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Sorption of pesticides by microplastics, charcoal, ash, and river sediments

Marufa Fatema[1] · **Annemieke Farenhorst**[1]

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Abstract

Purpose In addition to sediments, pesticides can be sorbed to other constituents present in rivers including ash, charcoal, and microplastics. Pesticide sorption by microplastics has been studied for hydrophobic compounds such as the legacy insecticide DDT (dichlorodiphenyltrichloroethane) but not for current-use herbicides. The purpose of this study was to investigate to what extent 2,4-dichlorophenoxyacetic acid (2,4-D) (weak acid), atrazine (weak base), and glyphosate (zwitterion) are sorbed by microplastics (i.e., fiber, polyethylene beads, polyvinyl chloride (PVC), and tire fragments) and other river constituents (i.e., ash, charcoal, suspended and bottom sediments). DDT was included in the study to provide reference data that could be compared to known literature values.

Methods Batch equilibrium experiments were conducted following Guidelines 106 of the Organisation for Economic Co-operation and Development. Experiments utilized either a 1:100 solid/solution ratio with 0.1 g of a river constituent as the sorbent or a 1:5 solid/solution ratio with the sorbents consisted of 1.9 g bottom sediments mixed with 0.1 g of a river constituent. Background solutions included 0.01 M $CaCl_2$ or 0.01 M KCl, deionized water, and river water.

Result Individual microplastics always sorbed >50% of DDT. Current-use herbicides had a weak affinity for microplastics (< 6%) except that a substantial amount of glyphosate was sorbed by PVC (32–36%) in 0.01M KCl and DI water. When river water was used as a background solution, rather than 0.01M KCl or deionized water, there was much less glyphosate sorption by PVC, ash, charcoal, and both sediments. This suggested that ions present in river water competed for sorption sites with glyphosate molecules. Across background solutions, sorption by sediments decreased in the order of DDT (91–95%) > glyphosate (36–88%) >atrazine (5–13%) >2,4-D (2–5%). Sorption of 2,4-D, atrazine, and DDT by ash and charcoal was always > 90% but < 35% for glyphosate. Relative to bottom sediments alone, the presence of ash or charcoal (5% by weight) with sediments significantly increased the sorption of 2,4-D, atrazine, and DDT. Microplastic additions (5% by weight) had no impact on all four pesticides' sorption by sediments.

Conclusion Microplastics are not a strong sorbent for current-use herbicides, although there are exceptions such as glyphosate by PVC. Ions present in river water competed with glyphosate for sorption sites of river constituents. Hence, the types and concentrations of ions present in rivers might have some influence on the partitioning of glyphosate between the water column and solid phase, including glyphosate fate processes in rivers.

Keywords Batch equilibrium sorption · 2,4-Dichlorophenoxyacetic acid (2,4-D) · Atrazine · Glyphosate · DDT · Ash · Charcoal · Microplastics · Sediments · Background solution

1 Introduction

Pesticides enter rivers due to a number of processes such as atmospheric wet and dry deposition, rainfall and snowmelt runoff, as well as ground water recharge (Squillace et al. 1993; Waite et al. 2004; Cabrerizo et al. 2019). Depending on their physicochemical properties, pesticides in rivers reside in either the water column or sediments or in both

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✉ Marufa Fatema
fatemam@myumanitoba.ca

[1] Department of Soil Science, University of Manitoba, Ellis Building, 13 Freedman Crescent, Winnipeg, Manitoba R3T2N2, Canada

matrices (Rawn et al. 1999; Glozier et al. 2012; Ensminger et al. 2013; Ronco et al. 2016; Gamhewage et al. 2019). Mathematical models that estimate pesticide fate in rivers commonly include a sorption parameter (e.g., Kd, Koc) because when a pesticide is sorbed by sediments, it has different degradation rate and transport potential than when the same pesticide is dissolved in the water column. There is substantial information available on pesticide sorption parameters as it relates to sediments (Chefetz et al. 2004; Gaultier et al. 2009; Dollinger et al. 2015; Gamhewage et al. 2019) but less so as it relates to other potential constituents present in rivers such as microplastics, ash, and charcoal.

