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Short Communication

Sorption behavior of real microplastics (MPs): Insights for organic micropollutants adsorption on a large set of well-characterized MPs

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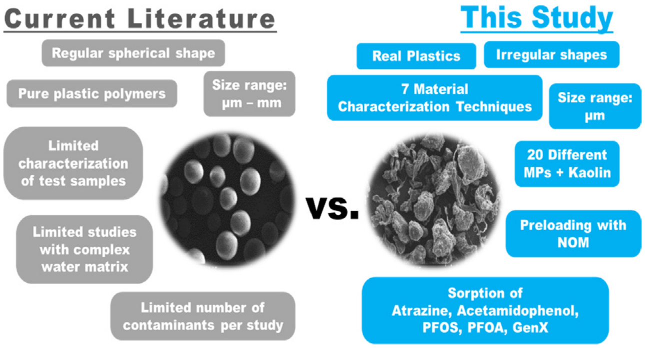
HIGHLIGHTS

- Real MPs have heterogeneous compositions and contain additives and fillers.
- MPs exhibited fast sorption kinetics (<24 h).
- NOM preloading on MPs increased their adsorption affinity towards micropollutants.
- Real MPs show different sorption behavior from pure polymers.
- Results from studies on pure polymers shouldn't be generalized for all MPs.

GRAPHICAL ABSTRACT



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ABSTRACT

Microplastics (MPs) have been recognized as transport vectors for micropollutants in the natural water environment and the food web; therefore, the sorption behavior of contaminant on MPs has recently gained an increased attention. However, a consensus has not yet been reached and information about the adsorption of water contaminants on real MPs remains elusive. Herein, we raise the question of "*Should we continue using pure polymers as surrogates for real MPs*?" This first systematic study compared the adsorption of multiple micropollutants (i.e. a pesticide, a pharmaceutical, and perfluoroalkyl substances (PFAS)) on a large set of MPs (i.e. 20 well-characterized MPs) and kaolin. Material characterizations results showed various physicochemical and compositional differences between real and pure MPs. Pure polymers had lower normalized uptake values than real MPs in most cases. This was attributed to the surface roughness and/or the presence of fillers (e.g. talc and glass fiber) in real samples. Further, preloaded MPs with natural organic matter (NOM) showed an increased uptake of micropollutants due to forming a complex with NOM and/or co-sorption. These findings indicate that employing real MPs in research studies is critical for obtaining environmentally meaningful results, and the evaluation of MPs sorption behavior without NOM preloading can result in a significant underestimation for their actual values. We also provided an outlook the key areas for further investigations.

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1. Introduction

Approximately 300 million tons of microplastics (MPs) are released annually into the environment that can be generally categorized, depending on their sources, into primary or secondary MPs (Hartmann et al., 2019). Primary MPs include microbeads, pellets, and other forms that are produced in small sizes (Pico et al., 2019), and they are widely used in personal care products (e.g. toothpastes, facial cleaners, shampoos), industrial cleaning products (e.g. rust scrubbers), and the production of plastic products (i.e. pellets) (Auta et al., 2017). Secondary MPs, on the other hand, are the degradation byproducts from large plastic pieces (e.g. plastic fibers) (Galafassi et al., 2019). The degradation occur in environment as a result of one or a combination of factors such as, biodegradation by living organisms, photodegradation by UV light, thermooxidative degradation under low temperature, thermal degradation at high temperature, and hydrolysis in water (Andrady, 2011; Song et al., 2017). There is a growing concern about toxicological risks associated MPs after their direct (i.e. misidentification or indiscriminate consumption of MPs for feeding) or indirect (i.e. trophic transfer along the food web) uptake by living organisms (Alimba and Faggio, 2019). One major concern is the adsorbed toxic chemicals on MP surfaces (Verla et al., 2019), which is a function of physicochemical properties of MPs and contaminants, and environmental conditions. Therefore, the interactions between MPs and different organic/inorganic contaminants have attracted an increasing attention, as summarized in several recent reviews (Alimi et al., 2018; Rodrigues et al., 2018; Wang et al., 2018; Yu et al., 2019; Zhang et al., 2019).

