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Sorption of Pb and Cu on different types of microplastics

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Abstract. The studies on the effect of different plastic properties (e.g., types, shapes, presence of additivies) on the sorption of contaminants in the agricultural environment are limited. In this study, Cu and Pb, the commonly found heavy metals in the environment, were used to investigate the sorption capacities of microplastics (MPs). The Pb sorption capacity increased in the order of polystyrene (PS)<polyethylene (PE)<polyvinyl chloride (PVC). The estimated Cu sorption capacity was greater for the PE films than the PE fragments, while the sorption strength was greater for the PE fragments. This suggests that the shapes of MPs can affect the contaminant sorption capacities. With the PE fragments, the Pb sorption capacity was greater than the Cu sorption capacity by 10-12 times. Also, the Pb and Cu sorption capacities were greater for the PE fragments with additives than the PE fragment without additives. After the sorption of Pb or Cu on MPs, the toxic effects of the Pb or Cu solutions were decreased, suggesting that the toxic effects of contaminants can be affected by the co-presence of MPs in the environment. Overall, the results show that different types and shapes of MPs and the presence of additives can affect the heavy metal sorption capacities of MPs.
Keywords: Cu; microplastics; Microtox; Pb; sorption

1. Introduction

Plastics in soil and water environments can accumulate in living organisms through food chain and threaten human health, and this has emerged as a major environmental concern (FAO 2021, Kim and Choi 2022). In agricultural environments, vast amounts of plastics have been used for various purposes. For example, mulching films have been used to control weed, water content, and crop quality. Also, plastic films have been used to make greenhouses for crop cultivation. However, improper management of the used plastic products in agricultural environments can lead to the generation of microplastics (MPs), which, in turn, can impose negative effects on soil environment (Hwang 2016). Plastics with a size of 5 mm or less are classified as MPs (Hur and Jho 2021). According to the Korean agricultural film waste generation statistics of 2020, 53,589 ton of low-density polyethylene (LDPE) used for greenhouses, 156,422 ton of LDPE used for mulching films, 81,517 ton of high-density polyethylene (HDPE), and 15,631 ton of others (polyvinylchloride (PVC), ethylene vinyl acetate (EVA), polyolefin (PO)) were generated as wastes, and only 6 ton, 98,223 ton, 96,791 ton, and 17 ton were collected, respectively (Statistics Korea 2022). The LDPE films were the most generated plastic wastes in the agricultural environment, but only about 47% of this was collected. This suggests that a significant amount of the generated plastic wastes remains in the soil environment.

Microplastics (MPs) remaining in soil can affect not only the soil properties but also the terrestrial ecosystem (Corradini *et al*. 2019). Also, MPs in soil can enter deeper soil through various routes (e.g., during agricultural tillage, through soil cracks) causing damage to the soil structure (Wang *et al*. 2021). The MPs in soil can be absorbed and accumulated by organisms residing in soil (Gao *et al*. 2022). In addition to the particles of MPs, several chemicals (e.g., plasticizers, flame retardants, surfactants) that are added during the manufacturing process of plastics to improve the properties of plastics can leach out to the environment during the degradation of plastics (Koelmans *et al*. 2014, Dissanayake *et al*. 2022, Zhu *et al*. 2022).

Many studies reported on the interaction of MPs with organic or inorganic contaminants (Gao *et al*. 2019, Öz *et al*. 2019, Guo *et al*. 2020, Zhang *et al*. 2020, Dissanayake *et al*. 2022). For example, the adsorption capacities of per-fluorooctanesulfonamide on different types of MPs were in the order of polyethylene (PE)>PVC>polystyrene (PS) (Dissanayake *et al*. 2022). In another study, among various types of MPs (polypropylene (PP), PE, polyamide (PA), PVC, polyoxymethylene (POM); 4 mm), PVC and PP adsorbed more heavy metals (Pb, Cu, Cd) than the other MPs, and the adsorption capacity of each heavy metal was different for each type of MPs (Gao *et al*. 2019). For PP, PVC, and PE, the sorption capacities decreased in the order of Pb, Cu, and Cd, and the sorption capacities decreased in the order of Cu, Pb, and Cd for PA and POM (Gao *et al*. 2019). The Cd adsorption on PE, PP, PVC, and PS (<75

