

(14) Removal of Micro-Organic Pollutants from the Effluent of Municipal Wastewater Treatment Plant by Sorption onto Synthetic Polymeric Adsorbents

下水処理場流出水中に含まれる微量有機汚染物質の合成ろ材による吸着除去

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Abstract, The sorption capacity of a synthetic polymer, polypropylene (PPL), and polypropylene coated with *n*-hexane (PPL-HEX) for hydrophobic organic compounds (HOCs) was evaluated from equilibrium batch sorption experiments. Three polycyclic aromatic hydrocarbons (PAHs), namely, phenanthrene, fluorene and naphthalene were used as HOCs. PAHs were quantified by the fluorescence quenching method. The effects of pH, ionic strength, and dissolved organic matter (DOM) on the sorption were investigated. Considering sorption capacity (as measured by the conditional sorption coefficient, K_s^c) and sorption rate, PPL-HEX is the better adsorbent for PAHs than PPL. Ionic strength and pH did not have any effect on K_s^c . Neither PPL nor PPL-HEX sorb DOM, and the presence of DOM in the aqueous phase did not show any significant effect on the K_s^c for sorption of PAHs onto PPL-HEX. The main advantage of using PPL or PPL-HEX as adsorbents is that since they do not sorb DOM, target HOCs do not compete with DOM for sorption onto PPL and PPL-HEX, as opposed to sorption onto activated carbon.

Key words, sorption, micro-organic pollutant, *n*-hexane, polycyclic aromatic hydrocarbons (PAH), polypropylene

1. Introduction

In many arid and semi-arid countries, reuse of municipal wastewater treatment plant effluents (MWTPE) for agricultural purposes is a common practice (WHO, 1993). The safety of such recycled water is an important consideration that has to be taken into account. One of the main concerns is that over the past few years there has been a tremendous increase in number, variety and quantity of complex organic chemicals used for industrial, agricultural and domestic purposes. These chemicals eventually find their way into municipal wastewater treatment plants (Clark *et al.*, 1991; McCarty and Reinhard, 1980; Paxeus *et al.*, 1991; Pinholster, 1995). Municipal wastewater is, therefore, a mixture of many complex organic materials only some of which have been identified. Even though the fate of these compounds in the environment is not fully understood at the moment, some of them are generally believed to have toxic, carcinogenic, mutagenic or teratogenic properties that may jeopardize public health.

The majority of the organic compounds of concern are hydrophobic with low aqueous solubilities and therefore strongly sorb onto soil. Once sorbed onto soil they are highly resistant to desorption back into the aqueous phase. As a result, contaminated soil continues to release the sorbed compounds into water at a slow rate and thereby provides a continuing source of surface and ground water contamination, in addition to possible contamination through the food chain. There is, therefore, need for more strict control of the quality of wastewater for irrigation. This calls for new economically feasible wastewater treatment technologies for the removal of these toxic organics if MWTPE are to be acceptable for agricultural reuse.

Adsorption by activated carbon (AC) is an established technology for removal of organic contaminants. AC treatment, however, has several limitations such as high cost of carbon regeneration and interference of DOM on the adsorption process due to competition between DOM and target organic compounds for adsorption sites on AC. In recent years, synthetic polymeric adsorbents have been considered as alternatives to AC (Van Vliet and Weber, 1981; Weber and Van Vliet, 1981a,b). One significant

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advantage of the synthetic polymeric adsorbents is that they have an insignificant sorption capacity for DOM.


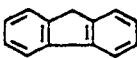
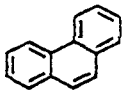
The objective of this research is to evaluate the feasibility of using a synthetic foam polymer, polypropylene (PPL), and polypropylene coated with *n*-hexane (PPL-HEX) as adsorbents for the removal of HOCs in MWTPE with the perspective of reusing the effluent for agricultural irrigation. PPL used in this research is manufactured by Daiwa Kogyo Co., Ltd. (Tokyo) from recycled PET bottles. PPL is highly hydrophobic, has high resistance to abrasion, and is highly porous (surface area 800 m²/m³, specific gravity 0.75). It is available in different sizes, ranging from powder form to 30 mm diameter. It was hypothesized that PPL being strongly hydrophobic, has a high affinity for HOCs. Coating PPL with *n*-hexane further enhances the hydrophobicity of PPL. To achieve the objective of this research, basic equilibrium batch sorption experiments were performed. Sorption capacity is expressed in terms of the conditional sorption coefficient, K_s^c . The effects of DOM, pH, and ionic strength on the sorption were also investigated.

