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Adsorption of antibiotics and iopromide onto single-walled and multi-walled carbon nanotubes

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HIGHLIGHTS

- Lincomycine, sulfamethoxazole, and iopromide adsorbed onto CNT.
- Freundlich isotherm model well fit adsorption of all target compounds.
- Adsorption of target compounds more onto single walled CNT than multi-walled CNT.
- Higher specific surface area of single walled CNT causing it to adsorb more organics.

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ABSTRACT

Engineered carbon nanotubes (CNTs) have shown a great promise for many remediation applications. The adsorption of two antibiotics (lincomycine and sulfamethoxazole) and one contrast medium (iopromide) on single-walled carbon nanotubes (SWCNT) and multi-walled carbon nanotubes (MWCNT) was investigated using batch adsorption experiments. These selected pollutants have high detection frequencies in aquatic environments. The adsorption results were compared with those of conventional powdered activated carbon (PAC). Adsorption isotherms for all pollutants on CNTs and PAC were nonlinear and could be described reasonably well with the Freundlich isotherm model. The adsorption generally followed the order SWCNT > PAC > MWCNT. The relatively low adsorption on MWCNT was probably due to its lower specific surface area than other carbon materials. However, correlation of adsorption to the surface area of carbon materials suggests other factors such as properties of adsorbate and type of interaction between pharmaceuticals and CNTs may also contribute to the adsorption processes. Implications of the adsorption results for the removal of pharmaceuticals from aqueous solution using CNTs are briefly discussed.

1. Introduction

There is an increasing concern on the impact of pharmaceuticals on drinking water supplies, which are usually not easily biodegradable and pose a risk of their deleterious effects to human beings and ecosystems [1–4]. Among the various pharmaceuticals, antibiotics have raised issues of antibiotic resistant bacteria and genes in the aquatic environment [5–9]. Besides antibiotics, there is also a concern on X-ray contrast medium, which has been detected in wastewater effluents, surface water, and drinking water at concentrations ranging from 0.5 to 15 $\mu g\,L^{-1}$ [4,10–12]. Wastewater treatment plants (WWTPs), which receive waste from hospitals or radiological clinics, have particularly shown high concentrations. Removal of pharmaceuticals and the contrast medium in water is immense important to meet the urgent need to clean water. Effective and sustainable water treatment technologies are critically required to meet the global demand of purified water.

Since the discovery of carbon nanotubes (CNTs) in 1991, engineered CNTs have shown great potential in many medical and environmental remediation applications [13,14]. CNTs contain cylindrical graphite sheets, which have very high van der Waals index [15]. The benzenoid rings of graphite sheets have sp^2-hybridized carbon atoms with high polarizability. These properties of CNTs make them superhydrophobic materials that may also strongly interact with aromatic pollutants through π–π coupling/

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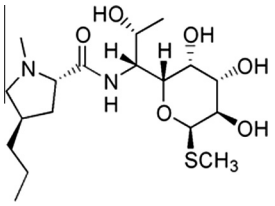
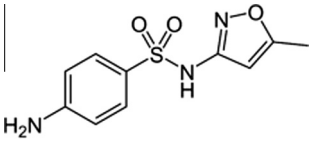
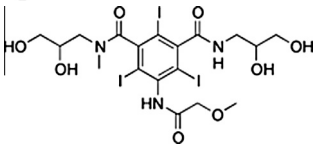
Table 1
Properties of studied antibiotics and contrast medium.

Name	Molecular formula	Structure	M.W.	M.V. (Å^3)[a]	Water Sol. (mg/L)	pK_a	K_{ow}
LCN (antibiotics)	$C_{18}H_{34}N_2O_6S$		406.5	384.9	927	7.8	0.56
SMX (antibiotics)	$C_{10}H_{11}N_3O_3S$		253.3	204.6	610	pK_{a1} = 1.7[b] pK_{a2} = 5.6[c]	0.89
IPR (contrast medium)	$C_{18}H_{24}I_3N_3O_8$		791.1	445.3	23.8	9.9[b]	−2.05

[a] van der Waals molecular volume (from http://www.chemicalize.org).
[b] [25].
[c] [26].

stacking [16,17]. Examples include nitroaromatics and amino- and hydroxyl-substituted aromatic compounds [18,19].

