in C1 and an increase in C4 (Fig. 5b). The reduction in C1 did not exhibit a clear

relationship with different MP sizes and concentrations. However, a more pronounced reduction of C1 was observed at medium and high MP concentrations compared to low MP concentrations. The size of MPs has almost no impact on C1. The increase in C4 was most distinct in the Small MP size (p=0.034), while no significant differences were observed across the different MP concentrations.

4.5. Leaching of PAEs

In the PAEs assay by GC-MS, four out of five control soil samples showed DEP concentrations below the LOD, indicating no detectable DEP. All MP-treated samples exhibited increased DEP concentrations and high variability (Fig. 6). Although no statistically significant differences in DEP concentrations were observed among MP-treated groups in different sizes, the Small size group showed slightly higher DEP concentrations in soils. Similarly, the High concentration group displayed the highest DEP levels across the different concentration groups. Notably, there were outliers in the Small size and High concentration MP-treated groups.

5. Discussion

5.1. Microplastic impacts on soil pH and EC

Soil with MPs showed a slightly higher pH than the control group, which aligns with findings from other studies. [6,33–35] However, the difference was only around 0.20 units, which is not considered substantial when compared to the pH fluctuations observed during incubation. This change is similar to a study that used a 1 % PE-MP concentration, where the pH change was 0.16 units after 3 months. [36]

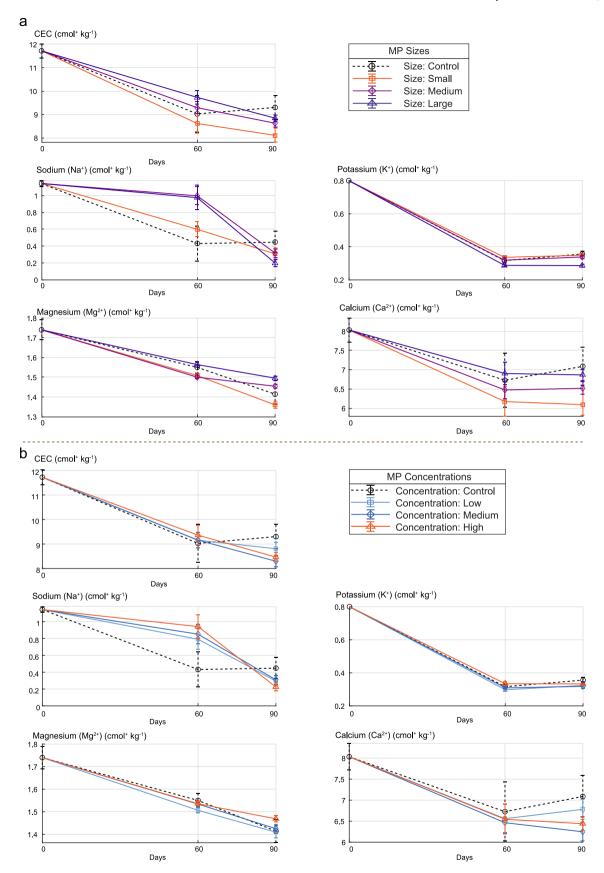


Fig. 3. The changes of soil cation exchange capacity (CEC) and cations during soil microcosm incubation. a: The changes of soil CEC and cations by different sizes of MPs across all concentration categories, and b: The changes of soil CEC and cations by different concentrations of MPs across all size categories. Note. Error bars represent SE.

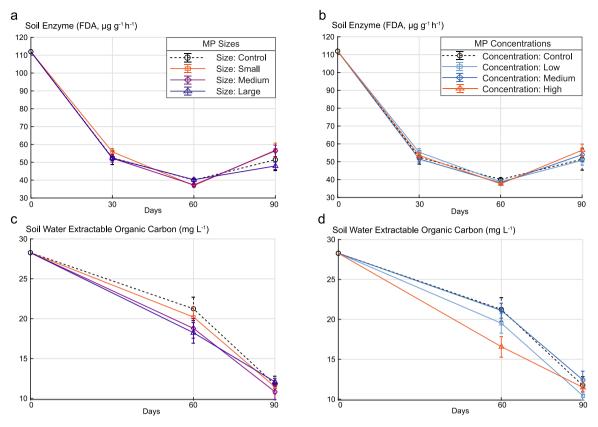


Fig. 4. Changes in soil enzyme activity (by FDA) and water-extractable organic carbon (WEOC) across different MP treatments. a: Soil enzyme activity by MP size across all categories of MP concentrations, b: Soil enzyme activity by MP concentration across all categories of MP sizes, c: WEOC by MP size across all categories of MP concentrations, and d: WEOC by MP concentration across all categories of MP sizes. Note. Error bars represent SE.