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μm) increased in the order of PE<PP<PS<PVC (Guo *et al*. 2020). The Cd adsorption on different sized PE MPs (48-58 μm, 100-154 μm, 0.6-1.0 mm, 1.0-2.0 mm) increased with decreasing size of MPs, and this was described better with the Langmuir isotherm model than the Freundlich isotherm model (Zhang *et al*. 2020). Another study observed that the Freundlich isotherm model is more suitable than the Langmuir isotherm model for the Pb adsorption on different types of granular MPs (polyethylene terephthalate (PET), PA, EVA; 0.5-1 mm) (Öz *et al*. 2019). The organic and inorganic contaminants sorbed on MPs can cause the spread of contamination as MPs can act as carriers, and the sorbed contaminants can be desorbed again imposing toxic effects (Wang *et al*. 2021).

The heavy metal adsorption on MPs has been studied continuously; however, more studies are still needed due to the various properties of MPs including types, shapes, and sizes and the various types of contaminants that can interact with MPs. In addition, the studies on the effect of plastic additives on the sorption of contaminants by MPs are limited (Wang *et al*. 2021). Therefore, this study used two types of heavy metals and three types of MPs to investigate the effect of the types and shapes of MPs and the presence of additives in MPs on the heavy metal sorption. As the target heavy metals, Cu, which is an essential element, and Pb, which is a non-essential element, were used as they are commonly detected in the soil environment (Azeez *et al*. 2015). As the target MPs, PE, PVC, and PS, which are commonly detected in the environment, were used. Ecotoxicity experiments were also conducted to investigate the changes in the toxic effects by the sorption of heavy metals on MPs.

2. Materials and methods

2.1 Preparation of MPs fragments and films

In this study, MP fragments and MP films were used in the sorption experiments. MP fragments without additives were prepared from PE, PVC, and PS pellets. The PE, PVC, and PS pellets without additives were purchased from LG Chem (Korea), Pountek Ltd. (Korea), and INEOS Styrolution (Germany), respectively. PE fragments were also prepared from a commercial product of PE beads used for making accessories (IKEA, Thailand). The PE beads may contain metal-based additives such as stabilizers, pigments for color, anti-corrosion agent, and flame retardants (Turner and Filella 2017, Turner and Filella 2021, Akimzhanova and Guney 2022). The pellets and beads were ground using a grinder (SHMF-3080SS, Hanil, Korea) with the injection of liquid nitrogen, and sieved through a 125 μm stainless steel sieve to collect MP fragments of <125 μm. MP films were prepared from black PE mulching film (Yeji Industry, Korea). The film was cut to have <5 mm length and <5 mm width.

2.2 Pb sorption on different types of MPs

The Pb sorption experiments using different types of MPs (i.e., PE, PVC, PS) were carried out in 50 mL tubes containing a Pb solution (20 mL) and each type of MPs (0.2 g). The Pb solution was prepared with $PbCl_2$ (>99%, Junsei, Japan) to have the initial concentration of 9±1 mg L^{-1} and this was serially diluted to prepare the Pb solutions of different concentrations (on average, 0-9 mg L^{-1}). The initial pH values of all the Pb solutions used in the sorption experiments were 5.3±0.3. The tubes were shaken at 45 rpm on a reciprocal shaker (SH30L, FinePCR, South Korea) for 48 h. After 48 h-sorption, the residual Pb concentrations were analyzed using ICP-OES (Thermo Scientific, iCAP 7000 Series, USA) after filtering through a 0.45 μm filter. The experiments used 3-5 replicates for each condition.