2. Materials and Methods

2.1 Selection of Representative HOCs

Three polycyclic aromatic hydrocarbons (PAHs), namely, naphthalene (Naph), fluorene (Fluo) and phenanthrene (Phen), were selected as representative HOCs. The physical and chemical characteristics of these three PAHs are given in Table 1.

Table 1. Physical and Chemical Properties of Naphthalene, Fluorene and Phenanthrene.

	Naphthalene	Fluorene	Phenanthrene
Structure			
Molecular Weight	128.2	166.2	178.2
Log K_{ow}	3.36	4.18	4.57
Aqueous Solubility (mg/L)	31.5	1.82	1.12

PAHs were selected because they represent categories of environmentally significant pollutants and several PAHs have been reported to be present in MWTPE. A commercial humic acid from Aldrich Chemical Co. was used as a surrogate of organic matter. All reagents used were of analytical grade and of the highest purity available (>97% for PAHs) and were used as purchased without further purification.

2.2 Experimental Aqueous Phase

The experimental aqueous phase consisted of distilled de-ionized water with a NaHCO₃/Na₂CO₃ buffer. The pH was adjusted with HCl or NaOH, while the ionic strength was adjusted with NaCl. A 0.02 wt% of sodium azide was added to minimize the biological transformation of the PAHs (McCarthy and Jimenez, 1985). Aqueous PAH solutions were prepared by transferring PAH solutions in methylene chloride to a glass container followed by evaporation of the methylene chloride. The experimental aqueous phase was then introduced and mixed by stirring until steady fluorescence signals were achieved.

Sorption isotherms are linear if the equilibrium aqueous phase organic compound concentration is below 10⁻⁵ M or below one half of the aqueous phase solubility, whichever is less (Karickhoff *et al.*, 1979; McCarty *et al.*, 1981). To keep the sorption isotherms within the linear range, the initial nominal aqueous PAH concentrations were set at 0.9, 0.8 and 0.5 mg/L for naphthalene, fluorene and phenanthrene, respectively.

2.3 Batch Sorption Experiment

Sorption was done in 50 mL glass centrifuge tubes at 25°C ($\pm 1^\circ\text{C}$). Prewashed polypropylene (size 0.30 to 0.42 mm) was first soaked in water (or *n*-hexane) for 24 hours. Excess water (or *n*-hexane) was then poured off and the PPL (or PPL-HEX) was transferred to centrifuge tubes containing PAH solutions (for PPL-HEX, the ratio of hexane sorbed to polypropylene ranged from 60 – 100 wt%). All centrifuge tubes were filled to the top to minimize head-space and wrapped with aluminum foil to prevent photodegradation. The tubes were then equilibrated by shaking on a rotary shaker at 20rpm for 21 hours, allowed to settle for 3 hours and then centrifuged at 5,000g for 15 minutes. Analysis of the aqueous phase was done by fluorescence, at excitation/emission wavelengths of 280/330 nm, 271/308 nm and 293/365 nm for naphthalene, fluorene and phenanthrene, respectively.