A wide range of studies have highlighted the persistence of microplastics in surface waters in North America, Europe, as well as Asia (Moore et al. 2011; Eriksen et al. 2013; Castañeda et al. 2014; Free et al. 2014; Lechner et al. 2014; Anderson et al. 2017). For example, the water column of the Red River was found to contain more than 800,000 microplastics km^{-2} at a sampling location in the Province of Manitoba, Canada (Warrack et al. 2017). Microplastics are per definition particles ≤5 mm and might include fiber, polyethylene (PE) beads, polyvinyl (PVC), and tire fragments, all of which have been detected in surface waters (Thompson et al. 2009; Arthur et al. 2009; Castañeda et al. 2014; Wagner et al. 2014; Warrack et al. 2017; Sighicelli et al. 2018; Wang et al. 2018; Leads and Weinstein 2019). Microplastics are present in the water column but also in bottom sediments because microplastics are likely to settle at some rate depending on factors such as polymer density. Graca et al. (2017) found that the density of microplastics were predominantly >1 g cm^{-3} when detected in the surface layer of marine bottom sediments but <1 g cm^{-3} when detected in the surface layer of beach sediments.

When present in water systems, microplastics are believed to act as carriers for transporting contaminants, but studies have predominantly focused on persistent organic pollutants such as legacy insecticide DDT (dichlorodiphenyltrichloroethane), as well as on pharmaceuticals and personal care products. For example, in batch equilibrium studies, these chemicals were shown to have a high sorption affinity for materials such as PVC, PE powder, polystyrene, and tire fragments (Bakir et al. 2014; Lee et al. 2014; Wu et al. 2016; Hüffer et al. 2019; Sharma et al. 2020). DDT and some of its metabolites have been detected in microplastics residing in surface waters, at concentrations ranging from 22 to 7,100 ng g^{-1} microplastic (Rios et al. 2007). DDT also continues to be detected in bottom sediments of surface waters throughout North America and elsewhere (Lembcke et al. 2011; Kuo et al. 2012; Kurek et al. 2019).

Current-use herbicides 2,4-dichlorophenoxyacetic acid (2,4-D) (weak acid; pKa=2.87 at 25 °C), atrazine (weak base; pKa=1.70 at 25 °C), and glyphosate (zwitterion; pKa= 2, 2.6, 5.6, and 10.6 at 25 °C) are being detected in both the water column and bottom sediments of North American rivers (Wan et al. 2006; Glozier et al. 2012; Ronco et al. 2016; Basiuk et al. 2017; Gamhewage et al. 2019). It is well known that carbon-rich substances such as ash and charcoal might retain these herbicides to some extent (Alam et al. 2000; Karapanagioti et al. 2004; Igwe et al. 2012; Ghosh and Singh 2013). Ash and charcoal loadings to rivers particularly occur when large regions are impacted by forest fires such as in recent years in Australia and Canada (Parks Canada 2018; Alberta Agriculture and Forestry 2019; Woodward 2020). In addition to atmospheric deposition of ash and charcoal, the increased surface runoff and erosion occurring post fire is responsible for enhancing the transport of sediment, ash, charcoal, and other debris to surface waters (Anshari et al. 2001; Karapanagioti et al. 2004; Smith et al. 2011; Mansilha et al. 2019; Rhoades et al. 2019). Coal fly ash released by coal burning power plants can also enter surface waters, for example, due to waste water discharge or atmospheric deposition (Eriksen et al. 2013).

