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Kinetics and Size Effects on Adsorption of Cu(II), Cr(III), and Pb(II) Onto Polyethylene, Polypropylene, and Polyethylene Terephthalate Microplastic Particles

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Due to its small size, large specific surface area and hydrophobicity, microplastics, and the adsorbed contaminants may together cause potential negative effects on ecosystems and human beings. In this study, kinetics and size effects on adsorption of Cu(II), Cr(III), and Pb(II) onto PE, PP and PET microplastic particles were explored. Results indicated that the PE and PET microplastics have the higher adsorption capacity for Cu(II), Cr(III), and Pb(II) than that for PP microplastic. The adsorption capacity was affected by microplastic types and metal species. Among the three metals, Pb(II) had the largest adsorption amount on microplastic particles, especially on PET particles. Moreover, the adsorption capacities of microplastics increase with the decrease of particle size. The metal adsorption capacity of <0.9 mm microplastics is greater than that of 0.9–2 mm and 2–5 mm microplastics. The size effect on metal adsorption was largest for PE microplastic. More attention should be paid in case of the coexistence of heavy metals and tiny PE and PET microplastics in the environment.

Keywords: microplastics, kinetics, metal, adsorption, size effect

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INTRODUCTION

Microplastics have already posed potentially risk for human health through transmission and accumulation in food chain (Yang et al., 2015; Xu et al., 2019) as they have already been widely detected in food (Liebezeit and Liebezeit, 2013, 2014; Yang et al., 2015). In the future, the environmental exposure risk of microplastic may be elevated as the plastic production is expected to increase to 318 million tons annually in 2050 (Neufeld et al., 2016). Furthermore, environmental microplastics could be a carrier for heavy metals transport from river to sea due to its small size, large specific surface area and hydrophobicity (Wang et al., 2017). As commonly detected pollutants in the environment (Zhang et al., 2018), heavy metals such as Cu, Cr, and Pb were also frequently detected in environmental microplastics (Selvam et al., 2021), and the metal concentration of microplastics was even similar or higher than that of the sediment phase (Ashton et al., 2010). It indicated that microplastics were able to enhance the mobility of heavy metal along river-coast-sea system. Once the metal-contained microplastics are ingested by aquatic organisms, these metals

may be released in the organism, causing further damage to the function of the organism. Then threaten human health via gradual accumulation in food chain (Fries et al., 2013). Therefore, the potential risk of microplastics and the heavy metal to freshwater-marine ecosystems would be both intensified. Therefore, to investigate the affinity of heavy metals to microplastics is essential to estimate the coexisting toxicity of heavy metals and microplastics in aqueous environments (Xu et al., 2018).

Although microplastics have ability to adsorb heavy metals (Koelmans et al., 2016; Wang et al., 2019; Fu et al., 2021), the adsorption capacity varies with the type of microplastics and heavy metals because of the difference in physicochemical properties of various microplastics and heavy metals. For example, polystyrene and film microplastic have greater adsorption capacity for Cu(II) than polyvinyl chloride, polyethylene, fishing line fibers and bottle cap particles, due to the conducive physicochemical properties of film microplastic (Almeida et al., 2020; Gao et al., 2021a). Compared with Cu and Cd, Pb showed the higher affinity to microplastics, because it is more likely to efficiently bind to function group on microplastics to promote the adsorption (Gao et al., 2019, 2021b). In addition, particle size is the generally essential factor influencing adsorption. In the environments, microplastics will be further fragmented into smaller part due to environmental dynamics, thereby affecting the adsorption capacity of heavy metals on microplastics (Gao et al., 2019; Zhang et al., 2020).

Therefore, it is important to investigate how microplastic size and type affect the interaction with heavy metals, which heavy metal has the most potential to be absorbed onto microplastics, and how the interaction will change with time. Adsorption kinetics are a conventional method to identify the temporal change of adsorption process, and the model parameters would attribute to reveal the possible adsorption mechanism (Almeida et al., 2020; Purwiyanto et al., 2020). Here, polyethylene (PE), polypropylene (PP) and polyethylene terephthalate (PET), which are three mostly used and typical types of plastics in the world (The Essential Chemical Industry (ECI), 2016a,b, 2017), are selected to study the adsorption kinetics and size effect for the three typical metal ions of Cu(II), Cr(III), and Pb(II) to test the hypotheses: (a) different temporal change in metal adsorption for different microplastics; and (b) larger metal adsorption for smaller microplastic particles.