Sorption of PAHs onto humic acid was investigated by equilibrating PAH solutions with varying concentrations of humic acid at 20rpm for 24 hours using a rotary shaker after which the PAH and humic acid concentrations were analyzed. To investigate the effect of DOM on sorption of PAHs onto PPL and PPL-HEX, varying amounts of PPL or PPL-HEX were added to PAH solutions initially pre-equilibrated for 24 hours with 5 mg-organic carbon/L humic acid as described above. This was followed by equilibration for a further 24 hours then the PAH and humic acid concentrations were analyzed. The absorbance at 254 nm was used to quantify the concentration of humic acid. Free PAH in the aqueous phase was measured by fluorescence, while the high-pressure liquid chromatography (HPLC) method (with fluorescence and absorbance detectors) was used to measure total PAH concentration (free PAH plus PAH bound to humic acid). The HPLC mobile phase (flow rate at 1 mL/min) was set at 80:20 (v:v) acetonitrile in water. All experiments involving humic acid were done at pH 7 and ionic strength 0.06 M.

2.4 Fluorescence Quenching Method

PAHs were analyzed by the fluorescence quenching analytical technique. In this method, the sorbed fraction of PAHs is determined from the fractional decrease in the intensity of fluorescence upon the addition of the adsorbent (Gauthier *et al.*, 1986). The linear equilibrium adsorption model for such a system is given by:

$$q_e = K_p C_e \quad (1)$$

In Equation (1); q_e , K_p , and C_e are the amount of PAH adsorbed per unit weight of adsorbent, the equilibrium partition coefficient, and the aqueous-phase concentration of the PAH at equilibrium, respectively. Taking a mass balance on the PAH gives:

$$C_{\text{tot}} = [\text{PAH-S}] + [\text{PAH}] \quad (2)$$

In Equation (2); C_{tot} , $[\text{PAH-S}]$, and $[\text{PAH}]$ are the respective total, sorbed and aqueous phase concentrations of PAH. Assuming linear sorption, Equations (1) and (2) may be combined and simplified to give an equation in the form of the Stern-Volmer equation:

$$F_0 / F = 1 + K_s^c S_{\text{tot}} \quad (3)$$

In Equation (3); F_0 and F are the fluorescence intensities in the absence and presence of the adsorbent respectively; S_{tot} is the adsorbent concentration (g/L) and K_s^c is the conditional sorption coefficient (L/g). In this research, fluorescence intensities were measured with increasing amounts of S_{tot} . The conditional sorption coefficient (K_s^c) was obtained as a slope of the F_0 / F vs. S_{tot} plot. Corrections for the inner filter effect were done as described by Shimizu (1990).

3. Results and Discussion

3.1 Sorption onto PPL and PPL-HEX

Figure 1 shows the variation of fluorescence intensity with time for sorption onto PPL and PPL-HEX. Fluorescence intensity remained constant after about 12 and 3 hours for sorption onto PPL and PPL-HEX

respectively. Thus, an equilibrium was reached in about four times faster for sorption onto PPL-HEX than for sorption onto PPL. Mass transport steps involved in the sorption onto PPL are bulk transport of PAHs

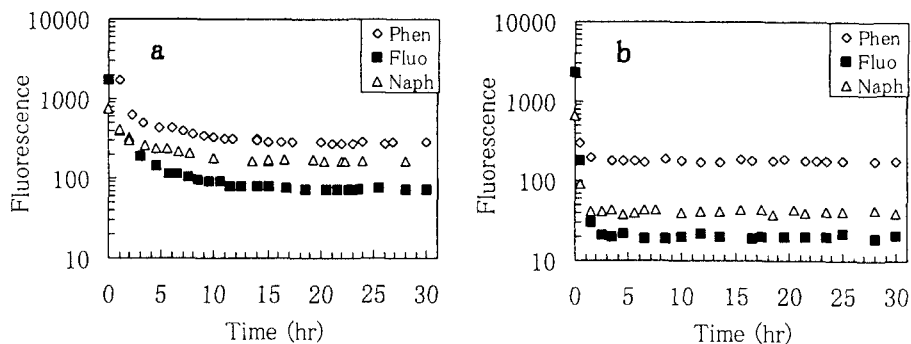


Figure 1. Variation of Fluorescence Intensity with Time for Sorption onto (a) Polypropylene (PPL) and (b) Polypropylene Coated with *n*-Hexane (PPL-HEX).