2.3 Cu sorption on different shapes of PE MPs

The Cu sorption experiments using different shapes of PE MPs (i.e., fragments and films) were carried out in 50 mL tubes containing a Cu solution (20 mL) and PE MPs (0.2 g). The PE fragments and films were prepared from commercial bead and mulching vinyl products. The effect of the presence of additives on the Cu sorption was also studied by using the PE fragments prepared from the PE pellets without additives and the commercially sold PE beads. The Cu solution was prepared with $CuCl_2$(>99%, Sigma Aldrich, Germany) to have the initial concentration of 10±5 mg L^{-1} and this was serially diluted to prepare the Cu solutions of different concentrations (on average, 0-10 mg L^{-1}). The initial pH values of all the Cu solutions used in the sorption experiments were 5.5±0.1. The tubes were shaken at 45 rpm on a reciprocal shaker (SH30L, FinePCR, South Korea) for 48 h. After 48 h-sorption, the residual Cu concentrations were analyzed using ICP-OES (Thermo Scientific, iCAP 7000 Series, USA) after filtering through a 0.45 μm filter. The experiments used 3-5 replicates for each condition.

2.4 Sorption isotherm models

The amount of Pb or Cu sorbed on the MPs was determined using the Eq. (1) (Ju *et al*. 2021). The sorption experiment data were fitted to two isotherm models, the Langmuir isotherm model (Eq. (2)) and Freundlich isotherm models (Eq. (3)) (Benhafsa *et al*. 2022, Ju *et al*. 2021, Kumar *et al*. 2022).

$$q_e = \frac{(C_0 - C_e) \times V}{W} \quad (1)$$

$$q_e = \frac{Q_m K_L C_e}{1 + K_L C_e} \quad (2)$$

$$q_e = K_F C_e^{1/n_F} \quad (3)$$

where q_e is the amount sorbed at equilibrium (mg g^{-1}), C_0 is the initial concentration of Pb or Cu (mg L^{-1}), C_e is the equilibrium concentration (mg L^{-1}), V is the volume of Pb or Cu solution, W is the weight of MPs used in the sorption experiment, Q_m is the maximum monolayer sorption capacity of the MPs (mg g^{-1}), K_L is the sorption equilibrium