Studies on adsorption of pharmaceuticals onto CNTs are forthcoming [20–24]. The focus of the present study is on the removal of selected pharmaceuticals and a contrast medium through their adsorption onto CNTs, which have different structural and surface properties. The antibiotics under study were lincomycine (LCN) and sulfamethoxazole (SMX), which have amide and sulfonamide moieties, respectively (Table 1). The contrast medium was iopromide (IPR) that has also amide moieties (Table 1). These pollutants have been detected in water and wastewaters [4]. The tested CNTs for sorption experiments were single-walled CNTs (SWCNTs) and multi-walled CNTs (MWCNTs). The experiments with powdered activated carbon (PAC) were also performed for the comparative purpose. Sorption studies on SMX using CNTs have been carried out [20], but no similar studies with LCN and IPR are known in literature. The objectives were (i) to investigate the sorption behavior of the selected pharmaceuticals onto CNTs, (ii) to understand the influence of particle size and surface area of engineered carbon materials on the interaction between the studied molecules and CNTs, and (iii) to evaluate the potential of CNTs for enhanced removal of micropollutants.

2. Materials and methods

2.1. Standards and reagents

SWCNTs (purity > 95%, length 1–5 μm, and outer diameter 1.5 nm) and MWCNTs (purity > 95%, length 1–5 μm, and outer diameter 15 ± 5 nm) were purchased from Nano Lab (Newton, MA, USA) and were used without further purification. Based upon the information provided by the manufacturer, both CNTs have a hollow structure and were produced by a conventional chemical vapor deposition (CVD) method. Coconut-based PAC was obtained from Dongyang Carbon Co., Korea (Cheonan, Korea). Prior to use, the PAC was ground to reduce their particle sizes down to 60–140 mesh (100–250 μm).

Three pharmaceuticals, i.e., LCN (Sigma–Aldrich, St. Louis, MO, USA), SMX (Sigma–Aldrich, St. Louis, MO, USA), and IPR (USP, Rockville, MD, USA) all of reagent-grade were purchased for use. Stock solutions of three pharmaceuticals (concentration: 12,000 mg L^{-1}) were prepared by dissolving into HPLC-grade methanol (J.T. Baker, Philipsburg, NJ, USA).

2.2. Sorption experiments

All sorption experiments were carried out using a batch reactor. Sorption experiments onto three adsorbents (i.e., SWCNT, MWCNT, and PAC) were performed as a function of pharmaceutical concentration. The stock solutions of target pharmaceuticals were obtained by dissolving their solids into methanol, whereby the content of methanol in the final aqueous phase did not exceed 0.1% of total volume. Before adsorption experiments, the equilibrium between dried adsorbent and NaCl background solution was initially obtained by mixing appropriate amounts of the adsorbent and 0.01 M NaCl background solution into 40 mL amber EPA vials, equipped with Teflon-lined screw caps (Samsung Tech, Kyunggi, Korea). The resulting mixture was shaken for 24 h.

In the adsorption experiments, a certain volume of the concentrated stock solutions of LCN, SMX, and IPR (concentration: 12,000 mg L^{-1}) were added into tubes containing equilibrated adsorbent in NaCl background solution. The tubes were sealed with Teflon-lined screw caps and shaken at 150 rpm at 20 ± 1 °C. For kinetic study, the supernatants were withdrawn from the tubes after shacking them for 0, 6, 24, 72, 120, and 264 h, and filtered using a 0.2 μm pore size filter. For equilibrium study, the tubes containing the adsorbents were shaken for 72 h. The filtered solutions were stored in a dark place at 4 °C before subjected to analyses. At the end of each adsorption experiments, the equilibrium pH was measured; it was 6.0 ± 0.2.

2.3. Analysis

The concentrations of pharmaceuticals in the supernatant were measured using a liquid chromatography–mass spectrometry/mass spectrometry (LC–MS/MS) technique. The LC–MS/MS system consisted of LC (LC-20A, Shimadze, Kyoto, Japan) and a triple quadruple MS (API-3200, AB-Sciex, MA, USA) with an electrospray ionization (ESI) probe. The column used for analyzing LCN and SMX was a Shim-pack XR-ODS II (length: 75 × 3.0 mm; particle size:

