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论文题目: Study on the Adsorption Characteristics of Tilmicosin by Polyethylene Microplastics

Study on the Adsorption Characteristics of Tilmicosin by Polyethylene Microplastics

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Abstract: Microplastics and antibiotics are two types of emerging contaminants widely detected in the aqueous environment. Microplastics as solid particles have the potential to adsorb dissolved state antibiotics in water, thus altering the behavior and fate of antibiotics in the water column. In this study, the adsorption behavior of polyethylene (PE) microplastics on TIL was systematically investigated using Tilmicosin (TIL) as the target antibiotic. The results showed that the adsorption of TIL by PE was superior to that of Pefloxacin, Difloxacin, Ciprofloxacin, Enrofloxacin, and Lincomycin, etc. The initial reaction rate of TIL adsorption by PE was fast and the process was in accordance with the quasi-secondary kinetic model, which was the result of multiple processes, including boundary liquid film diffusion, intraparticle diffusion, and binding to the adsorption site. The adsorption isotherms are well fitted by the Freundlich model, indicating that the adsorption of PE on TIL is a multilayer non-uniform process. Increasing temperature and decreasing particle size can promote the adsorption of PE on TIL to some extent, while increasing salinity will decrease the adsorption of TIL.

Keywords: microplastics, antibiotics, adsorption

Content Index

Microplastics are organic polymers with a scale of 5 mm or less and can be generally classified into primary and secondary microplastics [1,2]. Primary microplastics are mainly found in personal care products such as toothpastes and facial cleansers, and can enter the water environment through household drainage systems with the rinsing process. Secondary microplastics are fragments formed by the splitting and degradation of large plastic and synthetic fibers under the action of ultraviolet radiation, wind and sand blowing, and water surface waves. Microplastics are able to migrate between terrestrial, freshwater and marine environments. Compared to bulk plastics, microplastics have a reduced ability to photodegrade, resulting in their persistence in the environment for hundreds, or even thousands, of years [1,2].

Microplastics are widespread around the world and their contact with the biosphere is inevitable. Because microplastics are small in particle size, not easily degradable, and ideal carriers of organic pollutants and heavy metals, numerous native plants, protozoa, zooplankton, fish, turtles, and birds are able to ingest microplastics and transfer and enrich them through food chains and food webs, ultimately posing health risks to organisms, including humans [1-3]. Because of this, humans are becoming increasingly concerned about microplastics as an emerging pollutant.

Pharmaceutical and personal care products (PPCPs) are also an emerging class of contaminants, with more than 4000 different substances ranging from tens of ng/L to even tens of mg/L already found in waters around the world (e.g., production of wastewater from pharmaceutical companies) [3-5]. PPCPs can be ingested by aquatic organisms and are mobilized and enriched along food chains and food webs, posing environmental risks. Microplastics present in the aqueous environment are ideal carriers of numerous contaminants due to their small size and relatively large surface area. Microplastics can interact with PPCPs, thus changing the distribution and morphology of PPCPs in water and creating new environmental health risks [6].

Antibiotic drugs are an important class of PPCPs. Residual antibiotics in environments such as natural water bodies and soils can harm microbial communities and induce the

formation of resistance genes [7]. The pathways of antibiotics in the aqueous environment are complex, and the general view is that adsorption is the key to influence the transport and transformation of antibiotics in the aqueous environment [8]. Antibiotics are ionic organic pollutants, hydrophilic in nature, and their physicochemical properties are significantly different from hydrophobic organic matter. Regarding the adsorption of organic pollutants by microplastics, previous studies have focused on hydrophobic organic pollutants, but there are fewer studies on hydrophilic organic pollutants [3,4]. The adsorption characteristics of Tylosin on polypropylene surface were studied by Jingwen Pang [9] et al. The adsorption characteristics of sulfamethoxazole on polyethylene surface were studied by Xu [4] et al. The adsorption characteristics of ciprofloxacin on polystyrene and polyvinyl chloride surfaces were studied by Liu [3] et al. The differences in the adsorption of ciprofloxacin between virgin and aged microplastic particles were compared. Kaina Zhang [10] et al. investigated the adsorption mechanism of polyethylene and polystyrene on hyoscyamine. However, there are many inconsistent findings in previous studies, and the commonality and properties of antibiotic adsorption by microplastics are unclear due to the wide variety of microplastics and antibiotics, which need to continue to be studied in depth.