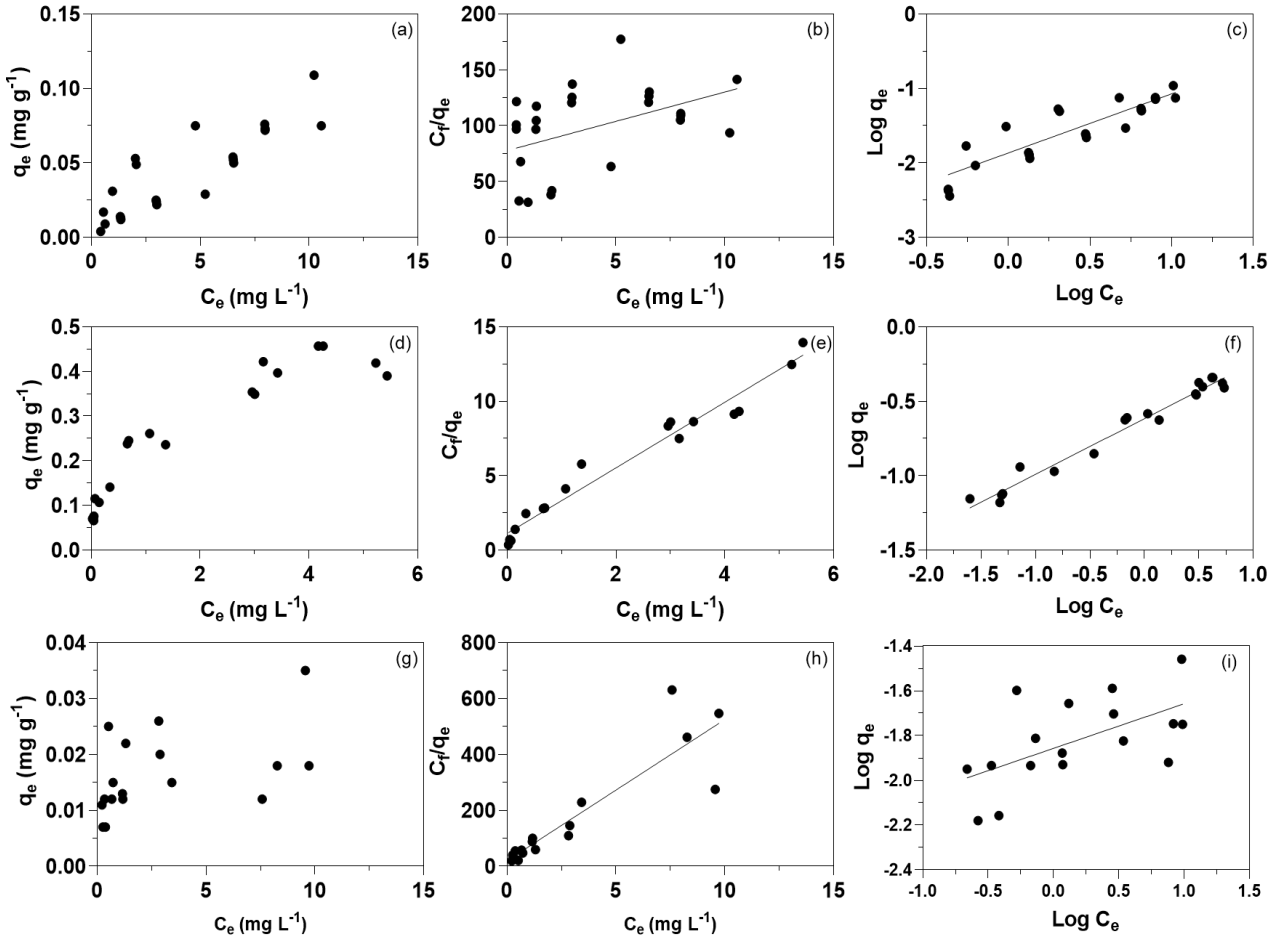

Fig. 1 (a) Data obtained from equilibrium sorption experiments, (b) fitted results to linearized Langmuir isotherm, (c) fitted results to linearized Freundlich isotherm for Pb sorption on polyethylene (PE) microplastics (MPs), (d) data obtained from equilibrium sorption experiments, (e) fitted results to linearized Langmuir isotherm, (f) fitted results to linearized Freundlich isotherm for Pb sorption on polyvinyl chloride (PVC) MPs, (g) data obtained from equilibrium sorption experiments, (h) fitted results to linearized Langmuir isotherm, (i) fitted results to linearized Freundlich isotherm for Pb sorption on polystyrene (PS) MPs. The MPs (fragments of <125 m) were prepared from PE, PVC, and PS pellets without additives

constant (L mg^{-1}), K_F is the Freundlich constant ($mg^{1-(1/n)}$ $L^{1/n}$ g^{-1}), and n_F is the heterogeneity factor (unitless).

2.5 Microtox® assay

With the Pb or Cu sorption experiments using the PE fragments with additives, the changes in the acute toxic effects of the Pb or Cu solutions before and after sorption on MPs were compared to assess the effect of sorption-driven change in the metal concentration of the supernatant on toxicity. The toxic effects were determined by running a Microtox® assay following the 81.9% basic test procedure provided by the manufacturer. Briefly, the bioluminescent bacteria, *Aliivibrio fischeri*, were exposed to the Pb or Cu solutions before and after sorption experiments for 15 min. The changes in the bioluminescence were measured using the MicrotoxLX analyzer (Modern Water, USA) and the half maximal effective concentration (EC50) were determined using triplicate samples.

3. Results and discussion

3.1 Effect of different types of MPs on Pb sorption

The results of the Pb sorption on different types of MPs were plotted in Fig. 1. The isotherm parameters and regression coefficients are shown in Table 1. The Langmuir isotherm model assumes that the adsorption takes place in a monolayer at the surface and there is no interaction between adsorbates (Al-Ghouti and Da'ana 2020). On the other hand, the Freundlich isotherm model assumes multilayer sorption (Al-Ghouti and Da'ana 2020). Based on the R^2 values, the Pb sorption on PE fitted the Fruendlich isotherm model better than the Langmuir isotherm model, while the opposite was observed with PS (Table 1). With PVC, both isotherm models can be used to describe the Pb sorption behavior with high R^2 values (0.9684-0.9693) (Table 1). Previous study observed similar Pb sorption behavior on PVC (particle size = 75-89 μm) (Lin *et al*. 2021).