2.2 μm; Shimadzu, Kyoto, Japan). In analyzing IPR, ACE C18 AR (length: 50 × 2.0 mm; particle size: 3.0 μm; Shimadzu, Kyoto, Japan) was used. Mobile phase used in analyzing LCN and SMX was mixture of 5 mM ammonium acetate and acetonitrile while mixture of 0.1% formic acid and acetonitrile was used for analyzing IPR. The column temperature was 40 °C and the injection volume of a sample was 5 μL. The ionization mode was ESI positive and the ESI probe capillary's voltage was set at +4.5 kV. The N_2 gas flow rate was 10 L min^{-1} and the temperature of MS interface was set at 350 °C. Information on the performance of the analytical method used in the study is summarized in Text SM-1 and Tables SM-1 and SM-2.

3. Results and discussion

3.1. Characteristics of adsorbents

The structural properties of carbon materials used in the study are given in Table 2. Both CNTs have similar lengths, but differ in outer diameter and BET surface areas (Table 2). The BET surface area of SWCNT is approximately four times higher than that of MWCNT. Comparatively, the BET surface area of PAC is the highest; a slightly more than that of SWCNT. The size of PAC used in this study is 60–140 mesh (100–250 μm).

3.2. Adsorption of LCN

Initially, adsorption kinetics using LCN was performed on all three adsorbents. The results are presented in Fig. 1. Apparent equilibrium of LCN for MWCNT was reached within 6 h, while adsorption equilibrium for SWCNT and PAC was obtained after 70 and 140 h, respectively, although more than 90% equilibrium of LCN for PAC was observed in less than initial 100 h. The adsorption of LCN can be characterized by two processes of different kinetics in which a fast initial adsorption to outer surfaces was followed by a slow diffusion into interlayers of carbon materials. The sorption kinetics of LCN for SWCNT or MWCNT was faster than that for PAC (Fig. 1). This was somewhat unexpected because surface area of PAC was higher than that of MWCNT (see Table 2). It seems that the LCN molecules may be fitting better into the interstitial area of MWCNT than that of the adsorption sites of PAC, causing the observed trend in the adsorption kinetics of LCN on carbon materials (Fig. 1).

Table 2
Properties of CNTs and PAC.

Carbon Material	Outer diameter (nm)	Length (μm)	BET surface area (m^2 g^{-1})	Average pore diameter (nm)[a]
SWCNT	~1.5	1–5	1020	3.5
MWCNT	15 ± 5	1–5	235	12
PAC	–	–	1130	1.8

[a] Determined by calculating 4 V A^{-1} by BET for each carbon material.

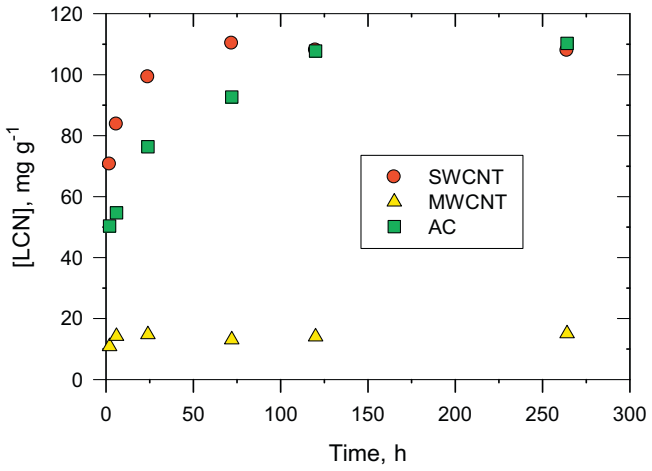


Fig. 1. Adsorption kinetics of lincomycin onto carbon nanotubes and activated carbon.

3.3. Adsorption isotherms

Initially, the study on adsorption equilibrium was performed for LCN. The results of adsorbed concentration (q, mmol kg^{-1}) versus aqueous-phase concentration at equilibrium (C_e, mmol L^{-1}) are presented in Fig. 2A. As can be seen, the sorption capacity of LCN on SWNTs is much higher than that on MWCNTs. Adsorption of LCN to the surfaces of CNTs and PAC was non-linear. The order of adsorption was SWCNTs > PAC > MWCNTs, which may be related to surface area (see Table 2). Initially, the results of Fig. 2A were interpreted using the Langmuir adsorption isotherm. The fitting of the data using the Langmuir isotherms was not good (R^2 < 70%). This indicates that adsorption of LCN on studied CNTs and PAC was not pure monolayer type. Similar results were also seen for the adsorption of aromatic hydrocarbons by carbon nanomaterials [24].