Tilmicosin is a semi-synthetic macrolide antibiotic for livestock developed in the UK in the 1980s. Tilmicosin inhibits all gram-positive and some gram-negative bacteria, mycoplasma and spirochetes, and is particularly effective against livestock respiratory infections [11]. The widespread use of Tilmicosin has led to its detection in the environment, but it is not clear about its behavior once it enters the environment.

In this project, a typical microplastic, polyethylene (PE) is selected, and the adsorption kinetics and thermodynamics of Tilmicosin on PE surface is studied to further clarify the adsorption characteristics of Tilmicosin on microplastics. The results of the study will contribute to the understanding of the interactions and effects of emerging contaminants in the aqueous environment.

1 Materials and Methods

1.1 Experimental materials

Tilmicosin (TIL) and Difloxacin (DIF) were purchased from Dr. Ehrenstorfer, Germany. Tylosin (TYL) and Enrofloxacin (ENR) were purchased from Cayman Corporation, USA. Pefloxacin (PEF), ciprofloxacin (CIP), and lincomycin (LIN) were purchased from TRC, Canada. Polyethylene (180 μm, 550 μm) was purchased from Youngling Electromechanical Technology, Shanghai. Polyethylene (48 μm) was purchased from Sinopec. Sodium chloride (analytical purity) was purchased from Sinopharm Chemical Reagent Co. UV-visible spectrophotometer was DR6000 from Hash, USA. The constant temperature shaker was SKY-100B type from Shanghai Sukun Industrial Co. Ultrapure water was prepared by Milli-Q ultrapure water instrument.

1.2 Experimental method

1.2.1 Adsorption effects of polyethylene microplastics on various antibiotics

A stock solution of 10 mg/L of Tylosin (TYL), Pefloxacin (PEF), Difloxacin (DIF), Ciprofloxacin (CIP), Enrofloxacin (ENR), Lincomycin (LIN), and Tilmicosin (TIL) was prepared separately. Add 20 mL of each of these antibiotic solutions to a series of small glass vials with Teflon gasket caps, then add 50 mg of polyethylene (PE) to each vial and tighten the caps. Unless otherwise noted, the PE microplastics used in the experiments were 180 μm particles. All glass vials were placed in a constant temperature shaker and shaken at 25 °C and 190 rpm for 48 h. Afterwards, the samples were removed and filtered through a 0.45 μm microporous membrane and the filtrate was collected. The absorbance of each stock solution and filtrate was measured at the maximum absorption wavelength of each antibiotic determined by pre-experiment using UV-Vis spectrophotometer. 289 nm, 272 nm, 273 nm, 270 nm, 271 nm, 270 nm, 290 nm were tested for TYL, PEF, DIF, CIP, ENR, LIN, and TIL, respectively. The absorbance was calculated based on the change of The removal rate was calculated based on the change of absorbance. All experiments were set up with 2~3 parallel samples (same below).

1.2.2 Effect of microplastic addition on adsorption effect

A series of TIL solutions of 10 mL in a volume of 10 mg/L were prepared and divided into two groups. All TIL solutions were placed in a constant temperature shaker and shaken at 25 °C and 190 rpm. The samples were taken at the specified time intervals and quickly separated by solid-liquid separation through a 0.45 μm microporous filter membrane, and the absorbance of the filtrate was measured at 290 nm. The concentration of TIL in the filtrate was calculated from the TIL standard curve determined by the pre-experiment, and then the removal rate and adsorption amount were calculated.

1.2.3 Adsorption kinetics experiments

Prepare a 10 mg/L TIL solution. Add 30 mg PE to a series of 10 mL TIL solution, put into a shaker at 25 °C, 190 rpm, and take samples at 3, 5, 10, 30, 90, 120, 300, 600 and 1440 min to determine the adsorption amount.

1.2.4 Isothermal adsorption experiments

Add 30 mg of PE to a series of 10 mL of TIL solution. set the mass concentration of TIL to 5, 8, 10, 15 and 20 mg/L. Place in a shaker and shake at 25° C, 190 rpm for 24 h and measure the adsorption. The above steps were repeated, the shaker temperature was lowered, and the adsorption amount was measured after 24 h of shaking at 15 °C and 190 rpm.