MATERIALS AND METHODS

Chemicals and Materials

$Cu(NO_3)_2$, $Cr(NO_3)_3$, and $Pb(NO_3)_2$ were purchased from Aladdin Bio-Chem Technology Corporation (Shanghai, China). HNO_3 was purchased from Bohua Chemical Reagent Corporation (Tianjin, China). All chemicals were analytical grade or higher purity. PE, PP, and PET pellets with particle size of 5mm were purchased from Yousuo Chemical Technology Corporation (Shandong, China). Before use, the PE, PP, and PET pellets were crushed using a high-speed crusher. The crushed microplastic particles were then sequentially sieved through 20-, 10-, and 4- mesh screens in order to separate the particle sizes 2–5, 0.9–2, and <0.9 mm. The morphology of PE, PP, and PET microplastics were observed with a scanning electron microscope (Tescan Mira 4). To prevent contamination, all the lab materials were soaked in 10% (v/v) HCl solution for at least 48h, rinsed at least three times with deionized water (conductivity < 0.1 mS cm^{-1}) and dried in an oven at 50^{o}C.

Adsorption Experiments

The first experiment was to investigate temporal change of metal adsorption onto microplastics. Three microplastics (PE, PP, and PET) with same particle size < 0.9 mm were mixed with 50 mL solutions of Cu(II) with concentration of 5 mg L^{-1} in centrifuge tubes. The adsorption of Cr(III) and Pb(II) were also conducted simultaneously at the same condition. The medium is the deionized water. Samples were shaken at 150 r/min in a constant temperature water bath shaker at room temperature (∼25^{o}C). Sub-samples after 1, 2, 4, 8, 24, 72, 120, 168, 240, 312, and 384 h were taken, respectively.

The second experiment was to investigate the influence of microplastic particle size on adsorption. PE, PP, and PET microplastics with particle size 2–5, 0.9–2, and <0.9 mm were used. A series of centrifuge tubes, respectively containing 0.5 g microplastic with different size and 50 mL solutions of Cu(II) with concentration of 5 mg L^{-1}, were shaken for 240 h at 150 r/min in a constant temperature water bath shaker at room temperature (∼25^{o}C). At the same time, the adsorption of Cr(III) and Pb(II) were also conducted at the same condition. The medium is the deionized water.

At the terminal of shaking step, the mixture was immediately filtered with filter paper with a pore size of 15 to 20 μm. The trapped microplastics were collected, then were dried and transferred to a series of 10 mL centrifuge tubes. Then, 5 mL 2% HNO_3 was added to these tubes and ultrasound for 10 min to extract metal ions from microplastics. Finally, the mixture after ultrasonic was filtered with a syringe filter and the filtered solution was transferred to a clean PP centrifuge tube for quantification analysis. An inductively coupled plasma mass spectrometry (ICP-MS, Elan DRC-e, PerkinElmer) was used to analyze the heavy metal contents using the certified reference material (CRM). The detection limits for the three metals are 1 ppt and the recoveries for all are above 90%. All the treatments were in duplicate. The amount of heavy metal adsorbed by per unit mass of microplastic (q) could be calculated by Eq. (1).

$$q = \frac{VC}{m} \quad (1)$$

where, m (g) was the mass of microplastics used in adsorption, V (L) was the volume of the added solution with 2% HNO_3, C (μg L^{-1}) was the concentration of heavy metals after ultrasonic, respectively.

Kinetic Models

Four kinetic models were used to describe the kinetic adsorption of Cu(II), Cr(III), Pb(II) onto PE, PP, and PET microplastics.