Despite the increased number of studies, the adsorption of contaminants on MPs in water remains elusive due to three critical issues. First, a great majority of the studies have used virgin/pure polymers from commercial vendors and tended to generalize their observations from these adsorption tests (Alimi et al., 2018). The real MPs, however, have other constituents, in addition to the plastic polymer (i.e., plasticizers, stabilizers, pigments, fillers, extenders, solvents, and other additives) with a very high potential to influence their adsorption behavior. Second, the lack of rigorous characterization of MPs and the limited number of MPs samples in previous studies lead to superficial discussion with some inconsistent conclusions about the sorption affinity of different MPs towards pollutants (Alimi et al., 2018; Hartmann et al., 2019). For instance, fillers from different sources (e.g. calcium carbonate, talc, mica, and carbon black) are being used for different practical and economic purposes; such as, reducing the products' cost and altering the plastic's mechanical, thermal and/or chemical properties. Moreover, commercial virgin MPs come in the form of smooth micron-sized spheres, which implies that – although the manufacturers do not reveal such proprietary information – they might have been synthesized via emulsion aided methods (Ganesan et al., 2014). In such a case, exogenous surfactants are routinely added to the reaction during synthesis, which could consequently alter the surface characteristics (e.g. hydrophilicity) (Omi et al., 1995). These additives would indeed change the adsorption behavior of real MPs when compared to their virgin polymeric surrogates, and yet previous studies have overlooked these important aspects (Hüffer et al., 2018). Finally, MPs in natural waters are continuously surrounded by and in contact with background organic matter (Alimi et al., 2018; Rodrigues et al., 2018). As a result, understanding the sorption behavior of real MPs in the presence of natural organic matter rather than in distilled and deionized water is more representative.

Herein, we considered such limitations in the literature and studied systematically the adsorption behavior of a pesticide (i.e. atrazine), a pharmaceutical (i.e. acetamidophenol), and three perfluoroalkyl substances (i.e., PFOS, PFOA, GenX) on fourteen well-characterized MPs (i.e. crushed MPs from real products) and compare with six virgin plastic polymers (i.e. PE, PP, PA66, PMMA, and PLA) as well as kaolin (i.e. model suspended sediment in water environment). We also investigated the effect of natural organic matter (NOM) preloading on MPs' surfaces, which is a common phenomenon in the natural environment, on the adsorption behavior of MPs. The target pollutants were mainly selected due to their environmental significance, as the primary objective of the study was to examine and compare the adsorption behavior of crushed plastics products to mimic the 'real MPs' with those of virgin plastic polymers and the factors that influence the adsorption process under environmentally relevant conditions.

2. Materials and methods

The target compounds in this study were atrazine (Sigma Chemical Co., USA), 4-Acetamidophenol (Sigma Chemical Co., USA), perfluorooctanoic acid (PFOA; Acros Organics Corporation, USA), perfluorooctanesulphonic acid, potassium salt (PFOS; Matrix Scientific, USA), and hexafluoropropylene oxide dimer acid (GenX; Wellington Laboratories Inc., Canada). Selected physicochemical characteristics of the five target compounds are provided in Table S1.

2.1. MPs collection

Samples from different polymer types (i.e. polyethylene [PE], polypropylene [PP], polyethylene terephthalate [PET], polystyrene [PS], acrylonitrile butadiene styrene [ABS], poly(methyl methacrylate), polylactic acid [PMMA], nylon) were collected either as commercial products or crushed recycled plastics from a local plastic recycling company in SC, USA. All samples were washed with tap water followed by distilled de-Ionized water and dried at room temperature until further use. Pure PE-1 (75–90 μm) and PMMA-2 (27–45 μm) were purchased from Cospheric Co., CA, USA. Other pure samples were purchased online from Edwards Industrial Sales Inc., MI, USA.

2.2. MPs crushing

All samples were cut to pieces with maximum size of 1 cm. Then, the cut pieces were frozen in liquid nitrogen and crushed to <500 μm diameter using a Fritsch Pulverisette-14 (Fritsch GmbH, Germany) (Fig. 1). The crushed samples were dried at room temperature and stored until further use. Pure PE and PMMA were washed, dried and used without crushing.