We hypothesize that because of their contrasting physicochemical properties (Table 1), pesticides will each show distinct characteristics in their relative affinity for common constituents detected in rivers, including sediments, ash, charcoal, and a range of microplastics. The objective of this batch equilibrium study was to investigate to what extent 2,4-D, atrazine, and glyphosate are sorbed by microplastics (i.e., fiber, polyethylene beads, polyvinyl (PVC), and tire fragments) and other river constituents (i.e., ash, charcoal, and suspended and bottom sediments) and how this compares to the sorption potential of legacy pesticide DDT by these river constituents.

2 Materials and methods

2.1 Pesticides and sorbents

Pesticides used were 2,4-dichlorophenoxy acetic acid [ring-$^{14}C(U)$] (>99% radiochemical purity; specific activity 55 mCi $mmol^{-1}$) with analytical grade 2,4-D (99%), atrazine [ring-$^{14}C(U)$] (>99%, 160 mCi $mmol^{-1}$) with analytical grade atrazine (99%), glyphosate [phosphonomethyl-^{14}C](>99%, 55 mCi $mmol^{-1}$) with analytical grade glyphosate (99% chemical purity), and 4,4′-dichlorodiphenyltrichloroethane [ring-$^{14}C(U)$] (>99%, 12.8 mCi $mmol^{-1}$) with analytical grade DDT (99%). Pesticides were purchased from American Radiolabeled Chemicals, Inc., St. Louis, MO, USA, and Sigma-Aldrich Co., St. Louis, MO, USA.

Sorbents were suspended and bottom sediments, ash, charcoal, as well as fiber, PE beads, PVC, and tire fragments because these are among the most globally reported microplastics detected in the environment (Thompson et al. 2009; Engler 2012; Warrack et al. 2017; Hüffer et al. 2019).

Sediments were collected from the Red River at a location near Selkirk, Manitoba (50°08′42.2″N 96°51′51.3″W). The Red River (~880 km) has a drainage area of 288,000 km^2 (Newton 2016) with ~80% of its length flowing through the USA before entering Canada. A continuous flow centrifuge (US Centrifuge M512) was used to collect suspended sediments in centrifuge aluminum bowl, while water was pumped continuously through the device using high-power generator for 4–6 h. Bottom sediments (15 cm^3) were collected using an Ekman Dredge. Sediments were placed into sterile amber plastic bags (Fisher Scientific International, Inc., Fair Lawn, NJ) and transported to the laboratory. Samples were air-dried and sieved (<2 mm). Activated charcoal Norit® and fly ash BCR® were purchased from Sigma-Aldrich Co., St. Louis, MO, USA. The activated charcoal is a chemically activated (by phosphoric acid) powder derived from wood with particle sizes ranging from 10 to 90 µm and a specific surface area (SSA) of 1400 m^2 g^{-1}. According to the manufacturer, the charcoal contained calcium (200 mg kg^{-1}), iron (150 mg kg^{-1}), and phosphate (3.5% on a mass basis). The fly ash is derived from pulverized coal with trace elements with particle sizes ranging from 0.5 to 40 µm and a SSA of 900 m^2 g^{-1}. PE microbeads with particle sizes of 250–300 µm were used as model polymer typical of personal care products and purchased from Cospheric LLC (Santa Barbara, CA, USA). Tire pieces (<5 mm) were collected from Reliable Tire Recycling Center, Winnipeg, Manitoba. Polyvinyl chloride (PVC) pipes were purchased from The Home Depot, Winnipeg, Manitoba, and finely shredded into small pieces (<5 mm). Polyester fibers (<5 mm) were gathered from the insides of a newly bought pillow.