Additionally, our results showed that the pH change pattern was similar in both control and MP-treated groups, suggesting that the influence of MPs is not strong enough to alter the overall pH trend in the studied soil.

The pH difference between the control and MP-treated groups became larger over time (Fig. 2), indicating that PE-MPs may contribute to a shift in soil pH dynamics. Soil organic matter and clay content play critical roles in soil buffering capacity, as they can neutralize excess H* ions or absorb and release H* ions depending on the soil pH. [37] The surface charge of PE-MPs varies with pH, where the isoelectric point (IEP) for PE-MPs is around 3.2. [38] At pH values above 3.2, PE-MPs tend to have a negative surface charge, allowing them to adsorb positively charged ions, such as H* ions. Therefore, similar to organic matter and clay content, PE-MPs may act to hold onto H* ions, influencing soil pH dynamics (Fig. 7). Additionally, physical blocking of clay sites by PE-MPs could also contribute to pH alterations.

Other types of MPs, such as polypropylene (PP) and polylactic acid (PLA), have also been shown to influence soil pH. Polypropylene caused a slight decrease or increase in soil pH, but PLA showed a more pronounced impact. [33,36,39] These differences are likely due to variations in degradation rates and products, where faster-degrading plastics like PLA may release acidic compounds that alter soil chemistry more significantly than stable polymers like PE. [33] While MPs influenced soil pH to some extent, the effect did not appear to override the natural dynamics typically driven by environmental factors such as temperature and water availability. [33,40] It appears that the presence of MPs did not significantly disrupt these natural processes. However, most data come from short-term lab incubations. [33,36] The long-term, cumulative effects of MPs on soil pH under realistic environmental conditions remain poorly understood.

The MP-treated groups exhibited more stable EC values throughout the incubation period. This trend may be attributed to the increased electrical resistance caused by the insulating properties of PE-MPs, which could limit ion mobility in the soil. [41] Additionally, PE-MPs may adsorb metal ions and salts, reducing the concentration of free ions in the soil solution, thereby stabilizing EC during incubation. [42, 43]

Overall, the influence of MP contamination on soil pH and EC appeared to be limited in this study, likely offset by other factors such as irrigation water quality, temperature, rainfall, and land management practices. [44] While the MP-treated groups exhibited slightly higher pH values and more stable EC throughout incubation, the differences were minor and did not show significant changes against the natural trend. However, in real agricultural settings, the continuous introduction of MPs through plasticulture and irrigation practices could cumulatively affect soil pH and EC over longer periods. In subtropical and tropical environments, such as northeastern Australia, these effects may still be moderated by dominant regional climate conditions.

5.2. Microplastic impacts on soil CEC

Our results showed a decrease in CEC in MP-treated soils, consistent with findings from previous studies. [45–47] This decline in CEC may be linked to interactions between MPs and soil particles, reducing the soil's ability to retain essential cations. Similar to soil pH, the surface charge of PE-MPs may also play a critical role in soil CEC. [48] Given their IEP being approximately 3.2, PE-MPs can exhibit surface charge behavior similar to clay particles. However, their low chemical reactivity and complex surface morphology suggest that PE-MPs primarily interact with soil through physical rather than chemical mechanisms. [41] Our previous Fourier-Transform Infrared Spectroscopy (FTIR) analysis confirmed that the milling process did not significantly alter the chemical structure of the PE particles, particularly in terms of hydroxyl (–OH) groups, indicating that the MPs retained their original compounds and chemically inert properties after milling. [15] This suggests