The molecular structures and the photographs of MPs tested in this study are provided in Figs. S1 and S2, respectively. The details of material characterizations using Brunauer-Emmett-Teller (BET) surface area analyzer, elemental analyzer, scanning electron microscope (SEM), attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy, differential scanning calorimeter (DSC), X-ray Diffraction (XRD) spectroscopy, and thermogravimetric analysis (TGA) are provided in Section S2. The experimental procedures for the sorption tests and analytical procedures are described in Sections S3 and S4.

3. Results and discussion

3.1. MPs have heterogeneous and different compositions

The sources of all MPs in this study and the complete characterization results for the specific surface area (SSA), elemental analysis, glass transition temperature, melting temperature, and stepwise TGA analysis are listed in Tables S2 and S3 in the Supporting information. Generally, MPs were nonporous and had low SSA in the range of <1–3.5 m^2/g, while kaolin showed to have higher SSA of 10.5 m^2/g (Table S2). Four samples that were made of polyethylene (PE) had distinct morphologies and compositions. The plastic shopping bag (HDPE-4) was in the state of a thin film with ~27 wt% $CaCO_3$ filler, as indicated by the stepwise TGA degradation profile (Fig. S3). However, commercial pure PE polymer (PE-1) was in the state of 75 μm

Fig. 1. Plastic samples were crushed using Fritsch Pulverisette-14.

spherical particles with smooth surface and has been widely used as a model MP in previous studies (Johansen et al., 2018; Rummel et al., 2016; Schirinzi et al., 2016; Velzeboer et al., 2014). Similarly, out of four polypropylene (PP) samples (i.e. PP 1–3 and MIX-3), two of them were found to be composites with talc (PP-3: ~4%) and glass fiber (MIX-3: ~50%) that were apparently added for reinforcement purposes (Sathishkumar et al., 2014). The glass fiber could be clearly seen in the SEM images (Fig. S4), while the talc was identified by FITR spectra at wave number 1017 cm^{-1} (Fig. S5). It should be highlighted that the calculated values of the degree of crystallinity are approximate, since the exact compositions of some MPs are uncertain. For example, although the homopolymer PP is the most widely used, PE copolymers are being used sometimes in the market (Maddah, 2016). The PE or other polymeric segments are incorporated into the PP chain to further ameliorate the properties, as a result, the melting temperature (T_m) may be reduced. This can explain the lower T_m of PP-3 and MIX-3 (i.e. 150 and 155 °C) than typical PP homopolymer, such as PP-1 and PP-2 (i.e. 162 and 165 °C) (Table S3 and Fig. S6) (Maddah, 2016). As shown in Fig. S4, PP-1 was bead-shaped with larger dimension (400 μm) and less regular geometry than the PE-1 microspheres. This agrees with features of some particles manufactured by the phase separation method, which requires less stabilizing reagent (Sahil et al., 2011).

Three samples can be categorized as polyamide (PA), namely Nylon-1, PA66-4, and MIX-1. Among them, Nylon-1 (i.e. nylon rope) was found to be a blend of PA and PP, which demonstrated each individual melting/crystallization peaks in the DSC curve (Fig. S6) as well as in XRD spectra (Fig. S7). Nylon-1 is composed of PA66, while the MIX-1 is composed of PA6. This could be reflected on the difference in T_m of Nylon-1 (i.e. 240–265 °C for PA66) from the PA6 at 210–220 °C. The inorganic fillers were found to account for ~35 wt% of PA66–4 and ~30 wt% of MIX-1 (Fig. S3), while the latter was glass fiber as can be observed in the SEM image (Fig. S4). While we did not characterize the hydrophobicity of the polymers, the measured characteristics and the general properties of the polymer can reveal indirect information. For instance, PP, PE, PS are highly hydrophobic based on their molecular structure, while PA contains polar moieties in its chain that adsorb moisture as revealed by the weight reduction and enthalpy of vaporization during the TGA and DSC measurements, respectively (Figs. S3 and S6). These characterization results document various physicochemical and compositional differences between real and virgin MPs. Therefore, employing real MPs in adsorption studies and their meticulous material characterizations by multiple techniques are imperative when investigating the sorption interactions between plastic debris and other contaminants.