in solution phase and intraparticle transport within the pore spaces in PPL. Bulk transport is rapid because of mixing and therefore, intraparticle transport, which is the slower step, is the rate determining step and accounts for the slow rate of sorption onto PPL. In the case of PPL-HEX, sorption onto the *n*-hexane layer is rapid because it involves bulk transport of the PAHs from the aqueous phase to the *n*-hexane phase. Also some sorption sites on PPL are possibly blocked by the presence of *n*-hexane on the surface of PPL and are therefore not available for sorption. As a result, there is a relatively faster establishment of equilibrium in the PPL coated with *n*-hexane than in PPL not coated with *n*-hexane, and therefore, the overall rate of sorption onto PPL-HEX is faster than that onto PPL. The 24-hour reaction time adopted in this research was considered adequate to attain equilibrium.

The intercept and slope of Stern-Volmer plots for the sorption of PAHs onto PPL and PPL-HEX under various experimental conditions are shown respectively in Tables 2 and 3. Figures 2 and 3 show typical Stern-Volmer plots. In Figures 2 and 3, PPL (g/L) and PPL-HEX (g/L) are the respective concentrations of dry polypropylene and polypropylene treated with *n*-hexane, which correspond to S_{tot} in Equation (3). All the plots are linear with a correlation greater than 98%. All slopes are significantly different from zero at the 95% confidence level. The intercepts are not significantly different from unity at the 70% confidence level. In this research, it was not possible to achieve the recommended range of 1.25 – 5.00 for F_0/F (Banerjee *et al.*, 1985) for sorption experiments due to high sorbent:solution ratios used (2 – 10 g/L). Within this limitation, the plots are considered to be in conformity with the Stern-Volmer Equation (3) and the slopes correspond to the conditional sorption coefficient, K_s^c .

The effect of pH and ionic strength on sorption onto PPL and PPL-HEX is shown in Figures 4 and 5. The K_s^c does not show any dependence on pH or ionic strength. Since the PAHs are highly hydrophobic and non-ionic, hydrophobic interactions are the dominant mechanisms involved in the sorption.

The average values of the K_s^c over the experimental ranges of pH and ionic strength for sorption onto PPL are 10.31, 5.69 and 0.93 L/g for phenanthrene, fluorene and naphthalene, respectively (Table 2). The corresponding values for sorption onto PPL-HEX are 19.68, 11.82 and 2.09 L/g (Table 3). The K_s^c values for sorption onto PPL-HEX are greater than those for sorption onto PPL by factors of 1.91, 2.08 and 2.25 for phenanthrene, fluorene and naphthalene, respectively. Therefore, in terms of sorption capacity, PPL-HEX is better than PPL as an adsorbent.

3.2 Sorption onto Humic Acid

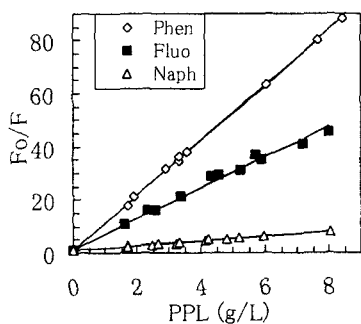
The Stern-Volmer plots for the sorption onto humic acid are shown in Figure 6. The slopes are significantly different from zero at the 95% confidence level and the intercepts are not significantly different from unity at the 95% confidence level. The K_s^c values are 3.16×10^{-2} and 1.02×10^{-2} L/mg-organic carbon for phenanthrene and fluorene, respectively. The K_s^c for sorption onto humic acid were used to check the mass balance of PAHs for experiments involving sorption onto PPL-HEX in the presence of humic acid.

Table 2. Parameters of Stern-Volmer Plots for Sorption onto Polypropylene (PPL).