The Freundlich sorption model, represented by an empirical equation (Eq. (1)), was considered to describe the adsorption isotherms of Fig. 2A:

$$q_e = K_F C_e^n \quad (1)$$

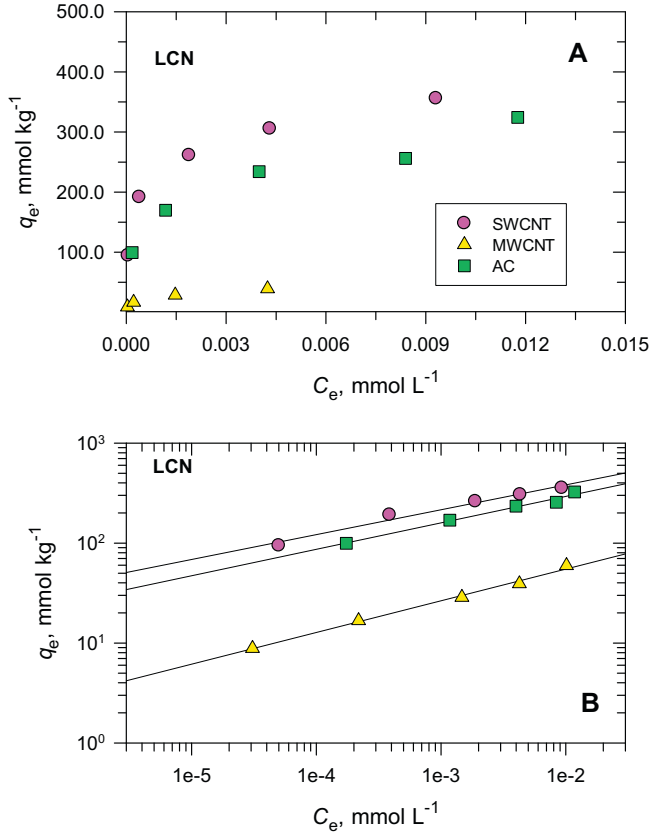


Fig. 2. (A) Adsorption isotherms as adsorbed concentration (q, on unit mass basis) versus aqueous-phase concentration (C_e) at equilibrium for lincomycine. (B) Plots of Freundlich isotherms of the data in (A).

where C_e is the aqueous phase concentration of sorbate at equilibrium (mmol L^{-1}), q_e is the adsorbed mass of sorbate per unit mass of sorbent (mmol kg^{-1}), K_F is the Freundlich affinity coefficient (mmol^{1-n} L^n kg^{-1}), and n (dimensionless) is the Freundlich linearity index. The Freundlich model was fitted to the adsorption data very well (R^2 > 96%) (Fig. 2, the solid lines are fitted lines). The fitting parameters of the Freundlich model are given in Table 3. The values of n were smaller than 1, which suggests high adsorption nonlinearity. This further suggests that more heterogeneous adsorption sites due to particular adsorption interactions. A wider pore size distribution of LCN on carbon materials may also occur [20].

As shown in Fig. 3, results of adsorption isotherm of SMX and IPR onto CNTs and PAC could also be reasonably described by the Freundlich model (Fig. 3). The fitted parameters are also presented in Table 3. The values of n were less than 0.5; again suggesting the nonlinear adsorption of SMX and IPR onto carbon materials under study. Interestingly, values of n obtained in the present study (~0.2–0.4) were similar to those observed for adsorption of nitrobenzene and phenol (Table 3). It seems adsorption of different pharmaceuticals onto carbon nanomaterials may be influenced by the moieties of the adsorbate molecule. Such adsorption behavior could also been seen for other pharmaceuticals such as tetracycline, triclosan, and tylosin [20,24] (Table 3).

The Freundlich affinity coefficient, K_F, obtained from the isotherms, had lower value for MWCNTs than those for SWCNTs and PAC. The general trend was SWCNTs ⩾ PAC > MWCNTs. There was no clear trend among the adsorption of pharmaceuticals onto carbon materials. This is indicating possible roles of chemical properties/structures of adsorbate on adsorbent, which have also been suggested in the adsorption of ibuprofen and triclosan onto CNTs [21]. The present study showed relatively higher values of K_F than those obtained in previous studies on the adsorption of pharmaceuticals onto CNTs (see Table 3).