3.2. Most MPs adsorbed PFOS and PFOA > acetamidophenol, GenX and atrazine

The adopted pH value in this study was 6.5 ± 0.2, which influenced the form of target compounds. Atrazine and acetamidophenole were present in nonanionic form (Mohd et al., 2015; Sawhney and Singh, 1997), whereas PFOS, PFOA, GenX were in anionic form (Burns et al., 2008). Kinetic experiments on four MPs (i.e. PE-1, PP-1, Nylon-1, PLA-1) revealed that maximum uptake values for the target five contaminants could be, in general, reached within one day (Fig. S8). This agrees with the reported ranges in previous studies on MPs and other biodegradable plastics.

To the best of our knowledge, this is the first report of GenX and acetamidophenol (i.e. Paracetamol; a common pain reliever and a fever reducer) uptake by real MPs, and the first systematic study that compare the adsorption of multiple micropollutants on a large set of MPs. Despite the variations, Fig. S9 illustrates a notable difference in the uptake of the target five compounds on MPs. The N-heteroaromatic ring in atrazine can function as both π-donor and electron-acceptor with the negatively charged functional groups of the sorbent (Keiluweit and Kleber, 2009). Since both atrazine and acetamidophenol were mainly present in neutral and weakly polar form, their uptakes were mainly driven by hydrophobicity and π-π interactions. Previous studies on the sorption of atrazine on pure PE and PP at different pH values (i.e. pH 3–10) have also reported on a lower uptake at neutral pH with no effect of the ionic strength of the solution (Hüffer et al., 2019; Seidensticker et al., 2018). The uptake values of acetamidophenol and atrazine were comparable on PE, PP, PS, and ABS; however, the uptake of acetamidophenol was $\times 10^1 - \times 10^2$ times higher than atrazine by PA66 (i.e. nylon), PLA (i.e. biodegradable MPs), PET (i.e. plastic bottle), PMMA, and tire particles (Fig. S9). This was attributed to the increased polarity of MPs, as illustrated in their molecular structure (Fig. S1) (Brennecke et al., 2016).

The consistent trend in PFAS uptake (i.e. PFOS > PFOA > GenX) further indicates that hydrophobic interactions were the dominant removal mechanism (Llorca et al., 2018; Wang et al., 2015). The highest PFAS removal was by PA66 polymer (i.e. Nylon rope sample) and PLA polymer (i.e. biodegradable cup). Although no previous study has examined PFAS sorption on these polymers within the context of MPs, a recent study have evaluated PFAS recoveries from different filter types and found the nylon filter showed the lowest recovery (i.e. highest PFAS adsorption) (Lath et al., 2019). One possible reason might be the presence of polarized N—H bonds in the polymer chain (Fig. S1), which can facilitate PFAS uptake by hydrogen bonding (Ateia et al., 2019). It should be also