2.2 Batch equilibrium experiments and data analyses

Experiments were done in triplicates. One experiment utilized a 1:100 solid/solution ratio (0.1 g/10 mL) and included the four pesticides (2,4-D, atrazine, glyphosate, DDT), eight individual sorbents (suspended and bottom sediments, ash, charcoal, PE beads, PVC and tire fragments), and three background solutions (0.01 M CaCl$_2$ or 0.01 KCl, deionized water, river water). The other experiment utilized a 1:5 solid/solution ratio (2 g/10 mL), and sorbents consisted of 1.9 g bottom sediments mixed with 0.1 g of each of the eight sorbents (suspended and bottom sediments, ash, charcoal, fiber, tire, PVC, and PE beads). Although studies to date lack consistent and standardized methods/protocols to quantify microplastics present in bottom sediments (Campanale et al. 2020), we selected this ratio because the concentrations for microplastics, charcoal, or ash in rivers are relatively small in volume compared to bottom sediments (Corcoran et al. 2015; Anderson et al. 2016; Warrack et al. 2017; Campanale et al. 2020).

Stock solutions of pesticides were prepared at a concentration of 2.5 µg l^{-1}. A total of three different background solutions were used in experiments. This included standard ionic solution of 0.01 M CaCl$_2$ (pH of 5.97) for 2,4-D, atrazine, and DDT and of 0.01 M KCl (pH of 6.24) for glyphosate (OECD 106 2000), as well as deionized water (pH 6.35) and river water (pH 8). Pesticide solutions are usually prepared in 0.01M CaCl$_2$, but glyphosate forms complex formation with Ca^{2+} in the solution, and hence KCl is the preferred electrolyte in batch equilibrium studies using glyphosate (Glass 1987; Munira et al. 2018). The river water was analyzed for a range of chemicals such as total calcium (69.3 mg l^{-1}), magnesium (49.1 mg l^{-1}), and others (Table S1). Prior to use in experiments, river water was filtered through Whatman glass fiber membrane filters (0.45 µm pore size, 47 mm), as well as autoclaved at 121 °C for 20 min. Pesticide solutions (10mL) were added to 50-mL glass centrifuge tubes (PYREX™ Round-Bottom Glass Centrifuge Tubes) containing sorbents (0.1 or 2.0 g). Tubes were rotated in the dark for 24 h at 5 °C as most batch equilibrium studies utilize this duration (Wauchope et al. 2002) and preliminary experiments with atrazine and 2,4-D did not show significant differences between sorption at 24h and 48 h. Tubes were removed and centrifuged at 7,000 rpm for 50 min at 5 °C. Subsamples (1 mL) of supernatant were added to duplicated 6 mL scintillation vials, each containing 3 mL Ultima Gold™ scintillation cocktail (PerkinElmer, Inc., Waltham, MA, USA). Vials were lightly shaken and placed in dark for 24 h to reduce false positives by photoluminescence or chemiluminescence. Radioactivity was quantified using liquid scintillation counter (Tri-Carb 4910 TR, PerkinElmer, Inc., MA, USA) with a counting time of 10 min.

For each pesticide and background solution separately, one-way analysis of variance (ANOVA) (GLIMMIX procedure, SAS 9.4) was performed on percent pesticide sorbed. The percentage sorption data followed a beta distribution, and treatment mean differences were evaluated at α=0.05 with the Tukey multiple comparison procedure.

3 Results and discussion

As individual sorbents (0.1 g), microplastics never sorbed more than 6% of 2,4-D or atrazine (Fig. 1). Glyphosate also showed a weak sorption by microplastics, except that PVC fragments showed between 32 and 36% glyphosate sorption in 0.01M KCl and DI water (Fig. 1). Previous studies have reported that some fungicides and insecticides, as well as a flame retardant tris(2-chloroethyl) phosphate, also showed an affinity for PVC (Gong et al. 2019; Chen et al. 2020; Tatarczak-Michalewska et al. 2021). In contrast to these current-use herbicides, legacy compound DDT showed between 85 and 94% sorption by fiber, tire, or PVC fragments and between 55 and 66% sorption by

PE beads (Fig. 1). Using batch equilibrium studies, Bakir et al. (2014) also reported that DDT has a strong affinity for PVC and PE. Microplastics have hydrophobic properties, and hence DDT and other hydrophobic chemicals with relatively large octanol-water partition coefficients have a large affinity for microplastics (Moyo et al. 2014; Wu et al. 2016; Beckingham and Ghosh 2017; Li et al. 2018; Llorca et al. 2018; Zuo et al. 2019). Partitioning interaction and pore filling are the predominant mechanisms controlling the sorption of DDT to microplastics (Bakir et al. 2012; Endo and Koelmans 2016; Wang et al. 2020).