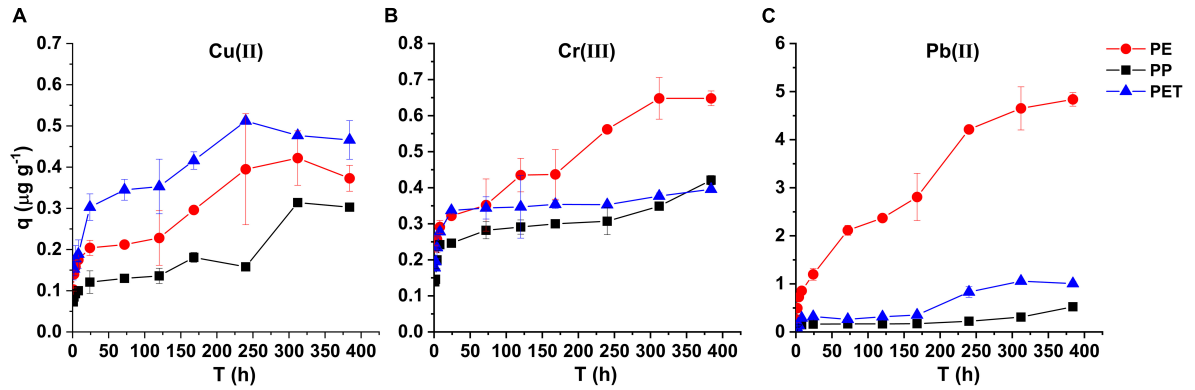

FIGURE 1 | (A) The Cu(II), **(B)** Cr(III), and **(C)** Pb(II) adsorption of PE, PP, and PET microplastics with a particle size of <0.9 mm at different adsorption time.

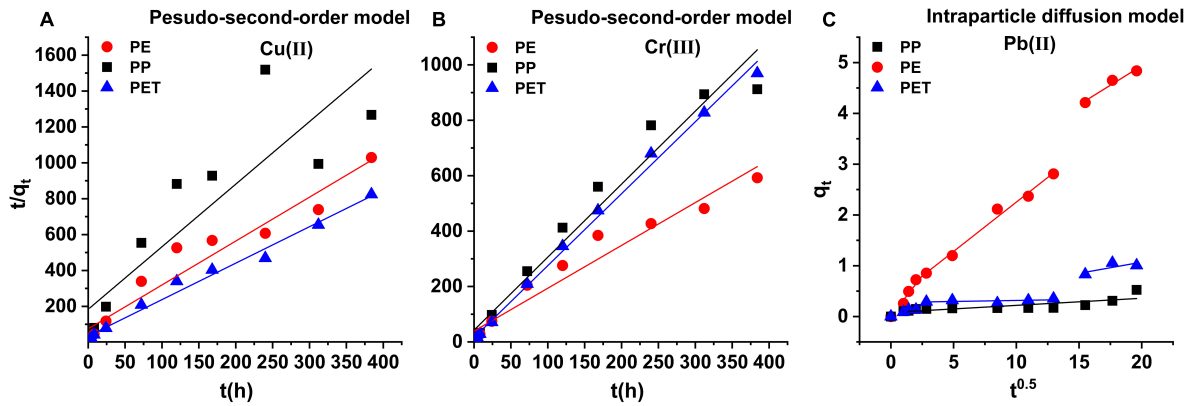

FIGURE 2 | (A) The pseudo-second-order kinetic model for Cu(II), **(B)** Cr(III) adsorption on PE, PP, and PET microplastics, and **(C)** the intra-particle diffusion model for Pb(II) adsorption on PE, PP and PET microplastics.

TABLE 1 | The fitting parameters of different models of Cu(II), Cr(III), and Pb(II) adsorbed onto PE, PP, and PET microplastics, respectively.