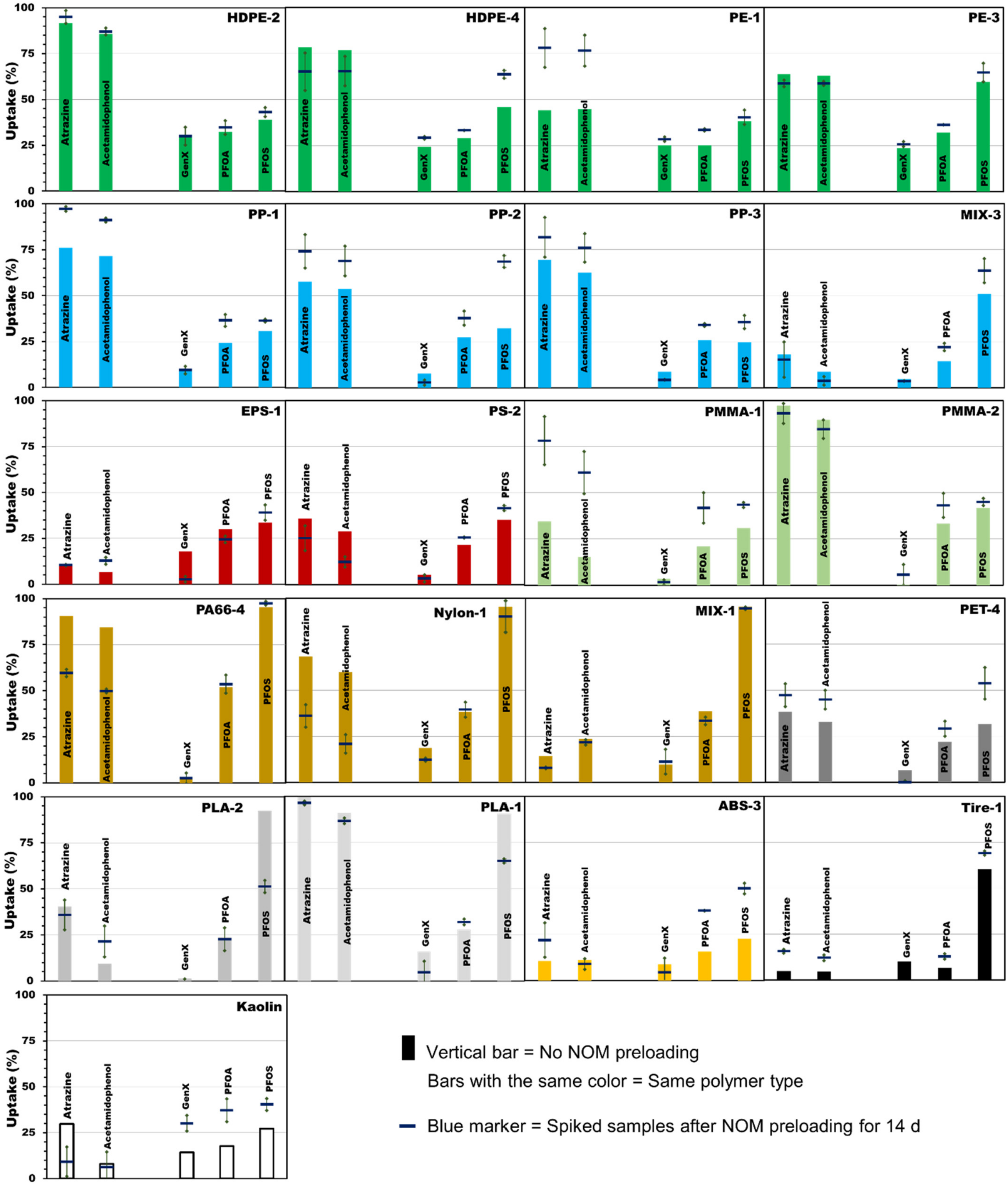

Fig. 2. Adsorption of the target five contaminants on all MPs and kaolin in distilled deionized water (vertical colored bars) and with NOM preloading (blue marker). All samples were preloaded with NOM for 14 d in lake water with 2 mg-C/L. The initial concentration of each compound was 50 μg/L at a dose of 30 mg of MPs in 20 mL, reaction time of 14 d and pH of 6.5 ± 0.2. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

noted that although MPs had much lower SSA than kaolin, the uptake values of all five compounds on MPs and kaolin were comparable (Fig. S9). These observations agree with a previous study on the sorption of atrazine and other micropollutants on soil and virgin PE (Xing et al., 1996). These observations suggest that although MPs have lower surface area than sediments/soils, MPs can play an important role in the sorption and transport of pollutants in natural environment (e.g. fresh water (Zhang et al., 2019) and soils (Hüffer et al., 2019)). Isotherm studies should give further insights on the sorption capacity of MPs; however, this is beyond the focus of this communication.

3.3. NOM preloading increased the adsorption on most MPs

Preloading is defined as the prior exposure of the adsorbent to NOM, which is assumed to occupy high energy sorption sites (Pelekani and Snoeyink, 1999). Since NOM is omnipresent in natural waters and wastewaters with concentrations much higher than organic micropollutants (Ateia et al., 2017), preloading MPs with NOM can give more realistic insights on the sorption behavior of MPs in natural water systems. The preloading in this study was conducted in surface water from Lake Hartwell, SC, USA with DOC concentration of 2.5 mg/L and specific ultraviolet absorbance at 254 nm ($SUVA_{254}$) of