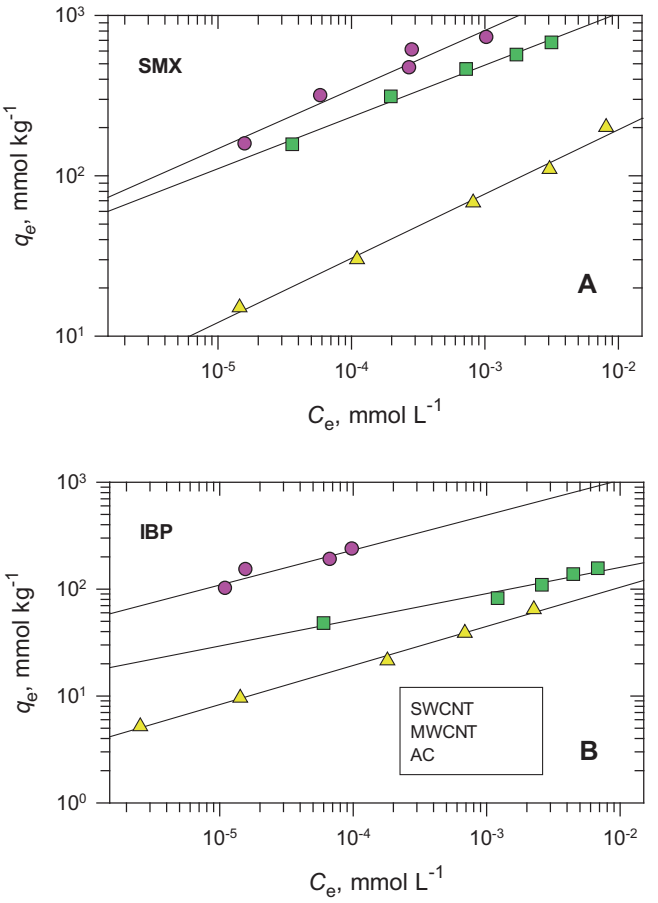


Fig. 3. (A) Freundlich adsorption isotherms for sulfamethoxazole (A) and iopromide (B).

Table 3
Freundlich model parameters, K_F and n ± standard deviation for adsorption/desorption on SWCNT, MWCNT, and AC.

Adsorbent	K_F, mmol^{1-n} L^n kg^{-1}	n	R^2	Refs.
LCN				
SWCNT	$(1.03 \pm 0.17) \times 10^3$	0.22 ± 0.02	0.985	This study
MWCNT	$(2.87 \pm 0.49) \times 10^2$	0.35 ± 0.03	0.987	This study
PAC	$(1.00 \pm 0.18) \times 10^3$	0.27 ± 0.03	0.968	This study
SMX				
SWCNT	$(6.38 \pm 2.91) \times 10^3$	0.31 ± 0.06	0.931	This study
	$(6.90 \pm 0.20) \times 10^2$	0.10 ± 0.006	0.962	[20]
MWCNT	$(2.01 \pm 0.51) \times 10^3$	0.48 ± 0.05	0.986	This study
	$(4.90 \pm 0.30) \times 10^2$	0.39 ± 0.009	0.994	[20]
PAC	$(3.78 \pm 0.46) \times 10^3$	0.30 ± 0.02	0.993	This study
IPR				
SWCNT	$(4.13 \pm 2.12) \times 10^3$	0.31 ± 0.05	0.953	This study
MWCNT	$(7.85 \pm 0.99) \times 10^2$	0.41 ± 0.02	0.997	This study
PAC	$(6.71 \pm 1.64) \times 10^2$	0.30 ± 0.04	0.967	This study
Tetracycline				
SWCNT	$(6.90 \pm 0.20) \times 10^2$	0.10 ± 0.006	0.962	[20]
MWCNT	$(3.20 \pm 0.10) \times 10^2$	0.17 ± 0.007	0.981	[20]
Triclosan				
MWCNT	5.26×10^2	0.09	0.98	[24]
Tylosin				
SWCNT	$(7.40 \pm 0.20) \times 10^2$	0.15 ± 0.006	0.986	[20]
MWCNT	$(3.50 \pm 0.10) \times 10^2$	0.22 ± 0.010	0.984	[20]
Nitrobenzene				
SWCNT	$(2.20 \pm 0.10) \times 10^3$	0.39 ± 0.009	0.994	[20]
MWCNT	$(1.75 \pm 0.08) \times 10^2$	0.34 ± 0.01	0.990	[20]
Phenol				
SWCNT	$(3.03 \pm 0.05) \times 10^2$	0.42 ± 0.005	0.998	[20]
MWCNT	$(6.4 \pm 0.10) \times 10^1$	0.34 ± 0.004	0.998	[20]