Ash and charcoal tend to have relatively high specific surface areas, and as individual sorbents, ash (0.1g) and charcoal (0.1g) sorbed > 90% of 2,4-D, atrazine, and DDT (Fig. 1). Previous studies reported that ash or charcoal sorbed between 54 and 87% of 2,4-D (Deokar et al. 2016), 32–99% of atrazine, and > 90% DDT (Gupta and Ali 2001; Ghosh and Singh 2013; Gonzalez et al. 2020). Suspended (0.1 g) or bottom (0.1 g) sediments sorbed > 90% DDT as well, but both 2,4-D (2–5%) and atrazine (5–13%) showed a weak affinity for sediments (Fig. 1). As individual sorbents (0.1 g), both suspended and bottom sediment sorbed a substantial amount of glyphosate in 0.01M KCl (86–88%) and DI water (67–68%). However, the amount of glyphosate sorbed by suspended and bottom sediment was only between 38 and 51% in the presence of river water (Fig. 1). This suggests that ions present in river water competed with glyphosate for sorption by suspended and bottom sediments. The river water contained a range of chemicals (Table S1) that are known to be sorbed by sediments including phosphorus, lead, cadmium, copper, and zinc (Phillips et al. 2004; Oh et al. 2009; Huang et al. 2015; Yin et al. 2016). As individual sorbents, ash (0.1g) and charcoal (0.1g) showed ~30% glyphosate sorption in 0.01M KCl and DI water solutions (Fig. 1). However, both PVC (~2%) and ash or charcoal (~10%) showed a much lesser glyphosate sorption in the presence of river water (Fig. 1). This suggests again that ions in river water competed with glyphosate for sorption by river constituents, including PVC, ash, and charcoal. The river water contained a range of chemicals (Table S1) that are known to be sorbed by PVC such as lead, arsenic, and cesium (Wankasi and Dikio 2014; Sing et al. 2020; Chen et al. 2020). Similarly, phosphate in river water might have competed with glyphosate for sorption by ash and charcoal because, for example, glyphosate sorption by wood biochar (also a carbon-rich substance) was reduced in the presence of phosphate because of competitive sorption mechanism (Cederlund et al. 2016; Hall et al. 2018). In addition, river water had a pH of 8, whereas the pH of the other two background solutions was less than 6.5. Previous studies have shown that the sorption of glyphosate by clays, soils, and sediments decreases with increasing pH because glyphosate is becoming increasingly negatively charged (McConnell and Hossner 1985; de Jonge and de Jonge 1999; Gimsing et al. 2003; Dollinger et al. 2015). Specifically, glyphosate sorption is reduced because of the increased negative charges of both soil and glyphosate result in greater electrostatic repulsion with rising pH (de Jonge and de Jonge 1999; Wauchope et al. 2002; Candela et al. 2007; Ololade et al. 2014).

Fig. 1 Pesticide sorption by individual solid constituent in $CaCl_2$, KCl, deionized water, and river water solutions (1:100 solid/solution ratio)