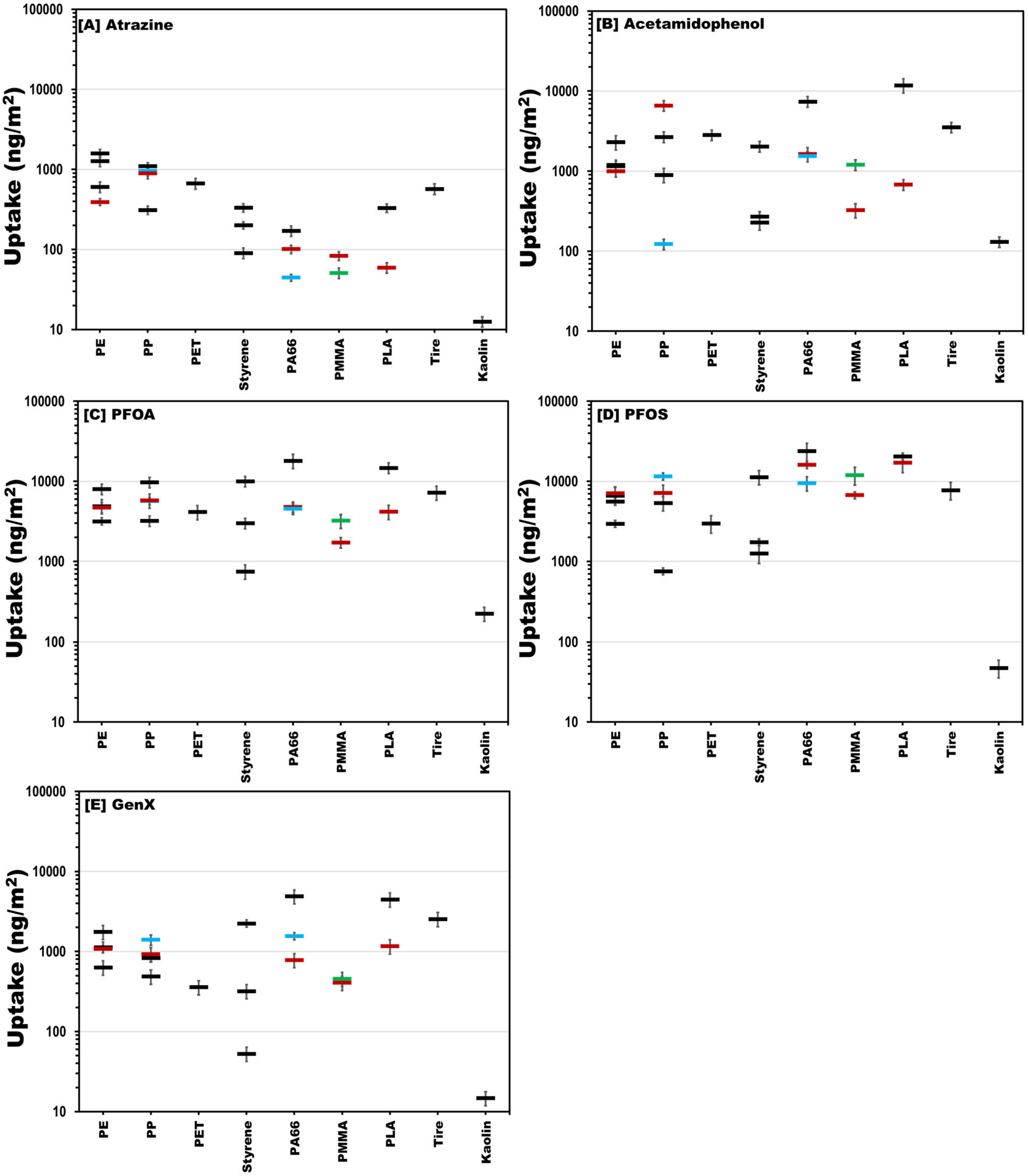

Fig. 3. Adsorption of the target five contaminant on all MPs and kaolin. The initial concentration of each compound was 5 μg/L at a dose of 30 mg of MPs in 20 mL and pH of 6.5 ± 0.2. Red marker indicates pure polymers. Blue marker indicates samples with mixed polymer types. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