1.2.5 Effect of particle size of microplastics on adsorption effect

A series of 10 mL of TIL solution at a concentration of 10 mg/L was divided into three groups. In the first group, 30 mg of PE with a particle size of 48 μm was added; in the second group, 30 mg of PE with a particle size of 180 μm was added; in the third group, 30 mg of PE with a particle size of 550 μm was added; the adsorption amount was determined by shaking at 25 °C and 190 rpm and sampling at the indicated time points.

1.2.6 Effect of salinity on adsorption effect

To a series of TIL solutions in a volume of 10 mL at a concentration of 10 mg/L, 0, 0.05, 0.15, 0.25, 0.30, and 0.35 g NaCl were added, and the solutions were mixed thoroughly

so that the background salinity was 0%, 0.5%, 1.5%, 2.5%, 3%, and 3.5%, respectively, and 30 mg PE was added to each. all samples were placed in a constant temperature shaker at The adsorption amount was measured after shaking for 24 h at 25°C and 190 rpm.

1.3 Data processing methods

Data were initially processed using Excel 2016 and then analyzed and plotted using Origin 2018. Removal rates were calculated according to Equation 1 and adsorption amounts were calculated according to Equation 2.

$$\text{removal efficiency} = \frac{(c_0 - c_t)}{c_0} \times 100\% \qquad \text{(Equation 1)}$$

$$q_t = \frac{(c_0 - c_t)\ V}{m} \qquad \text{(Equation 2)}$$

Where c_0 is the initial concentration (mg/L), c_t is the concentration at moment t (mg/L), V is the volume of the solution (mL), m is the mass of the adsorbent (g), and q_t is the amount of adsorption at moment t (mg/g).

2 Results and Discussion

2.1 Effect of polyethylene microplastics on the adsorption and removal of different antibiotics

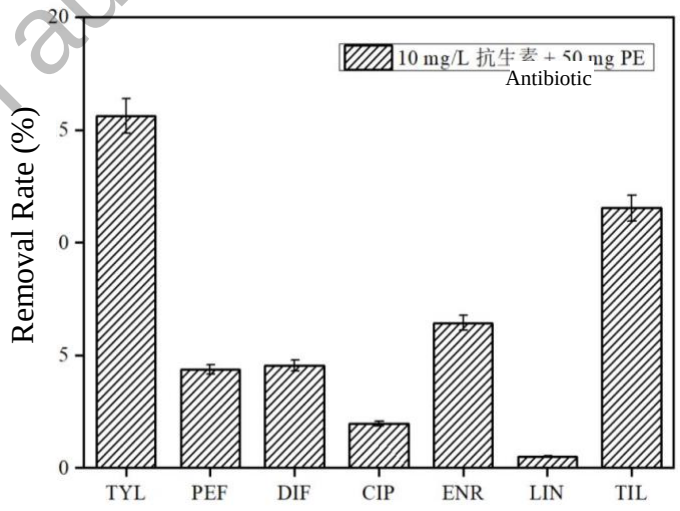

Figure 1. Adsorption and removal effect of PE on different antibiotics

The adsorption and removal of Tylosin (TYL), pefloxacin (PEF), Difloxacin (DIF), Ciprofloxacin (CIP), Enrofloxacin (ENR), Lincomycin (LIN), and Tilmicosin (TIL) from water by PE is shown in Figure 1. As can be seen from the figure, the removal rates of PE for TYL and TIL were 16% and 12%, respectively, which were higher than the other tested antibiotics. This indicates that the presence of microplastic particles in the aqueous environment is more likely to have an effect on the migration of TYL and TIL. Several previous studies have been done on the adsorption of TYL on polypropylene (PP) surfaces [9], so this study will subsequently focus on the adsorption behavior of TIL.

2.2 Effect of PE addition on TIL removal by adsorption

The effect of changing the PE amount on the TIL removal rate in water is shown in Figure 2. From the figure, it can be seen that when the initial concentration of TIL is the same, the higher the PE addition amount, the higher the TIL removal rate. After 24 h of reaction, the TIL removal rates corresponding to PE additions of 10, 30 and 50 mg were 18%, 21% and 22%, respectively. Since the effect of adding 30 mg and 50 mg PE on the final TIL removal rate was small, the PE addition amount was always set to 30 mg and the solid-liquid ratio was kept at 3:1000 in the subsequent experiments of this study.