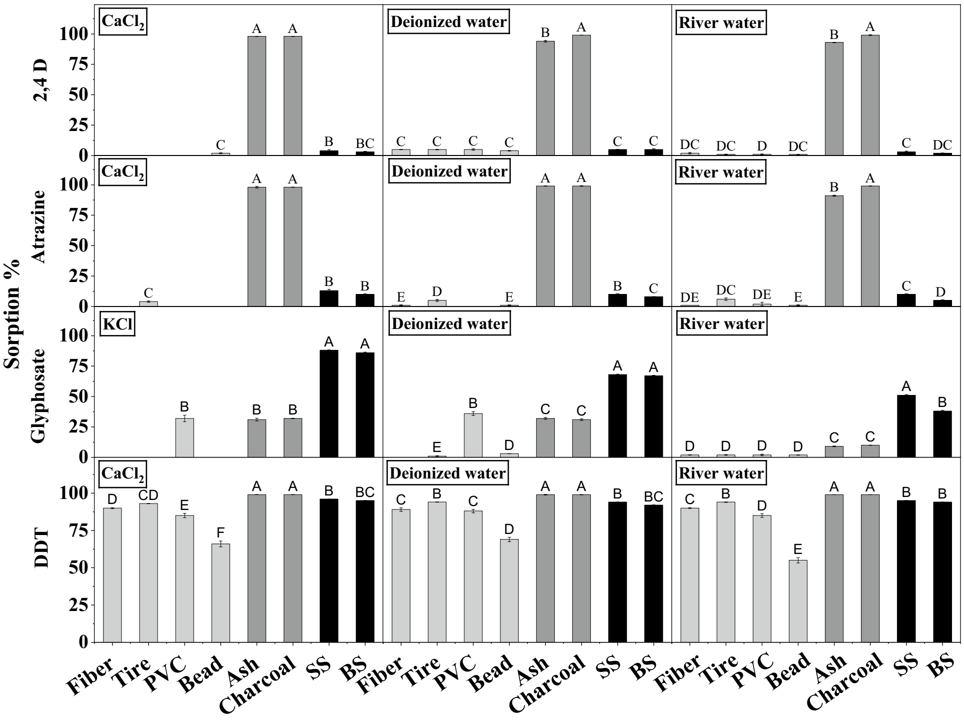
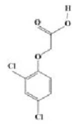
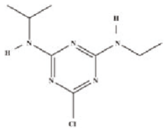
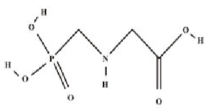
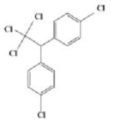


Table 1 Molecular structure and physicochemical properties of the pesticides used in the study (Sprankle et al. 1975; Footprint 2020; PubChem Compound Database 2020). Solubility = solubility in water (mg L^{-1}) at 20 °C; log Kow = log Kow at pH 7, 20 °C; Soil ½-life = soil half-life (days) at 20 °C; Sediment ½-life = sediment half-life (days), except for DDT (years)

	2,4-D	Atrazine	Glyphosate	DDT
				
Solubility	23,180	35	10,500	0.006
logKow	-0.83	2.70	-3.20	6.91
pKa@25^{0}C	2.87	1.70	2, 2.6, 5.6, 10.6	-
Koc	39.3	100	1424	151000
Soil ½-life	4.4	75	15	6,200
Sediment ½-life	18.2	80	9.9	>150 years

Regardless of the background solution, the amount sorbed of the legacy compound DDT, as well as of current-use herbicides 2,4-D and atrazine, typically decreased in the order of ash/charcoal>sediments>microplastics. Glyphosate sorption typically decreased in the order of sediments>ash/charcoal>microplastics. Consequently, regardless of the pesticide, microplastics tend to be less likely to sorb pesticides than other river constituents. For none of the four pesticides, there was a significant impact of the type of microplastics present in sediments on % pesticide sorbed by sediments. Bottom sediments with or without microplastics sorbed < 20% 2,4-D, ~55–58% atrazine, but > 95% of glyphosate and DDT (Fig. 2). In contrast, the presence of 0.1 g of either ash or charcoal with 1.9 g bottom sediments significantly increased the sorption of 2,4-D, atrazine, and DDT relative to 2.0 g bottom sediments only. The increased sorption was particularly profound for current-use herbicides 2,4-D and atrazine (Fig. 2). Relative to 2.0 g bottom sediments only, the presence of 0.1 g charcoal with 1.9 g bottom sediments significantly decreased glyphosate sorption. However, numerically, the differences between these two treatments were extremely small because > 95% of glyphosate was sorbed in both cases (Fig. 2). Other studies reported on the strong sorption of glyphosate by sediments (Xu et al. 2009; Degenhardt et al. 2012; Dollinger et al. 2015) and by soil (Shushkova et al. 2010; Munira et al. 2016; Munira and Farenhorst 2017; Munira et al. 2018).