The adsorption of the three target pharmaceuticals, particularly for IPR, the bulkiest adsorbate onto SWCNT was similar to or stronger than PAC in spite of their similar specific surface areas (Table 2). Mechanisms involve electrostatic interactions, hydrophobic interactions, hydrogen bonds, and - interactions adsorption of the target pharmaceuticals onto the carbon materials. Since the three pharmaceuticals under study are very hydrophilic, electrostatic interactions, and hydrogen bonds might also contribute to the adsorption, which requires thorough investigation in future studies.

In this study, the higher adsorption of the pharmaceuticals onto SWCNT than that onto PAC was attributed to the molecular sieving effect [27]; the average pore size of PAC is smaller than that of SWCNT (Table 2). Nonetheless, these results suggest that CNTs were more effective for adsorption of large molecules because surface areas on CNTs are likely more accessible to them than those on PAC.

This could be related to the surface area of CNTs, hence a plot was constructed between K_F versus the surface area (Fig. 4). LCN showed a positive correlation between K_F and surface area (R^2 = 0.98%). The results of the present study were combined with results of other study for SMX (Table 3). Adsorption of SMX had a weak relationship with the surface area (R^2 = 0.68%) (Fig. 4). The adsorption of IPR showed no relationship with the surface area (R^2 = 0.11%) (Fig. 4). Although other factors should also be considered, the low R^2 for IPR was attributed to the molecular sieving effect [27]. Correlations described in Fig. 4 suggest that there are other properties besides surface area control the interaction between adsorbate and carbon nanomaterials. Other properties include the nature of adsorption or interactions between pharmaceuticals and CNTs. The structure of the pharmaceutical and pore

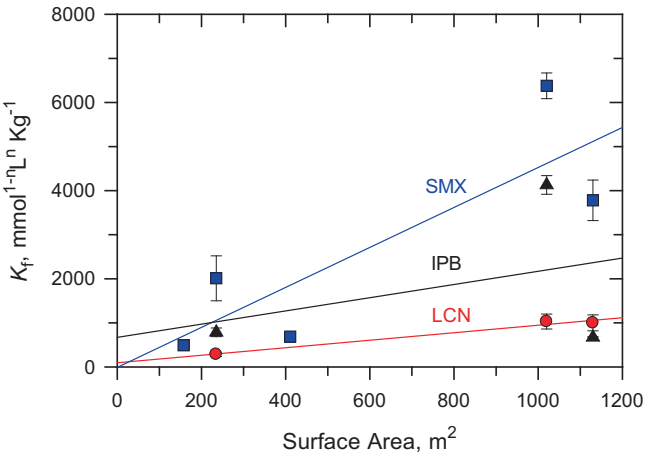


Fig. 4. Correlations of Freudlich affinity coefficient K_F with surface area of CNTs for different compounds.

structure of the CNTs used in the present study will have influence on such interactions. Overall, cumulative contributions of such different interactions and surface area will determine the adsorption affinity of adsorbate to the CNTs.

4. Conclusions

CNTs and PAC could effectively adsorb the three pollutants. The adsorption of LCN by different material was almost complete within 100 h and the adsorption kinetics had two phases, rapid step was followed by a relatively slow step. Among the different materials, SWCNT has higher potential to adsorb LCN, SMX, and IPR than MWCNT and PAC. The surface area of the material significantly controlled the adsorption of the pollutants, but other factors including the moieties on the pollutants may also contribute to the overall adsorption process of the pollutants on CNTs. In future, similar kinetics and isotherm studies in combination with spectroscopic characterization of surfaces of CNTs may give insight on influence of moieties of pharmaceuticals and the type of interactions between the pollutants and CNTs.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.cej.2014.06.035.

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