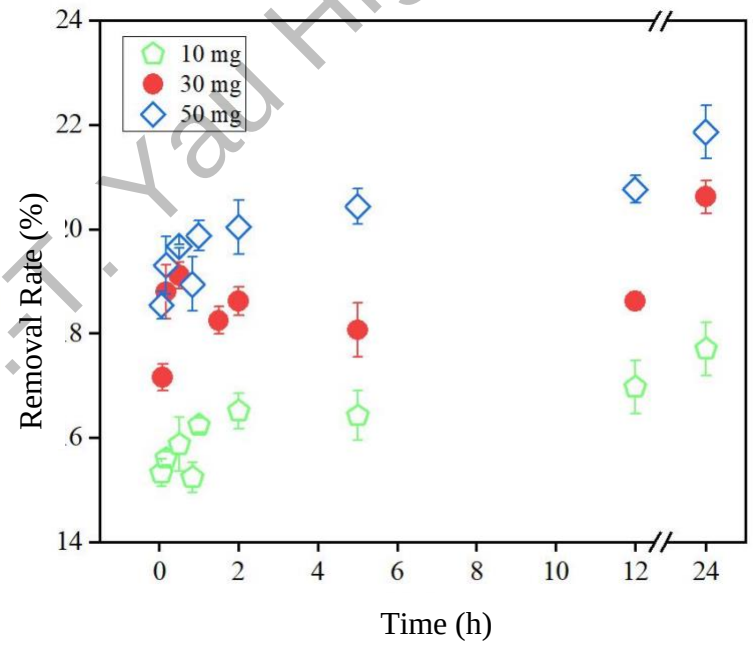

Figure 2. Effect of different PE additions on TIL removal rate

2.3 Kinetic properties of PE adsorption on TIL

The characteristics of the adsorption of PE microplastic particles on TIL with an initial concentration of 10 mg/L over time when the experiment was conducted at 25℃ for 24 h are shown in Figure 3. The adsorption amount grew very rapidly within 30 min at the beginning of the experiment, and the cumulative adsorption amount reached 89% of the total adsorption amount. The adsorption amount increased very slowly with time, and the reaction reached complete equilibrium after 24 h with the adsorption amount of 0.6943 mg/g.

The quasi-primary kinetic model (Eq. 3) and quasi-secondary kinetic model (Eq. 4) were used to fit the kinetic process of TIL adsorption on PE surface. The equations of the two models are as follows.

$$q_t = q_e(1-e^{-k_1 t}) \qquad \text{(Eq. 3)}$$

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \qquad \text{(Eq. 4)}$$

where q_t is the adsorption amount at moment t (mg/g), q_e is the equilibrium adsorption amount (mg/g), t is the reaction time (h), k_1 is the quasi-primary kinetic constant (h^{-1}), and k_2 is the quasi-secondary kinetic constant ($g \cdot mg^{-1} \cdot h^{-1}$).