		Cu(II)			Cr(III)			Pb(II)		
		PE	PP	PET	PE	PP	PET	PE	PP	PET
Pesudo-second-order model	q_e (μg g^{-1})	0.402	0.278	0.488	0.649	0.380	0.385	5.128	0.370	1.04
	k_2 (g/(μg·h)$^{-1}$)	0.0025	0.0005	0.0081	0.0107	0.0034	0.0094	2.217	0.0009	0.0123
	R^2	0.94	0.78	0.98	0.95	0.96	0.99	0.88	0.67	0.52
Pesudo-first-order model	q_e (μg g^{-1})	0.303	0.287	0.147	1.71	0.267	0.151	6.97	0.542	1.21
	k_1 (h^{-1})	0.0072	0.0076	0.007	0.014	0.006	0.0076	0.015	0.0066	0.104
	R^2	0.69	0.67	0.61	0.82	0.71	0.82	0.76	0.48	0.60
Elovich model	a (μg g^{-1})	21.8	32.3	16.5	15.7	27.4	29.8	1.09	26.7	7.63
	B (μg g^{-1} h^{-1})	0.046	0.031	0.061	0.065	0.037	0.034	0.73	0.038	0.131
	R^2	0.80	0.60	0.93	0.79	0.88	0.94	0.84	0.39	0.59
Intraparticle diffusion model	$k_{p,1}$ (μg g^{-1} h$^{-0.5}$)	0.111	0.015	0.019	0.022	0.0602	0.056	0.320	0.0464	0.014
	C_1 (μg g^{-1})	0.112	0.058	0.154	0.209	0.0727	0.120	0.0042	0.0252	0.078
	R^2	0.90	0.79	0.92	0.96	0.95	0.99	0.89	0.80	0.76
	$k_{p,2}$ (μg g^{-1} h$^{-0.5}$)					0.0062	0.002	0.194		0.0018
	C_2 (μg g^{-1})					0.222	0.327	0.311		0.149
	R^2					0.95	0.95	0.98		0.83
	$k_{p,3}$ (μg g^{-1} h$^{-0.5}$)					0.0276	0.0105	0.153		0.0727
	C_3 (μg g^{-1})					−0.126	0.191	1.87		−0.925
	R^2					0.93	0.99	0.93		0.85

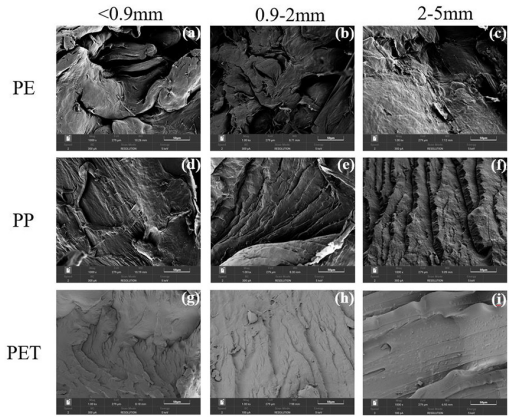

FIGURE 3 | (a–i) The morphology of PE, PP and PET microplastics.

The pseudo-second-order kinetic model:

$$\frac{t}{q_t} = \left(\frac{1}{k_2 q_e^2}\right)\frac{t}{+}q_e \quad (2)$$

The pseudo-first-order kinetic model:

$$\ln(q_e - q_t) = k_1 t + lnq_e \quad (3)$$

The Elovich kinetic model:

$$q_t = blnt + \frac{ln(ab)}{b} \quad (4)$$

The intra-particle diffusion model:

$$q_t = k_p t^{0.5} + C \quad (5)$$

where, q_t ($\mu g\ g^{-1}$) is the adsorption amount at the time of t (h); q_e ($\mu g\ g^{-1}$) is the saturated adsorption capacity of heavy metals at equilibrium; k_1 (h^{-1}) is the reaction rate constant of pseudo-first-order equation at equilibrium; k_2 ($g\ \mu g^{-1}\ h^{-1}$) is the reaction rate constant of the pseudo-second-order equation at equilibrium; a ($\mu g\ g^{-1}$) and b ($\mu g\ g^{-1}\ h^{-1}$) are the parameters of the Elovich equation; k_p ($\mu g\ g^{-1}\ h^{-0.5}$) is the constant of intra-particle diffusion model, C ($\mu g\ g^{-1}$) represents a conception about the thickness of boundary layer, describing the influence of thickness of boundary layer on adsorption.