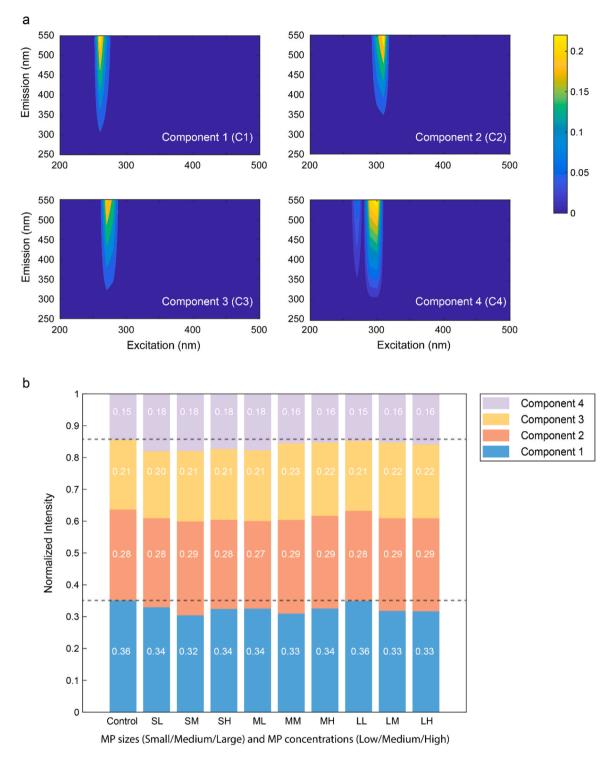


Fig. 5. Identified fluorescent components by PARAFAC analysis. a: The EEMs of PARAFAC components extracted from soil microcosms, b: The F_{max} for each identified component and estimating their relative abundance. Note. EEMs: Excitation-Emission Matrices, PARAFAC: parallel factor analysis, and F_{max} : maximum fluorescence intensities.

that PE-MPs affect soil CEC mainly through physical interactions. Our observations further indicate this, as the complex surface morphology of PE-MPs—characterized by roughness, crevices, and irregular shapes—likely traps these cations. Specifically, the rough, irregular surfaces of PE-MPs—featuring crevices and variable topography—likely trap cations and physically block access to soil exchange sites (Supplementary Figures S2–S4). [48] Their hydrophobic, film-like nature may further obstruct cation exchange between soil particles and the soil solution

(Fig. 7). Although the incubation period was short, soil organic matter may have begun interacting with the MP surfaces, increasing their hydrophilicity and potentially altering their interactions with soil cations. Such changes may contribute to a reduction in soil CEC through indirect mechanisms, such as the formation of organic coatings on MPs that retain cations and reduce their availability for exchange with soil particles. [49]

While both physical and chemical interactions may contribute to

Table 1Parallel Factor Analysis components based on previous studies.

ID	Excitation/ Emission (nm)	Components	References
C1	Ex 265 / Em 300-550	Ultraviolet A (UVA) fulvic-like substances. (Ex 265 / Em 460)	[28,29]
C2	Ex 310 / Em 340–550	Humic-like substances. (Ex 320 (250) / Em 400)	[26,27]
C3	Ex 270 / Em 320–550	UV/vis terrestrial Fulvic-like substances that are associated with high aromatic molecules of terrestrial origin.	[30–32]
C4	Ex 300 / Em 310-550	Humic-like substances, but higher aromatic content or microbial decomposition of organic matter.	[26,27]

CEC changes, their relative importance remains unclear and may not vary linearly with MP size. Notably, the smallest MP size group exhibited the greatest reduction in soil CEC, particularly for Ca²⁺ and Mg²⁺ (Fig. 3). This could be attributed to the larger specific surface area of smaller MPs, which enhances physical trapping, as well as the larger hydrated radii of divalent cations that increase their likelihood of becoming trapped within the crevices on MP surfaces or in the spaces between MPs and soil particles.

Although MPs have the potential to attract cations in the soil solution, other factors, such as soil pH, soil organic matter (SOM), and clay content, are also well-known factors that influence soil CEC. [50-52] In our study, CEC decreased over time in MP-treated soils, even though pH remained stable, suggesting that MPs might influence soil CEC independently of pH. Soil CEC can also fluctuate during incubation due to natural processes associated with these factors. In contrast, CEC in the control group slightly increased at 90 days, likely due to dynamic ion exchange processes during incubation, where initial desorption of Na* from soil exchange sites caused temporary increases. [53] The observed reduction in soil WEOC could be linked to the decrease in soil CEC, as SOM plays a crucial role in maintaining soil CEC. However, despite the continuous decline in WEOC (Fig. 4), the soil CEC in the control group remained stable after 60 days (Fig. 3), suggesting that MPs might influence soil CEC independently. Our results showed that medium and high MP concentrations caused greater reductions in exchangeable Ca²⁺, with the highest reduction observed at the high concentration level. However, the difference in CEC reduction between medium and high concentrations was not clearly defined by concentration alone. This

pattern likely reflects the complex interactions of PE-MP morphology with soil components, where surface roughness, crevices, and irregular shapes play a significant role in cation adsorption and physical blocking of exchange sites, limiting further impacts despite increasing concentration.