2.0 ± 0.5 L/mg.m (Ateia et al., 2018). We observed an overall increase in the uptake of the target contaminants on the preloaded MPs with a lake water NOM (Fig. 2). Previous studies have shown that the increase in NOM content on soil, nanoparticles, and PE polymers would increase atrazine adsorption on soil by forming a complex with NOM, cosorption, and cumulative sorption (Ling et al., 2006; Lu et al., 2009; Xing et al., 1996). Similar factors are likely affecting the sorption of atrazine and acetamidophenol on MPs in this study. Long-chain PFAS adsorption on soil also showed an increasing trend with NOM content; however, no significant increase in the adsorption of short-chain PFAS under the same conditions (Zhang et al., 2013). A similar trend could be observed in this study, which implies that co-sorption probably occurred between NOM and long-chain PFAS (i.e. more hydrophobic). These observations indicate that evaluation of MPs sorption behavior without NOM preloading might results in a significant underestimation for their actual values. Future studies should also examine the surface characteristics of MPs with and without NOM preloading.

3.4. The way forward

The main idea while designing this study was to run comparative tests to know if researchers should continue using pure polymers as surrogates for real MPs. Most studies in the literature have tested very limited number MPs (e.g. 2–5 MPs) and run adsorption experiments at different experimental conditions (i.e. target contaminants, initial concentrations, background water constituents) (Alimi et al., 2018; Rodrigues et al., 2018; Wang et al., 2018; Yu et al., 2019). Thus, using these scattered data points to rank sorption capacities of MPs is not realistic and can be somewhat misleading (Alimi et al., 2018; Wang et al., 2018). Herein, although all samples were evaluated under the same conditions, the results indicated no general trend or consensus for uptake of pollutants by real MPs. As shown in Fig. 3, large variations in the normalized uptake of different contaminants (i.e. $x10^1$ – $x10^2$) could be observed for samples with the same polymer type. Yet, pure polymers were found to have lower normalized uptake values than real MPs in most cases. This might be attributed to the surface roughness (Müller et al., 2018; Zhang et al., 2018) and/or the presence of fillers (e.g. talc and glass fiber) in real samples (Hartmann et al., 2019), as discussed above.

The physicochemical properties and characteristics of MPs, micropollutants, and background solution play an important role on the uptake, fate, and transport of micropollutants in natural water systems. Although sorption experiments on pure polymers can give some fundamental information on the kinds on interactions, they do not represent more complex set of interactions of real MPs in aquatic systems. The advancements in the detection and isolation of MPs from real water samples should be coupled with detailed material characterization to gain more insights on their behavior and impact on water quality. One key area requiring further investigation is to compare the MPs material characteristics (i.e. surface modification and functionalization) before and after pollutants' uptake. Controlled studies on the effect of different additives and fillers on the sorption behavior of real MPs will be important to understand in this complex system. It is also important to investigate the differences between aged pure polymers and aged real MPs. For instance, aging of pure polymers under UV irradiation (or solar light) doesn't give a realistic reflection of aging of real plastics. One of the reasons can be the use of UV stabilizers and other antioxidants in real plastics would result in aged MPs with different characteristics from pure polymers. Besides, more studies are needed to evaluate the effect of the physicochemical properties of micropollutants (e.g., molecular size, geometrical configuration, hydrophobicity, aromaticity, polarity, and substituent groups) on the uptake behavior of real MPs under different pH, ionic strength, and temperature. Since both MPs and micropollutants are abundant in wastewaters, future studies should also explore the effect of effluent organic matter (EfOM) on the sorption behavior of MPs. Such systematic investigations will give a realistic understanding on the impact and role of different uptake mechanisms; such as, hydrophobic, electrostatic, π-π, van der Waals, hydrogen bonding, and Lewis acid-base interactions. In addition, evaluation of real MPs in long-term experiments is inevitable, for which this data is currently not available in the literature.

CRediT authorship contribution statement

Mohamed Ateia: Conceptualization, Methodology, Investigation, Formal analysis, Visualization, Writing - original draft, Writing - review & editing.**Ting Zheng:**Methodology, Investigation, Formal analysis, Visualization, Writing - review & editing.**Stefania Calace:**Investigation, Visualization, Writing - review & editing.**Nishanth Tharayil:**Investigation, Resources, Writing - review & editing.**Srikanth Pilla:**Supervision, Funding acquisition, Writing - review & editing.**Tanju Karanfil:**Supervision, Funding acquisition, Writing - review & editing.

Declaration of competing interest

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

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Appendix A. Supplementary data

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