Ionic Strength		Stern-Volmer Plot		Correlation Coefficient (R)
		Intercept \pm SD	Slope (K_s°) \pm SD	
pH	(M)			
Phenanthrene				
9.70	0.10	1.057 ± 0.032	10.366 ± 0.316	0.999
9.95	0.05	1.086 ± 0.036	10.360 ± 0.345	0.994
9.81	0.01	1.008 ± 0.031	9.576 ± 0.292	0.999
6.18	0.05	1.378 ± 0.043	10.670 ± 0.335	0.997
3.00	0.05	1.103 ± 0.043	10.557 ± 0.409	0.983
K_s° Average =			10.31 ± 0.43	
Fluorene				
9.73	0.10	1.371 ± 0.045	5.786 ± 0.190	0.994
9.53	0.06	0.990 ± 0.030	5.876 ± 0.179	0.999
9.78	0.01	1.148 ± 0.036	5.204 ± 0.162	0.998
6.29	0.06	1.033 ± 0.032	5.113 ± 0.160	0.998
2.82	0.06	1.031 ± 0.033	6.454 ± 0.205	0.997
K_s° Average =			5.69 ± 0.55	
Naphthalene				
9.70	0.10	1.001 ± 0.031	0.901 ± 0.028	0.997
9.71	0.06	1.005 ± 0.030	0.905 ± 0.027	0.999
9.69	0.01	1.006 ± 0.031	0.910 ± 0.028	0.999
6.55	0.06	0.990 ± 0.031	0.971 ± 0.030	0.998
2.90	0.06	0.987 ± 0.030	0.969 ± 0.030	0.998
K_s° Average =			0.93 ± 0.04	
K_s° in L/g		SD = Standard Deviation		

K_s° in L/g

SD = Standard Deviation



Phen $F_o/F = 1.057 + 10.366 (PPL)$, $R = 0.999$
 Fluo $F_o/F = 1.371 + 5.786 (PPL)$, $R = 0.994$
 Naph $F_o/F = 1.001 + 0.901 (PPL)$, $R = 0.997$

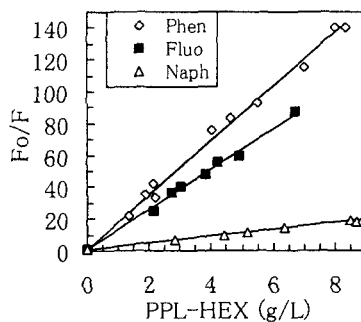
Figure 2. Stern-Volmer Plots for Sorption of PAHs onto Polypropylene (pH_{av} 9.71, Ionic Strength 0.10 M).

Table 3. Parameters of Stern-Volmer Plots for Sorption onto Polypropylene Coated with *n*-Hexane (PPL-HEX).

Ionic Strength		Stern-Volmer Plot		Correlation Coefficient (R)
		Intercept \pm SD	Slope (K_s°) \pm SD	
pH	(M)			
Phenanthrene				
9.66	0.10	1.313 \pm 0.041	16.996 \pm 0.535	0.997
9.90	0.05	1.103 \pm 0.036	20.373 \pm 0.657	0.996
9.83	0.01	1.461 \pm 0.049	14.796 \pm 0.497	0.993
6.30	0.05	0.718 \pm 0.024	21.957 \pm 0.719	0.995
2.91	0.05	0.884 \pm 0.027	24.299 \pm 0.740	0.999
K_s° Average =			19.68 \pm 3.81	
Fluorene				
9.74	0.10	0.903 \pm 0.028	12.577 \pm 0.392	0.998
9.78	0.06	0.993 \pm 0.031	11.059 \pm 0.341	0.998
9.74	0.01	0.727 \pm 0.023	11.512 \pm 0.360	0.998
6.30	0.06	0.937 \pm 0.031	12.659 \pm 0.417	0.994
2.68	0.06	0.951 \pm 0.031	11.280 \pm 0.367	0.995
K_s° Average =			11.82 \pm 0.75	
Naphthalene				
9.51	0.10	0.874 \pm 0.027	2.122 \pm 0.066	0.997
9.76	0.06	0.874 \pm 0.028	2.075 \pm 0.065	0.997
9.61	0.01	0.943 \pm 0.029	2.142 \pm 0.066	0.998
6.36	0.06	0.940 \pm 0.030	2.007 \pm 0.064	0.997
2.95	0.06	0.900 \pm 0.028	2.128 \pm 0.067	0.997
K_s° Average =			2.09 \pm 0.06	
K° in l/g		SD = Standard Deviation		