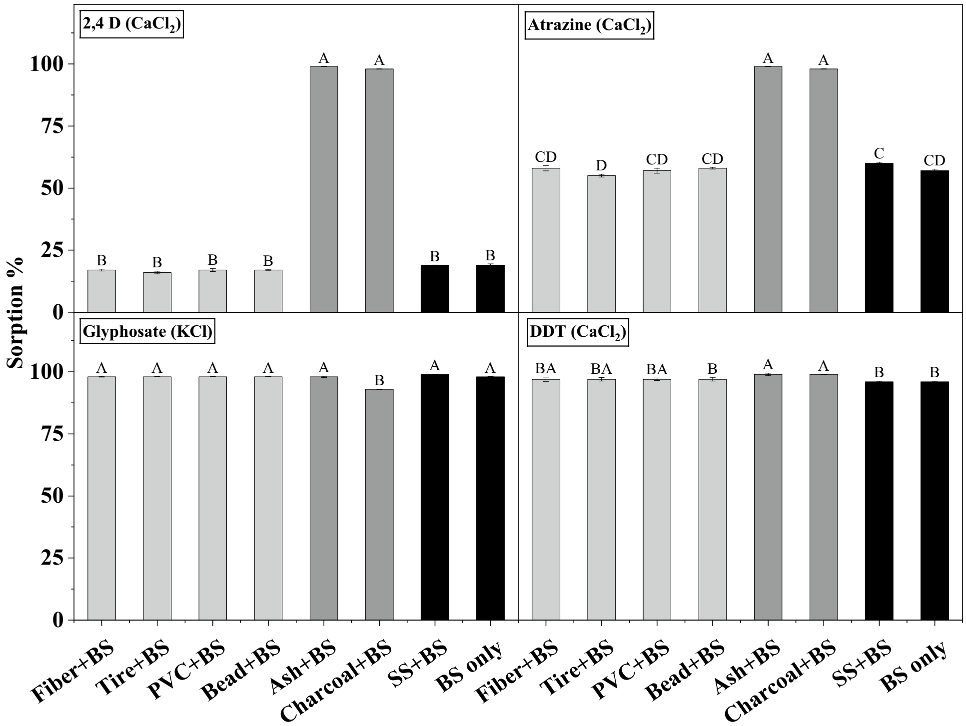


Fig. 2 Pesticide sorption by mixture of bottom sediment and other solid constituents in $CaCl_2$ and KCl solution (1:5 solid/solution ratio)

4 Conclusion

Based on the batch equilibrium conditions studied, we conclude that it is unlikely that microplastics will accumulate current-use herbicides such as 2,4-D, atrazine, and glyphosate. Although glyphosate is potentially retained by PVC, ions in river water appear to interfere with this glyphosate sorption. This might warrant including the types and concentrations of ions present in rivers when describing on glyphosate fate processes in rivers, including sorption by river constituents such as sediments and PVC. The amount of DDT sorbed by microplastics was substantial, and hence such partitioning coefficients should be included when modeling the fate of DDT in river systems. The presence of ash/charcoal in regions impacted by large forest fires is likely to increase the removal of 2,4-D and atrazine from the water column, both in the short term when ash/charcoal is suspended in the water column and in the long term when ash/charcoal has settled in bottom sediments. In contrast, the presence of microplastics is unlikely to impact the removal of 2,4-D, atrazine, and glyphosate from the water column in rivers.

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Declarations

Conflict of interest The authors declare no competing interests.

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