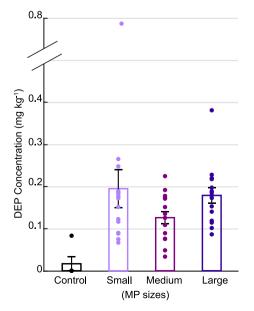
Some studies reported an increase in CEC in soil contaminated with MPs. A slight increase in CEC was observed with PE (300 μm), but a decrease was found with larger PE particles (600 μm). [54] Another study observed that soil CEC increased with LDPE. [55] However, their soil was heavily metal polluted, where CEC is greater than 40 cmol(+) kg^{-1} and they also included Al^{3+} in the CEC measurement. The increase in CEC may be attributed to differences in measurement methods, soil type, and experimental conditions, including greenhouse incubation, which could have influenced the results.

Overall, PE-MPs may directly impact soil CEC through complex interactions with soil particles and essential cations, particularly those with smaller particle sizes. This reduction in soil CEC is concerning, as it implies a weakened ability of the soil to retain vital nutrients such as Ca²⁺ and Mg²⁺, which are essential for plant growth. [56] Given that soil CEC supports nutrient retention and fertility, the impact of PE-MPs could weaken plant nutrient uptake and ultimately decrease crop productivity. [57,58] These findings highlight the importance of MP size in altering soil ion dynamics and emphasize the need for further investigation into the long-term impacts of MP contamination on soil health and agricultural sustainability.

5.3. Microplastic impacts on soil WEOC and enzyme activity

Previous studies have reported varying effects of PE-MPs on soil organic carbon (SOC) and dissolved organic carbon (DOC). Some studies found that PE-MPs decreased SOC and DOC, [59,60] while others reported insignificant effects [61,62] or even an increase in DOC. [46,63] These inconsistencies may stem from differences in MP properties (e.g., polymer type, size, and concentration), soil type and experimental conditions. [64] Additionally, MPs can be detected by various quantification methods and potentially inflate carbon measurements as carbonaceous organic materials. This methodological difference could explain the variability in reported results, as digestion or oxidation processes during the analysis may partially alter MPs, releasing additional carbon or influencing DOC measurements. [65]

Soil WEOC is a crucial component of soil-carbon dynamics, representing the most bioavailable fraction of organic carbon that directly



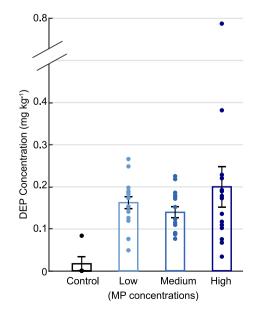


Fig. 6. Diethyl phthalate (DEP) concentrations in soil microcosms. Note. Error bars represent SE. Raw data points are displayed to illustrate variability.

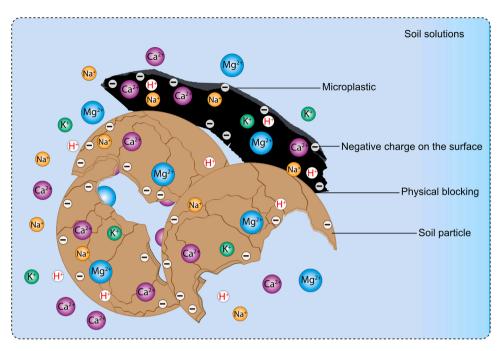


Fig. 7. Schematic representation of cation interactions with negatively charged soil particles and MP interference. Note. Conceptual illustration showing the interaction between cations and negatively charged soil particles within the soil solution. Adhered MPs are depicted on soil surfaces, emphasizing their potential to physically block cation exchange sites. This figure supports the discussion on how MP may alter ionic dynamics in soils.