Table 1 Langmuir and Freundlich isotherm parameters for Pb sorption on polyethylene (PE), polyvinyl chloride (PVC), and polystyrene (PS) microplastics (MPs), which were fragments prepared from the pellets without additives

	Langmuir			Freundlich		
	Q_m (mg g^{-1})	K_L (L mg^{-1})	R^2	K_F ($mg^{1-(1/n)}$ $L^{1/n}$ g^{-1})	n_F	R^2
PE	0.1904	0.0677	0.1965	0.0136	1.2594	0.7732
PVC	0.4536	1.9774	0.9684	0.2417	2.6759	0.9693
PS	0.0198	2.5530	0.8132	0.0139	5.0075	0.3430

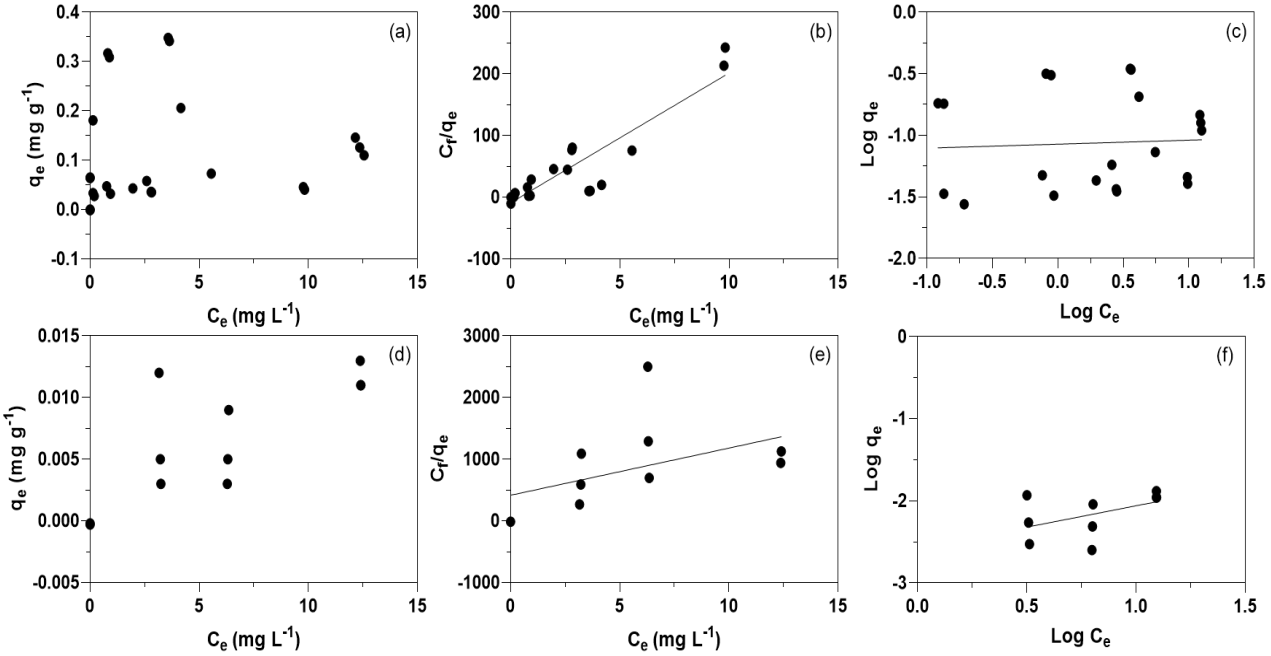

Fig. 2 (a) Data obtained from equilibrium sorption experiments, (b) fitted results to linearized Langmuir isotherm, (c) fitted results to linearized Freundlich isotherm for Cu sorption on polyethylene (PE) films (<5 mm × <5 mm), d) data obtained from equilibrium sorption experiments, (e) fitted results to linearized Langmuir isotherm, and (f) fitted results to linearized Freundlich isotherm for Cu sorption on PE fragments (<125 μm)

