

Application of Characterization Procedure in Water and Wastewater Treatment by Adsorption

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Abstract—The background organics in water and wastewater are necessary to fractionate into groups or components according to the difference in adsorbability. In this study, the background organics were fractionated in terms of the adsorptive strength described by the Freundlich isotherm constants k and n with the assumption that the fractionated components differ in the value of k but have the same value of n based on the IAST (Ideal Adsorption Solution Theory) using binomial concentration distribution. A simple characterization for water and wastewater with a certain amount of DOC in terms of adsorbability was applied to three types of organic mixtures contained in different water sources. The composition of each organic mixture was successfully evaluated to describe the IAE (Integral adsorption experiments) data of the total organic carbon by using this characterization procedure. Batch experiments as well as the membrane-PAC hybrid system experiments were conducted with three different types of PACs.

Key words: DOC (Dissolved Organic Carbon), Adsorption, Wastewater Treatment, PAC (Powdered Activated Carbon), Characterization Procedure, Membrane-PAC Hybrid System

INTRODUCTION

Adsorption processes can be used to remove dissolved organic carbon (DOC) such as humic acids, fulvic acids and organic carbon representing from 40 to 99% of the carbon content in water and wastewater. These DOCs in water and wastewater have a wide distribution of molecular weight, i.e., a variety of adsorption characteristics of the components.

The design of equipment for the adsorption process requires knowledge of the overall adsorption equilibrium of such complex systems, but classical procedures for multicomponent adsorption are unsuitable in these cases, mainly because of the impossibility of isolating and identifying the components by ordinary chemical analyses. Much of water and wastewater treatment requires adsorption treatment for purification or removal of DOCs whose identities are not completely known. The lack of information on adsorbate identity and composition made it difficult to apply adsorption theories to the design of treatment processes for these systems. One has to determine a few pseudospecies to handle such a mixture according to the multicomponent adsorption calculation method for unknown species in water and wastewater. The results of the earlier works have shown the utility of the characterization procedure to get information for the design of adsorption equipment. A proper characterization method is therefore required to describe such adsorption systems [Okazaki et al., 1981; Moon et al., 1991; Maria et al., 1994; Kim et al., 2002]. Okazaki et al. [1981] suggested a useful concept, the characteristic distribution of Langmuir coefficient, to describe adsorption equilibrium of wastewater in which many

unknown species exist. The wastewater encountered can then be characterized by its concentration frequency function, assuming that the number of pseudospecies is infinite. However, while conceptually simple and elegant, it is very difficult to implement this concentration frequency function in simulating batch and fixed bed adsorption systems. Considering this point, Jayaraj and Tien [1985] proposed a characterization procedure with a finite number of pseudospecies based on the concept of species grouping suggested by Calligaris and Tien [1982]. Assuming that the data of pure component isotherm may be described by the Freundlich equation with k and n as the coefficient and reciprocal of the exponent, pseudospecies may be composed of a number of parameters which characterize the solute adsorption affinity. They assumed that all the pseudospecies obey the Freundlich expression and the IAST (ideal adsorbed solution theory) is valid in describing multicomponent adsorption equilibrium [Yuasa et al., 1996; Matsui et al., 1998]. More recently, Maria et al. [1994] devised a continuous model for complex mixture adsorption using a rectangular, gamma or log-normal distribution of composition in terms of a characterizing variable, namely one of the adsorption constants.

In this work, the simple characterization procedure depicted by Moon et al. [1991] and Kim et al. [2001] was further tested for surface water and wastewater solutions in which organic and inorganic compounds exist together. From equilibrium experiments, it was found that these polluted solutions have a little unfavorable type isotherm on Powdered Activated Carbon (PAC). This paper presents the results obtained by the application of different methods for the characterization of DOC in water and wastewater and its fractions of different origin. It is shown that availability of advanced characterization techniques offers the potential for predicting the adsorbability of DOC without precise knowledge of the chemical

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structures present in the water and wastewater.

THEORETICAL APPROACH

The requirement that the calculated and experimental values agree with each other provides the basis for determining the concentration of the pseudospecies. An objective function, F , may be defined as:

$$F = \frac{100}{ND} \sum_{m=1}^{ND} [(C_{Texp} - C_{Tcal})/C_{Texp}]^m \quad (1)$$

The pseudospecies concentrations are determined by minimizing F , subject to the constraint that the sum of the pseudospecies concentrations equals the total concentration of the solution C_T . IAST-Freundlich model was used to classify DOC into components based on the adsorbability. ND is the number of data. The batch adsorption from a solution containing several unknown adsorbates can be characterized by a concentration density function, C_{io} . Let the volume of the solution be V and the mass of fresh adsorbate be M . Then, the solution and adsorbed phase concentrations, C_i and q_i , of the i -th species at equilibrium are related by the following mass balance equation.

$$V \frac{dC_i}{dt} + M \frac{dq_i}{dt} = 0 \quad (2)$$

The initial conditions of Eq. (1) are

$$C_i = C_{io}, q_i = 0, t = 0 \quad (3)$$

The integrated form of Eq. (1) is

$$C_i + (M/V)q_i = C_{io} \quad (4)$$

The initial concentration is given

$$C_{io} = x_{io} C_{To}, i = 0, 1, 2, \dots, N \quad (5)$$

where C_{To} is the total initial DOC concentration of the solution and x_{io} is the initial DOC fraction of the i -th species. The species, $i=0$, means a nonadsorbable species in the solution.