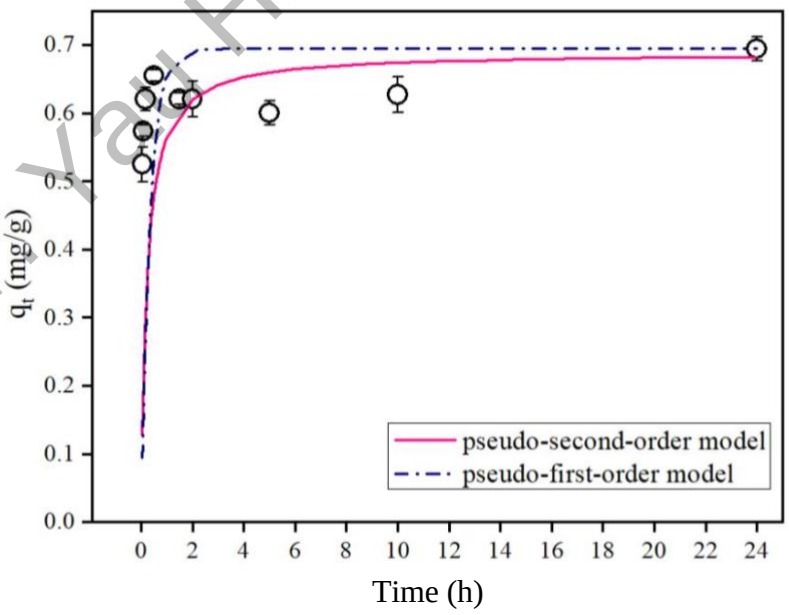

Figure 3. Adsorption kinetics of PE on TIL

The fitting results are shown in Figure 3 and Table 1. As can be seen from Table 1, the fitting of the quasi-secondary kinetic model (R^2 = 0.9976) is better than the quasi-first-order kinetic model (R^2 = 0.8945), which indicates that the whole adsorption process is the result of multiple adsorption stages acting together, such as the diffusion of antibiotics within the liquid film at the solid-liquid boundary to the outer surface of the microplastic, internal diffusion within the microplastic particles, and binding to the adsorption sites[10,12,13].

Table 1. Parameters for fitting the adsorption kinetics of PE on TIL

Microplastic	Quasi-primary kinetic model			Quasi-secondary kinetic model		
	q_e	k_1	R^2	q_e	k_2	R^2
PE	0.694	2.905	0.8945	0.689	6.567	0.9976

2.4 Isothermal adsorption properties of PE on TIL

The adsorption isotherm can reveal the equilibrium state of adsorbate in solution and adsorbent, and the change of temperature may affect this equilibrium state, so studying the adsorption isotherm first at different temperatures can further clarify the mechanism of adsorption occurrence. As shown in Figure 4, the adsorption of TIL by PE increased from 1.15 mg/g to 1.68 mg/g as the temperature increased from 15℃ to 25℃, indicating that the higher temperature was favorable for the adsorption of TIL by microplastics.

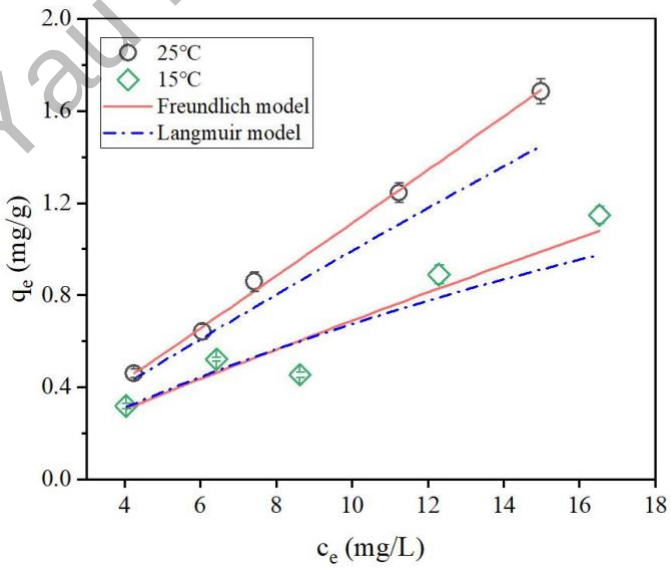

Figure 4. Adsorption isotherms of PE on TIL at different temperatures

Freundlich and Langmuir models were used to fit the isothermal adsorption experimental data. The equations of Freundlich model (Eq. 5) and Langmuir model (Eq. 6) are as follows:

$$q_e = k_f C_e^{1/n} \qquad \text{(Eq. 5)}$$

$$q_e = \frac{q_m b C_e}{1 + b C_e} \qquad \text{(Eq. 6)}$$

Where q_e is the equilibrium adsorption amount (mg/g), C_e is the equilibrium concentration (mg/L), k_f is the Freundlich constant (L/g), n is the dimensionless constant, q_m is the fitted maximum adsorption amount (mg/g), and b is the Langmuir adsorption constant (L/g).