K_s° in L/g

SD = Standard Deviation



Phen $F_o/F = 1.313 + 16.996 (PPL-HEX)$, $R = 0.997$
 Fluo $F_o/F = 0.903 + 12.577 (PPL-HEX)$, $R = 0.998$
 Naph $F_o/F = 0.874 + 2.122 (PPL-HEX)$, $R = 0.997$

Figure 3. Stern-Volmer Plots for Sorption of PAHs onto PPL-HEX (pH_{av} 9.64, Ionic Strength 0.10 M).

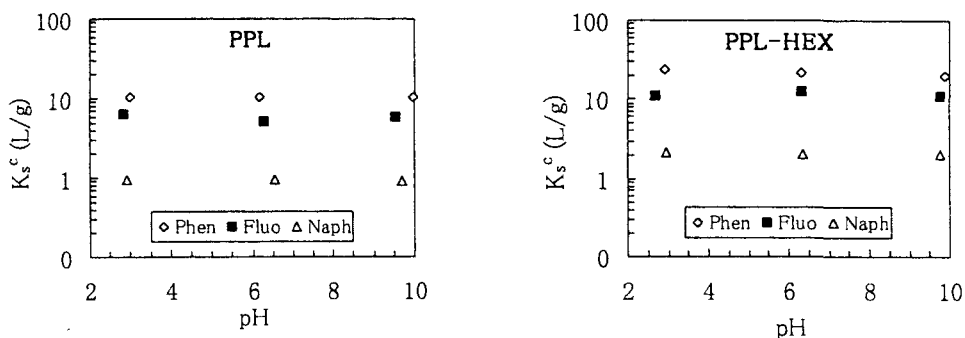


Figure 4. Effect of pH on K_s^c , Sorption of PAHs onto Polypropylene (PPL) and Polypropylene Coated with *n*-Hexane (PPL-HEX) (Ionic Strength 0.06 M).

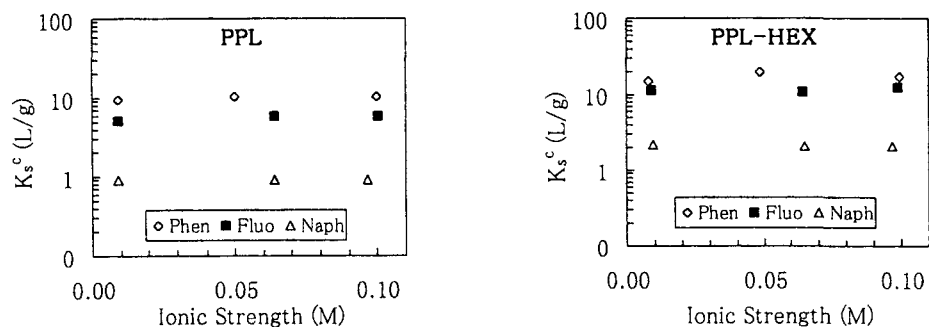
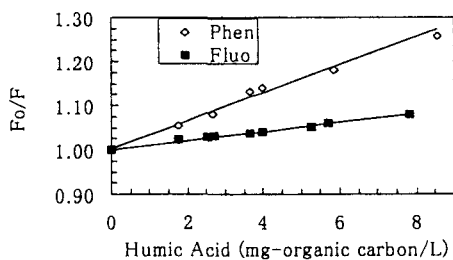


Figure 5. Effect of Ionic Strength on K_s^c , Sorption of PAHs onto Polypropylene (PPL) and Polypropylene Coated with *n*-Hexane (PPL-HEX) (pH_{av} 9.72).



$$\text{Phen. } F_0/F = 1.0035 + 0.0316 (\text{Humic Acid}), R = 0.9823$$

$$\text{Fluo: } F_0/F = 1.0004 + 0.0102 (\text{Humic Acid}), R = 0.9931$$

Figure 6. Stern-Volmer Plots for Sorption of Phenanthrene and Fluorene onto Humic Acid (pH_{av} 7.06, Ionic Strength 0.06 M).