influences microbial activity, nutrient cycling, and soil fertility. [66–68] In our study, soil WEOC declined over the incubation period across all treatments, with MP-treated groups exhibiting a more rapid decrease compared to the control (Fig. 4). However, the trend of WEOC decline was the same across the groups, with no significant difference between the control and MP-treated groups. This suggests that environmental factors, particularly temperature and moisture, have a stronger influence on soil WEOC dynamics than PE-MPs. [69,70] Nevertheless, the accelerated depletion of WEOC in MP-treated soils may indicate that higher MP concentrations contribute to faster carbon exhaustion, possibly by enhancing microbial activity and associated carbon consumption toward the end of incubation. These changes could potentially lead to shifts in microbial community structure and enzyme function.

Soil enzyme activities are among the fastest indicators of microbial responses to environmental stressors, including pollutants and other soil disturbances. [71,72] Soil enzyme activity is influenced by various factors, including temperature, moisture content, pH, SOM, and available nutrients. [73–76] In this study, soil FDA enzyme activity was measured to assess changes in soil quality and fertility. Initially, FDA decreased, but it started to increase after 60 days (Fig. 4). This may suggest that microbial activity and associated carbon consumption were enhanced towards the end of the incubation, leading to a more rapid depletion of carbon in MP-treated soils. Despite this recovery, the overall trend was similar across all treatments, suggesting that natural soil processes may have a stronger influence on enzyme activities than the addition of MPs.

The concentration of MPs used in this study may not have been sufficient to significantly alter soil enzyme activity. Previous studies with higher concentrations of PE-MPs (28 %) have shown a continuous decrease in FDA. [60] Similar to our result, a comparable concentration (0.02 %) of PVC also led to a decrease in enzyme activity, followed by a subsequent increase. [77] These findings suggest that the impact of MPs on soil enzyme activities may depend on the concentration and exposure time of the MPs, as well as environmental factors. [64]

The rapid decline in soil WEOC with MPs indicates changes in carbon availability and microbial activity, which could affect soil fertility and nutrient cycling. Ultimately, MP-contaminated soils may be more prone to losing fertility and compromising soil health. While the impact of MPs

on FDA aligned closely with natural soil processes influenced by temperature and moisture, soil WEOC and FDA tended to return to equilibrium with the control soil over time. However, high concentrations of MPs or different MP types may induce more profound and potentially lasting alterations to the soil ecosystem. [64]

5.4. Microplastic impacts on soil DOM

The shifts in soil DOM composition caused by MP contamination suggest significant alterations in SOM degradation processes (Fig. 5). Although 90 days is a relatively short period to observe drastic changes in soil DOM, the increased C4 (aromatic humic-like substances) and decreased C1 (fulvic-like substances) may indicate intensified processes of microbial decomposition or degradation of soil DOM by MP contamination. These findings align with previous studies, where the use of PE-MPs showed similar trends in soil DOM transitions, pointing to a negative priming effect in MP-contaminated soils. [78–80] However, our results did not show a clear linear relationship with MP size or concentration, although smaller-sized MPs and higher concentrations appeared to have a more pronounced effect on DOM transitions in soils. This suggests that smaller MPs, with their larger specific surface areas, may interact more significantly with microbial activity, potentially triggering stronger negative priming effects.

Contrasting priming effects were observed based on SOC content: soils with higher SOC showed a positive priming effect and increased labile DOM, while soils with lower SOC exhibited a negative priming effect that suppressed microbial activity and reduced labile DOM. [81] Additionally, environmental factors such as climate conditions may further modulate the effects of MPs on soil DOM. [82,83] This highlights that the impact of MPs on soil properties is less significant compared to other factors, such as environmental conditions and soil type. [78]

Further, different types of MPs showed varying impacts on soil DOM. Especially biodegradable MPs such as PLA, Polybutylene Succinate (PBS), Poly (butylene adipate-co-terephthalate) (PBAT), Polyhydroxyalkanoates (PHA) showed significantly higher soil DOC and a positive priming effect. [81,84,85] As PE-MPs did not show significant changes or increases in WEOC or DOC, [59,61,62] the mechanisms behind soil DOM alteration may differ between biodegradable and

non-degradable MPs. Biodegradable MPs may directly contribute to the sources of soil DOC and DOM, leading to a positive priming effect. In contrast, non-biodegradable MPs may alter soil DOM indirectly by affecting soil aggregates, aeration, moisture retention, and microbial communities. [60,79,86] Their presence can influence soil aggregate formation, potentially increasing macroaggregate stability and altering pore connectivity and aeration. [86] However, effects of MPs on soil aggregation show considerable variability depending on factors such as polymer type, concentration, soil properties, and environmental conditions. [79]