With PVC and PS where the R^2 values for the Langmuir isotherm model were relatively high, the maximum sorption capacity indicated by Q_m was greater for PVC than PS (Table 1). With PE and PVC where the R^2 values for the Freundlich isotherm model were relatively high, the K_F value related to the sorption capacity of sorbents was greater for PVC than PE (Table 1). When the Q_m values of the Langmuir isotherm model are compared, the Pb sorption capacities were in the order of PVC>PE>PS (Table 1). Similarly, one previous study reported that the Pb sorption capacities were in the order of PVC>PE>PS (Lin *et al*. 2021), and another study reported that the sorption capacities were in the order ot PVC>PE (Gao *et al*. 2019). Such differences in the Pb sorption on different plastics can be attributed to the different surface properties (Brennecke *et al*. 2016).

The n_F value of the Freundlich isotherm model is related to the magnitude of the sorption driving force, and high n_F value (>1) indicates greater sorption driving force at high concentrations but less force at lower concentrations (Park *et al*. 2015). Also, n_F >1 indicates physical sorption process, while n_F <1 indicates chemical sorption process (El Nemr *et al*. 2008). The n_F value for PE and PVC obtained in this study were greater than 1, and this suggests that physical Pb sorption process was favored over the chemical Pb sorption process (Vargas *et al*. 2012). Similarly, physical sorption processes were favored for the Pb and Cd sorption on PE and PVC (Ju *et al*. 2021).

3.2 Effect of different shapes of PE MPs on Cu sorption

The results of the Cu sorption on different shapes of PE MPs were plotted in Fig. 2. The isotherm parameters and regression coefficients are shown in Table 2. The Cu sorption on the PE films was fitted better to the Langmuir isotherm model than the Freundlich isotherm model, while the Cu sorption on the PE fragments was fitted to the Langmuir and Freundlich isotherm models with relatively low R^2 values of 0.2378-0.2530 (Table 2). When the Langmuir isotherm model parameters were compared, the PE films had a higher Q_m value than the PE fragments (Table 2). But the K_L value was higher for the PE fragments than the PE films (Table 2), and the higher K_L value indicates higher sorption strength (Sparks 2003). These

Table 2 Langmuir and Freundlich isotherm parameters for Cu sorption on polyethylene (PE) microplastic films and fragments

	Langmuir			Freundlich		
	Q_m (mg g^{-1})	K_L (L mg^{-1})	R^2	K_F ($mg^{1-(1/n)}$ $L^{1/n}$ g^{-1})	n_F	R^2
PE film	0.0476	-2.4266	0.8344	0.0849	30.8642	0.0030
PE fragments	0.0131	0.1834	0.2530	379.7519	1.9223	0.2378