3.3 Sorption onto PPL-HEX (in Presence of Humic Acid)

The amount of PAH sorbed onto PPL-HEX was determined by the following two methods: (1) Calculating the difference between the initial PAH concentration and the total PAH concentration in the aqueous phase after equilibration as measured by the HPLC method. (Hereafter referred to as the HPLC method). (2) Since the sorption coefficient for the humic acid is experimentally obtained and the concentration of free (unsorbed) humic acid and free PAH is known, the amount of PAH sorbed onto humic acid can be calculated from the sorption coefficient. The amount of PAH sorbed onto PPL-HEX can then be determined from a mass balance of the total PAH in the system. (Hereafter referred to as the K_s^c humic method).

The concentration of humic acid at the end of the experiment did not change. Humic acid was thus not adsorbed onto PPL-HEX. Similar experiments using PPL as the adsorbent also showed that PPL does not adsorb humic acid. Stern-Volmer plots were obtained for the two methods above for the sorption of phenanthrene and fluorene onto PPL-HEX. The HPLC method gives K_s^c values of 15.57 ± 0.46 and 12.41 ± 0.37 L/g for phenanthrene and fluorene, respectively; while the K_s^c humic method gives corresponding values of 15.53 ± 0.47 and 12.38 ± 0.35 L/g. It can be seen that the two methods give the same values of K_s^c within limits of experimental and analytical errors. The coefficient of correlation was greater than 99% for both methods. The K_s^c values compare with the average values of K_s^c for sorption onto PPL-HEX in absence of humic acid (Table 3). In calculating the above values of K_s^c , the concentration of PAH in aqueous phase [C_e in Equation (1)] is taken as the free PAH (PAH not combined with humic acid) in aqueous phase. These results suggest the presence of humic acid has no effect on the conditional sorption coefficient, K_s^c , of phenanthrene and fluorene onto PPL-HEX. In other words, humic acid does not compete with free PAHs for sorption onto PPL-HEX. However, in the presence of humic acid, those PAHs sorbed onto humic acid remain in solution and are not removed by sorption onto PPL-HEX. Thus, the presence of DOM in the aqueous phase interferes with the sorption of PAHs through the binding of PAHs onto DOM which cannot be sorbed onto the synthetic adsorbents.

4. Conclusions

This research accounts for the sorption of hydrophobic micro-organic pollutants onto polypropylene (PPL) and polypropylene coated with *n*-hexane (PPL-HEX). The following conclusions can be drawn from the results: (1) Both PPL and PPL-HEX adsorb PAHs (hydrophobic compounds). The sorption capacity (as measured by K_s^c) for sorption onto PPL-HEX is about 2 times greater than that for sorption onto PPL. Equilibrium is achieved in about 4 times faster with PPL-HEX than with PPL. Therefore, considering sorption capacity and sorption rate, PPL-HEX is the better adsorbent for PAHs than PPL. (2) Ionic strength and pH have no effect on sorption onto PPL and PPL-HEX. Sorption isotherms are linear and hydrophobic interactions are the dominant mechanisms involved in the sorption. (3) PPL and PPL-HEX do not adsorb humic acid and the presence of humic acid in the aqueous phase does not affect the sorption coefficient (K_s^c) of PAHs onto PPL-HEX. The finding that dissolved organic matter in form of humic acid does not compete with target PAHs for sorption of the PAHs onto PPL and PPL-HEX is a major advantage of these two adsorbents over activated carbon. We are currently investigating the practical use of PPL-HEX as a coalescing medium in upflow coalescence column filtration whereby HOCs are sorbed in hexane. As filtration proceeds, the hexane coalesces on the surface of PPL forming large droplets, which rise and collect above the water surface. The accumulated hexane is then separated from the aqueous phase. To remove target HOCs sorbed onto DOM which remain in solution and cannot be removed by PPL or PPL-HEX, a different scheme is also under investigation.

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