RESULTS AND DISCUSSION

Metal Adsorption Kinetics

The kinetics experiments results were shown in **Figure 1**. The maximum of Cu(II) and Cr(III), Pb(II) adsorption were 0.51, 0.64, and 4.78 mg g^{-1} for PET, PE and PE, respectively. For all the three metals, the adsorption capacity on PP particles was the lowest. The adsorption of PE, PP, and PET particles increased rapidly in the initial 24 h, and then changed slowly. In general, the adsorption rates and adsorption capacities followed the orders of PET > PE > PP for Cu(II), and PE > PET > PP for Cr(III) and Pb(II).

As shown in **Figure 2**, the kinetics of Cu(II) and Cr(III) adsorption onto the PE, PP, and PET microplastics were well regressed by the pseudo-second-order model (**Table 1**). The derived equilibrium adsorption capacities (q_e) of Cu(II) and Cr(III) for PP microplastic were the lowest, which was the same with the experimental results in **Figure 1**. It may be attributed to no functional group on PP microplastic compared with PET microplastic and the smoother surface of PP microplastic than PE microplastic (**Figure 3**). The adsorption capacity of Cu(II) for PET microplastic is greater than that for PE microplastic, while the adsorption capacities of Cr(III) for PET microplastic is smaller than that for PE microplastic. However, other researchers found that sequence of adsorption capacity was PE > PP > PET for both Cu(II) and Cr(III) (Godoy et al., 2019). The differences may be because the microplastics they used were from daily objects and may be aged. This may suggest that the adsorption capacity of heavy metals on microplastic greatly varies with the change of microplastics surface. In **Table 1**, the values of k_2 were lower than 0.01 g (μgh) $^{-1}$ for Cu(II) and Cr(III) adsorption. It did not only indicate that the adsorption rate was proportional to the number of unoccupied sites (Fan et al., 2021), but also revealed the adsorption of Cu(II) and Cr(III) onto the microplastics were a slow process, especially for the virgin microplastics with relatively homogeneous smooth surface (Li et al., 2019; Oz et al., 2019; Wang et al., 2020).

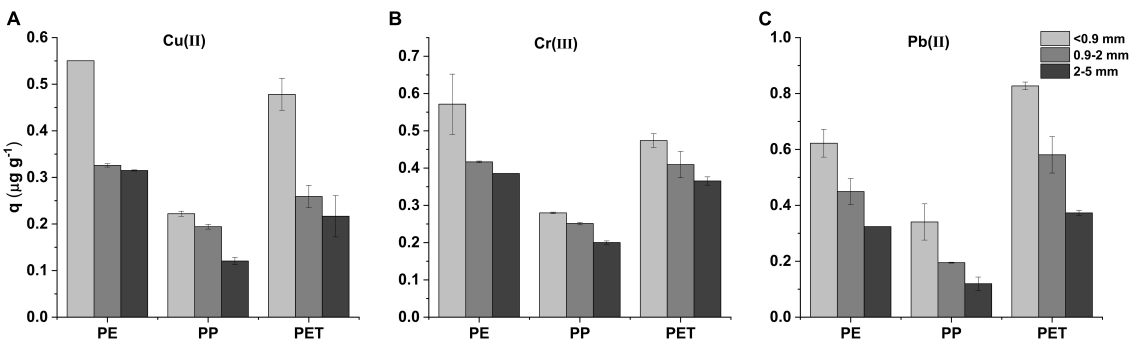

FIGURE 4 | (A) The Cu(II), **(B)** Cr(III), and **(C)** Pb(II) adsorption capacities of PP, PE, and PET microplastics with different particle sizes in 240 h.

Turner and Holmes (2015) and Wang et al. (2020) also found that the interaction between metals and microplastics was a long-term process even for the aged microplastics. It indicated that the microplastics might continue to accumulate heavy metals when the interaction time is long. However, further evidence is needed.