Table 2. Parameters for fitting the adsorption isotherm of PE to TIL

Temperature	Freundlich Model			Langmuir Model		
	n	k_f	R^2	q_m	b	R^2
25℃	0.9664	0.1027	0.9935	18.3486	0.005717	0.9887
15℃	1.1152	0.0874	0.9013	3.0788	0.02808	0.8839

Both the Freundlich and Langmuir models can be used to fit the adsorption isotherms as seen in Figure 4. The correlation fitting parameters are shown in Table 2. The Langmuir model assumes that the adsorbent surface is homogeneous and that all adsorption sites have the same affinity for the adsorbate, whereas the Freundlich model can describe monolayer or multilayer adsorption on a non-homogeneous surface where the adsorption sites on the adsorbent surface can have different affinities for the adsorbate [14]. By comparing the values of the correlation coefficient (R^2) in Table 2, it can be found that the Freundlich model can better model the adsorption isotherms, which indicates that the adsorption of PE microplastics on TIL is non-homogeneous adsorption and the adsorption sites on the surface of PE microplastic particles are not uniformly distributed. In addition, the parameter n in the Freundlich model also responds to the ease with which the adsorption reaction proceeds, with $n > 1$ making the adsorption reaction easy [14]. The fitted results with n values greater than or close to 1 indicate that the adsorption of PE on TIL proceeds more easily. k^f values of 0.0874 and 0.1027 at 15°C and 25°C, respectively, also indicate that the higher the temperature, the stronger the adsorption capacity of PE

on TIL. Studies by Jingwen Pang [9], Kaina Zhang[10], and Shouyi Chen[13] also showed that the Freundlich model is more suitable than the Langmuir model for fitting the adsorption of antibiotics such as Tylosin and oxytetracycline by microplastics such as polypropylene (PP), polystyrene (PS), and polyvinyl chloride (PVC), which may be one of the commonalities of the antibiotic adsorption characteristics of different microplastics.

2.5 Effect of PE particle size on adsorption of TIL

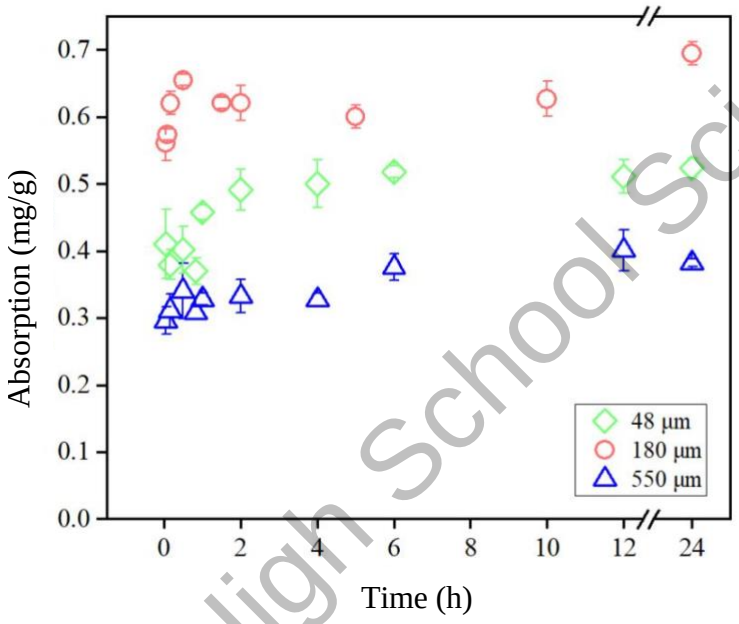

Figure 5. Effect of different particle sizes on TIL adsorption by PE

The effect of particle size of microplastic particles on TIL adsorption by PE is shown in Figure 5. The trend of TIL adsorption by PE with three particle sizes of 550 μm, 180 μm and 48 μm is approximately the same, but from the point of view of equilibrium adsorption, the largest adsorption is by PE with a particle size of 180 μm, followed by PE with a particle size of 48 μm, and the smallest is by PE with a particle size of 550 μm. In general, the smaller the particle size, the greater the number of particles at a certain mass, and therefore the greater the sum of the surface areas of the particles, i.e., the smaller the particle size, the greater the specific surface area. The larger the specific surface area of the adsorbent, the greater the number of adsorption sites and the greater the adsorption capacity. Therefore, the equilibrium adsorption of TIL was higher for PE particles with

small particle size (48 μm and 180 μm) than for PE particles with significantly larger particle size (550 μm). However, comparing the adsorption results of 48 μm PE particles and 180 μm PE particles on TIL does not follow this pattern. This may be due to the fact that the smaller the particle size and the larger the specific surface area, the more likely the particles are to undergo aggregation. The 48 μm PE particles suspended in the aqueous TIL solution may have aggregated into larger secondary particles, reducing the number of effective adsorption sites and making the adsorption amount lower than expected.