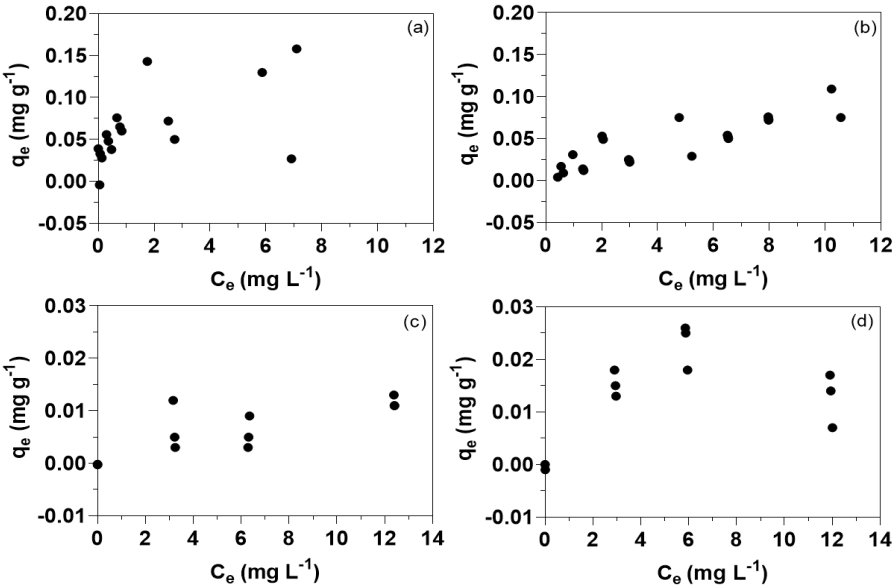

Fig. 3 Data obtained from equilibrium sorption experiments (a) for Pb on polyethylene (PE) fragments with additives, (b) for Pb on PE fragments without additives, (c) for Cu on PE fragments with additives, and (d) for Cu on PE fragments without additives

suggest that the PE films can adsorb more Cu than the PE fragments, but the sorption strength was greater for the PE fragments than the PE films.

3.3 Effect of the presence of additives on Cu and Pb sorption

The PE fragments prepared from PE pellets without additives and PE beads with additives were used to compare the Cu and Pb sorption. The sorption results are shown in Fig. 3 and the isotherm parameters and regression coefficients are shown in Table 3. When the sorption capacities of Pb and Cu are compared, the higher Q_m values for Pb suggest that the Pb sorption capacity of the PE fragments was greater than the Cu sorption capacity (Table 3). Both the Pb and Cu sorption capacities tend to be greater in the absence of additives as well (Table 3). Previous study also reported that the adsorption capacities of MPs can be promoted or hindered by the presence of additives such as flame retardants (Hahladakis *et al*. 2018). The sorption of contaminants on plastics can be affected by the crystallinity of plastics, which depends on the types and amount of additives in the plastics (Fred-Ahmadu *et al*. 2020).

The Pb sorption on the PE fragments without additives was fitted to the Freundlich isotherm model better than the Langmuir isotherm model, while the opposite was observed with the Pb sorption on the PE fragments with additives (Table 3). The Freundlich isotherm model could fit the Pb sorption on the PE fragments with and without additives with moderate R^2 values of 0.5609-0.7732 (Table 3). When the Freudlich isotherm parameters are compared, the K_F value related to the sorption capacity was greater for the PE fragments with additives (Table 3). Regardless of the presence of additives, the n_F values of >1 suggest that the Pb sorption on the PE fragments favored physical sorption processes over the chemical sorption processes (Table 3) (El Nemr *et al*. 2008).

With the Cu sorption, both the Langmuir and Freundlich isotherm models fitted the Cu sorption data with low R^2 values of 0.2378-0.3686 (Table 3). The Q_m values of the Langmuir isotherm model were similar regardless of the presence of additives, but the K_L value of the Langmuir isotherm model related to the sorption strength and the K_F value of the Freundlich isotherm model related to the sorption capacity were higher for the PE fragments with additives suggesting greater sorption capacity (Table 3).

Fig. 4 shows the changes in the toxic effects of the Pb and Cu solutions before and after sorption on PE fragments with additives. Fig. 4(a) shows that the toxic effect of the Pb solution decreased after sorption on the PE fragments. The highest toxic effects at the highest concentration tested were decreased from about 92% to about 17% (Fig. 4(a)). The estimated EC50 value of the Pb solution before sorption was 2.5% (i.e., only 2.5% (v/v) of the initial

Table 3 Langmuir and Freundlich isotherm parameters for Pb and Cu sorption on polyethylene (PE) microplastic fragments with and without additives

Heavy metal	Additives	Langmuir			Freundlich		
		Q_m (mg g^{-1})	K_L (L mg^{-1})	R^2	K_F ($mg^{1-(1/n)}$ $L^{1/n}$ g^{-1})	n_F	R^2
Pb	Present	0.1364	1.5124	0.7012	0.0693	4.662	0.5609
	Absent	0.1904	0.0677	0.1965	0.0136	1.2594	0.7732
Cu	Present	0.0131	0.1834	0.2530	379.7519	1.9223	0.2378
	Absent	0.0162	-3.4275	0.3040	0.0087	2.5465	0.3686