There was obvious discrepancy in Pb(II) adsorbed on PE, PP, and PET microplastics (**Figures 1**, **2**). The adsorption amount of Pb(II) on PE microplastic was the highest, which may attribute to the higher crystallinity, high pore volume and rough surfaces of PE microplastic (Wang and Wang, 2018; Zou et al., 2020). It indicated that the crystallinity of microplastic may be one of the essential factors influencing Pb(II) adsorption, even more important than function group for the virgin microplastics. The kinetics of Pb(II) adsorption on PE, PP, and PET microplastics were well fitted by the intra-particle diffusion model ($R^2 \geq 0.76$ in **Table 1**), implying the inter-particle diffusion process was the rate-controlling step. The negative influence of the boundary layer on adsorption over time decreased to the lowest ($C_3 < 0$) explained the keep growing in amount of adsorbed Pb(II) on PET microplastic. Although the order of C on PE microplastic was $C_1 > C_2 > C_3$, the adsorption amount continuously increased with time. Turner and Holmes (2015) also found the same trend when the added Pb(II) concentration was 5 mg L^{-1}. It implied that Pb(II) had strong prosperity of affinity and temporal accumulation on PE microplastic. However, the adsorption can quickly achieve the equilibrium at about 48h in the seawater medium (Holmes et al., 2012), because the ions existence would fasten the adsorption process and change the temporal procedure of adsorption.

Size Effect on Metal Adsorption

The results shown in **Figure 4** validated the heavy metal absorption on microplastics decreased with increasing particle size. With the decrease of microplastic size from 2–5 mm to <0.9 mm, the adsorption amount increased about 1.8–2.2, 1.3–1.5, and 1.94–2.83 times for Cu(II), Cr(III), and Pb(II), respectively. In addition, the amount of adsorbed Cr(III) varied more slightly with the particle size. Namely, the effect of particle size of PP microplastic on metal adsorption was relatively low.

The phenomenon may attribute to the more complex morphology and higher specific area with decrease of particle size (**Figure 3**), which can lead more unoccupied site for adsorption. For Cu(II) and Pb(II), the observed adsorption variations with particle size implied that the adsorption on microplastic was considerably related to the porosity. Compared with Cu(II) and Pb(II), the influence of particle size on Cr(III) adsorption was relatively small, which is similar to the tendency observed in other studies (e.g., Zhang et al., 2021). It may be attributable to the insensitive response of Cr(III) adsorption to the stratification variation of the microplastic surface. The relatively low influence of particle size on PP microplastic adsorption profitably emphasized the significance of crystallinity and function group.

According to the experimental results above, it may be inferred that the adsorption amount of microplastics to other metals may also possibly increase with decrease of particle size. Namely, microplastics with a smaller particle size in the environment may cause higher environmental risks as a carrier of heavy metals (Thompson et al., 2004; Zhang et al., 2020). With the aging process of microplastics in natural environment, such as UV-irradiation, acid and alkali corrosion, particle crushing, biofouling, it would become more toxic to the environment. This means the results in this study may be regarded as the lowest metal amounts absorbed by microplastics in the natural environment.

All the three heavy metals can be accumulated increasingly with time onto the three microplastics. The PET microplastic has the relatively rapid and strong ability to adsorb Cu(II) and PE microplastic has the relatively rapid and strong ability to adsorb Cr(III) and Pb(II). It means that the virgin microplastic PE and PET can be a conducive carrier for heavy metal transport in the environment and their environmental toxicity would be magnified, especially for the combination of Pb(II) and PE. The risk to environmental security would be further elevated due to the aging process of PP and PET in the environment (Han et al., 2021). Therefore, more attention should be paid to PE and PET microplastics if metal contaminants exist in the aqueous system.

DATA AVAILABILITY STATEMENT

The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

AUTHOR CONTRIBUTIONS

XH and SW: investigation, data curation, validation, and writing-original draft preparation. XY: writing, reviewing, and editing. RV: conceptualization, writing, reviewing, and editing. JF, LZha, WM, and LZhu: resources, writing, reviewing, and editing. XL: conceptualization, supervision, writing, reviewing, and editing. All authors contributed to the article and approved the submitted version.

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