While PE-MPs may not immediately alter DOC or DOM in soils like biodegradable MPs, they could still induce long-term changes in soil microbial dynamics. These changes may gradually lead to shifts in microbial community composition and a potential decrease in soil fertility over time. Additionally, the persistence of non-degradable PE-MPs in the soil could lead to a build-up of MP contamination and an increased abundance of smaller-sized fractions, posing a long-term environmental risk to soil health. To address these challenges, developing effective management practices and promoting the use of biodegradable alternatives could help mitigate the long-term impacts of MP contamination in soils.

5.5. Microplastics as PAE sources

Phthalates, the most common plasticizers, have raised concerns due to their potential endocrine-disrupting properties. The observed leaching of PAEs from PE-MPs suggests that MPs can release PAEs, although the relationship is not definitive. [87,88] All MP-treated groups exhibited high variability in DEP leaching except for the control, indicating that leaching can occur even with larger-sized MPs or at low concentrations. This high variability in PAE leaching in our results indicates that factors beyond MP size and concentration may influence the leaching mechanism (Fig. 6). While MP concentration and PAE concentration have shown positive correlations, [89] this may be attributed to the surface characteristics of MPs rather than their concentrations in soil, as interactions with soil particles can reduce or hinder the leaching of PAEs from MPs. [90] The morphology of milled PE-MPs showed that, whether small or large, they all have adhered smaller particles on their surface, some of which may be at the nanoscale (Supplementary Figures S2-S4). These adhered smaller MPs or nanoparticles may also contribute to the leaching of PAEs. Additionally, smaller MPs generally have a larger specific surface area, which could further increase the leaching potential of PAEs from MPs.

During MP fragmentation by mechanical abrasion or degradation, PAEs may be released more readily. [91] This could explain the non-linear relationship between MP size and PAE leaching, as MPs may release PAEs differently depending on their physical condition and environmental interactions. Microplastics exposed to solar radiation at the soil surface, such as mulch films and irrigation hoses, could increase the concentration of PAEs in soils. The degradation of MPs due to solar radiation makes their surface more hydrophilic, facilitating the leaching of PAEs. [10] Furthermore, additional mechanical abrasion from agricultural practices like tillage or ploughing could further accelerate the release of PAEs into the soil, intensifying their potential environmental impact. [91]

More PAEs were accumulated in soils with PE-MPs than in soils with biodegradable MPs, even with short-term exposure (8 weeks). [24] Considering that PE is the most common plastic used in mulch films and irrigation hoses, [3] the continuous use of plastics in agriculture may result in the high accumulation of PAEs in soils over time. The impacts of PAEs on soil health and fertility may not be distinct in the short term, but they can have significant long-term effects by reducing soil microbial activity and enzyme functions and disrupting nutrient cycling processes. [92,93] These disruptions can ultimately reduce soil productivity and crop yields. As PE-MPs are widely used in agricultural practices, their continued application raises concerns about the risks of PAEs on soil

ecosystems, emphasizing the need for further investigation into mitigation strategies.

5.6. Limitations and future directions

One limitation of this study is the absence of initial microbial community profiling, such as 16S rRNA gene sequencing, which restricts our ability to directly correlate changes in DOM and CEC with microbial dynamics. While our study focused on the alterations of soil chemical properties induced by PE-MPs, soil chemical properties are often influenced by physical and biological factors, such as microbial activity, root interactions, and soil structural changes like aggregation and porosity. These interactions are not unidirectional, but form part of a complex and dynamic system where chemical, physical, and biological properties continuously interact.

This study was also limited by its focus on PE-MPs alone, which may restrict the universality of the findings since real field soils contain diverse mixtures of MP polymers with varying physicochemical characteristics. Additionally, the microcosm experiment for 90 days may not fully replicate field conditions, where larger soil volumes and environmental factors could lessen or dilute the effects of MPs in soils.