2.6 Effect of salinity on TIL adsorption by PE

In natural water environments such as rivers, estuaries, and oceans, the concentration of salinity plays an important role. The salinity of water bodies in the estuary of the Yangtze River in China generally ranges from 2.71% to 2.927% [15], and the average salinity of the East China Sea ranges from 3.1% to 3.4% [16]. To simulate saline natural water bodies, salinity gradients from 0.5% to 3.5% were formed by adding NaCl to the PE-TIL solution system.

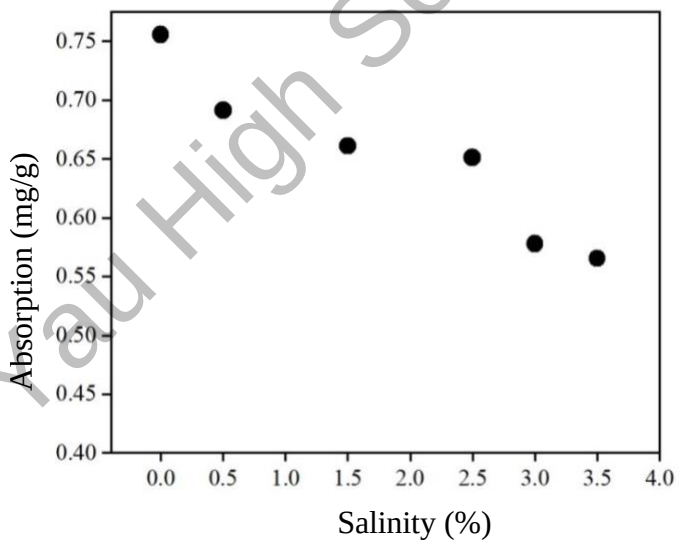

Figure 6. Effect of salinity on TIL adsorption by PE

As shown in Figure 6, the adsorption of PE on TIL was 0.76 mg/g when the solution did not contain NaCl. when the salinity was 3.5%, the adsorption decreased to 0.56 mg/g. As the salinity increased, the adsorption decreased gradually. This may be due to the fact that the increase in Na^+ content neutralizes part of the surface charge [3], thus weakening the

electrostatic interactions between PE and TIL. Also, the increase in salinity increases the viscosity of the solution, which inhibits the mass transfer process from the liquid phase to the solid phase [17]. The results of salinity experiments indicate that PE adsorbs more strongly to TIL in a freshwater environment, and thus the presence of PE is more likely to alter TIL migration.

3 Conclusion and Outlook

3.1 Key Findings

1) The adsorption capacity of polyethylene (PE) microplastics for Tilmicosin (TIL) is greater than that of antibiotics such as Pefloxacin, Difloxacin, Ciprofloxacin, Enrofloxacin and Lincomycin.
2) The adsorption kinetics of PE on TIL was in accordance with the quasi-secondary kinetic model, with the adsorption amount increasing rapidly within the first 30 min, followed by a slow rise to equilibrium. the isotherms of PE on TIL were suitable for fitting with the Freundlich model, indicating that the adsorption sites on the PE surface were heterogeneous and the adsorption on TIL was a non-homogeneous process.
3) The smaller the particle size of PE, the larger the specific surface area, and the greater the adsorption of TIL. However, particles with small particle size may also be aggregated, reducing the effective adsorption sites, which is not conducive to the adsorption of TIL.
4) Increasing temperature favors the adsorption of PE on TIL, while increasing solution salinity inhibits adsorption.

3.2 Outlook

1) Subsequent studies can characterize the microscopic surface structure, surface functional groups, and crystal structure of microplastics before and after the adsorption reaction using large analytical instruments such as scanning electron microscopy (SEM), infrared spectroscopy (FTIR), and X-ray diffraction (XRD) to further clarify the microscopic mechanism of antibiotic adsorption by PE.

2) The effect of pH on TIL adsorption by PE was also explored in this study, but the results of multiple parallel experiments could not be reproduced, so they are not shown in this paper and are subject to further study.
3) The microstructure and properties of different kinds of microplastics, or the same kind of microplastics made newly and aged, differ, so the adsorption properties of microplastics on TIL and other antibiotics can be studied in more depth.

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