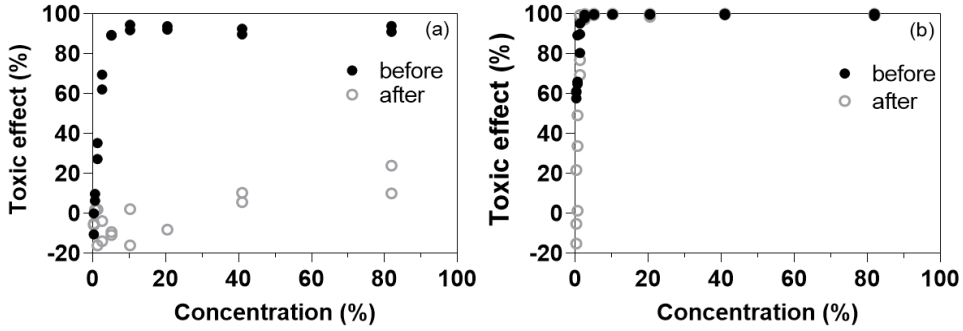

Fig. 4 Changes in the toxic effects on *Aliivibrio fischeri* after 15 min exposure to (a) Pb solution before and after sorption on polyethylene (PE) fragments with additives and (b) Cu solution before and after sorption on PE fragments. The concentrations were expressed as the percentage (v/v) of the initial solution in order to compare the toxicity changes before and after sorption

solution can decrease the bioluminescence by 50%), while the EC50 value after sorption was >100%. On the other hand, with the Cu solution, the toxic effects before and after sorption were similarly high at the highest concentration (Fig. 4(b)). However, the toxic effects at lower concentrations were lower after sorption than before sorption (Fig. 4(b)). The estimated EC50 value of the Cu solution before sorption was 0.11%, and the EC50 value after sorption was increased by about 8 times (i.e., 0.84%). This suggests that the Cu sorption on the PE fragments also reduced the toxic effects, but not as significantly as the Pb solution. These changes in the toxicity can be attributed to the sorption of Pb and Cu on the PE fragments reducing the available Pb or Cu. Although the Langmuir isotherm model was not fitted to the Pb and Cu sorption on the PE fragments with high R^2 values, the estimated Q_m values can support the differences in the toxicity changes. The Q_m value for the Pb solution was higher (i.e., 0.1904 mg g^{-1}) (Table 1) than that for the Cu solution (i.e., 0.0131 mg g^{-1}) (Table 2).

4. Conclusions

Various plastic products used in the agricultural environment end up generating MPs and the MPs generated can interact with other contaminants present in the environment. Various plastic types are used in the agricultural environment, and they end up in various shapes of MPs. But the studies on effect of different plastic properties (e.g., types, shapes, presence of additives) on the contaminant sorption capacities are limited. In this study, Cu and Pb, the commonly found heavy metals in the environment, were used to investigate the sorption capacities of MPs. The results show that the Pb sorption capacities were different for different types of MPs, and the Pb sorption capacity increased in the order of PS<PE<PVC. When different shapes (i.e., films and fragments) of the PE MPs were compared for the Cu sorption, the estimated sorption capacity was greater for the PE films, but the sorption strength was greater for the PE fragments. For the PE fragments, the Pb sorption capacity was greater than the Cu sorption capacity by 10-12 times. The estimated isotherm parameters related to the sorption capacity suggest that the Pb and Cu sorption capacities were greater for the PE fragments with additives than the PE fragment without additives. After the sorption of Pb or Cu, the toxic effects of the Pb or Cu solutions were decreased, and this suggests that the toxic effects of contaminants can be affected by the co-presence of MPs in the environment. Overall, the results show that different types and shapes of MPs and the presence of additives can affect the heavy metal sorption capacities of MPs. Therefore, more studies are needed to explore the effect of a wide range of properties of MPs found in the environment on the interaction with other contaminants (e.g., heavy metals) co-existing in the environment.

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