Future research should include investigations of mixed polymer types to better simulate actual MP contamination scenarios and consider larger-scale, long-term experiments. Incorporating realistic concentrations, particle sizes, and combined environmental stressors will help better understand the gradual and cumulative impacts of MPs. Such research offers more comprehensive assessments, aids in refining management practices, and ultimately informs policies addressing MP contamination in agricultural and natural soils.

6. Conclusions

This study has evaluated the impact of MPs on various soil properties, particularly soil pH, EC, CEC, WEOC, enzyme activity, DOM, and the presence of PAEs. The results indicated no definitive relationship between the size or concentration of PE-MPs and soil properties. However, the negatively charged surface of MPs may contribute to cation retention, while their complex morphology can trap ions and organic matter, which potentially affects ion dynamics and limits nutrient availability in the soil. In addition, MPs may induce negative priming effects and promote chemical leaching, further influencing soil chemical processes. The impact of MPs on the soil ecosystem is expected to increase over time with continuous inputs, potentially altering key soil functions. Additionally, these altered soil properties may negatively affect soil health and fertility, potentially leading to unforeseen and silent degradation that ultimately reduces crop productivity.

There is currently no complete mitigation strategy for MP contamination in soils. However, optimizing plastic mulch replacement cycles to minimize residue buildup, promoting the use and development of nontoxic biodegradable mulch alternatives, and implementing regular soil monitoring to detect early signs of MP pollution represent potential management approaches. While safe threshold concentrations for MPs in soils remain to be established, these practices may help reduce long-term risks to soil health and productivity until more comprehensive guidelines and solutions are developed.

Environmental Implications

This study investigates the effects of microplastics originating from real consumer products, applied at both field-observed and projected future concentrations. Findings show that microplastics can modify essential soil chemical properties, including pH, nutrient availability, and cation exchange capacity. While these changes may not be immediately visible, they have the potential to quietly degrade soil health over time. Given the complexity and buffering capacity of soil systems, such subtle shifts could accumulate and impact long-term

agroecosystem stability. These results underscore the importance of addressing microplastic pollution in soil through targeted monitoring, management practices, and forward-looking environmental policies.

Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript

CRediT authorship contribution statement

Li Li: Writing – review & editing. Pingan Song: Writing – review & editing, Supervision, Project administration, Funding acquisition. Guangnan Chen: Writing – review & editing, Supervision. John Dearnaley: Writing – review & editing. Yunru Lai: Writing – review & editing, Formal analysis. Yoonjung Seo: Writing – original draft, Formal analysis, Data curation, Conceptualization.

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Abbreviations

AAS, atomic absorption spectroscopy; AMDIS, Automated Mass Spectral Deconvolution and Identification System; ATR-FTIR, attenuated Total Reflectance - Fourier Transform Infrared Spectroscopy; BBP, butyl benzyl phthalate; CEC, cation exchange capacity; DBP, dibutyl phthalate; DEHP, bis (2-ethylhexyl) phthalate; DEP, diethyl phthalate; DMP, dimethyl phthalate; DOC, dissolved organic carbon; DOM, dissolved organic matter; EC, electrical conductivity; EEM, excitation-emission matrix; FDA, fluorescein diacetate; F_{max}, maximum fluorescence intensity; GC-MS, gas chromatography-mass spectrometry; IEP, isoelectric point; LDPE, low-density polyethylene; LOD, limit of detection; LOQ, limit of quantification; MP, microplastic; NIST, National Institute of Standards and Technology; PAE, phthalate acid ester; PAR-AFAC, Parallel Factor Analysis; PBAT, poly(butylene adipate-coterephthalate); PBS, polybutylene succinate; PE, polyethylene; PHA, polyhydroxyalkanoates; PLA, polylactic acid; PP, polypropylene; R.U., Raman unit; SEM, scanning electron microscopy; S/N, signal-to-noise ratio; SOC, soil organic carbon; SOM, soil organic matter; TOC-SSM, total organic carbon-solid sample combustion unit; WEOC, waterextractable organic carbon; WHC, water holding capacity.

Declaration of Competing Interest

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

Acknowledgment

(Word Style "TD_Acknowledgments"). Generally the last paragraph of the paper is the place to acknowledge people, organizations, and financing (you may state grant numbers and sponsors here). Follow the journal's guidelines on what to include in the Acknowledgments section.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jhazmat.2025.139668.

Data availability

No data was used for the research described in the article.

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