The characterization principle in this study is as follows. Once a solution is characterized and assuming that the IAST is applicable, one can estimate the equilibrium concentrations achieved by contacting the solution with a given amount of adsorbent. If the multi-component equilibrium can be described by the IAST, the relationships among concentrations are given by the following set of equations.

$$C_i = C_i^o z_i \quad (6)$$

$$q_i = q_i^o z_i \quad (7)$$

$$q_T = \left[\sum_{i=1}^N z_i / q_i^o \right]^{-1} \quad (8)$$

$$\pi_i = (RT/A) \int_0^{C_i} q_i^o / C_i^o dC_i^o \quad (9)$$

$$\sum_{i=1}^N z_i = 1.0 \quad (10)$$

$$q_i^o = k_i C_i^{o^{1/n}} \quad (11)$$

where z_i is the mole fraction of the i -th species in the adsorbed phase and C_i^o is the concentration of the i -th species in its single-species state, corresponding to the spreading pressure, π_i , at equilibrium. q_i^o is the equilibrium concentration corresponding to C_i^o . A is the area of the solution-adsorbent interface per unit weight of adsorbent. One may assume that all the pseudospecies obey the Freundlich expression like Eq. (11) to simplify the subsequent computation work required in both the characterization and the adsorption calculations. Integration of Eq. (9) yields

$$\Pi = \pi_i A / RT = nk_i C_i^{o^{1/n}} \text{ or } n \cdot q_i^o \quad (12)$$

From Eqs. (6) and (7), one can get the following relationship.

$$C_i^o = (\Pi / nk_i)^n \quad (13)$$

$$q_i^o = \Pi / n \quad (14)$$

Substituting Eq. (12) into Eq. (8), one has

$$q_T = q_1 = q_2 = \dots = q_N = \Pi / n \quad (15)$$

Eq. (14) represents that the total adsorption amount is equal to the adsorption amount of individual species at the single-species state. Substituting Eqs. (6), (7), (12), (14) into the material balance equation yields the mole fraction in the adsorbed phase as

$$z_i = \frac{C_{io}}{(\Pi / nk_i)^n + (M/V)(\Pi / n)} \quad (16)$$

$$\sum_{i=1}^N \frac{C_{io}}{(\Pi / nk_i)^n + (M/V)(\Pi / n)} - 1 = 0 \quad (17)$$

Π can be found by Newton's method from Eq. (17). Once Π is known, the equilibrium concentrations C_i and q_i can be calculated from the set of equations above. As the adsorption affinity is dependent upon both the Freundlich constants, one has some freedom to assign an arbitrary value to the reciprocal of the Freundlich exponent; n was assumed regardless of species and its value was determined by the optimization-search procedure. When a binomial distribution is used, the DOC fraction of the j -th species identified by a Freundlich coefficient, k_j , may be represented as follows [Kreyszig, 1977].

$$x_j = \sum_{j=1}^N s^j (1-s)^{N-j}, j = 0, 1, 2, \dots, N \text{ where } \sum_{j=1}^N N! / j!(N-j)! \quad (18)$$

The Freundlich coefficient, k_j , of the j -th species is assigned by the following equation to cover a wide range of k value.

$$k_j = k_j^3 \quad (19)$$

where k_s is the scale factor which represents the order of magnitude of the lowest k value for the solution in question. The range of the k value should be wide enough to cover all species, having a dense distribution at lower values.

The mass transfer rate between liquid and solid phases represented by the LDFA (Linear Driving Force Approximation) model assuming that overall mass transfer coefficient unchanged during experiments is the following equation, Eq. (20).

$$\frac{dq}{dt} = \frac{3 \times k_f}{R \times \rho_p} (c_i - c_s) = k_m (q_s - q) \quad (20)$$

where R =radius of adsorbent, k_f =overall mass transfer coefficient (m/s), ρ_p =density of particle (kg/m^3), k_m =mass transfer coefficient (1/s).

The adsorption rate of adsorbate by a PAC grain is linearly proportional to a driving force using the LDFA model, defined as the difference between the surface concentration and the average adsorbed-phase concentration. The IAE (Integral Adsorption Experiment) was used batch to get adsorption data as the basis for characterizing solutions of unknown compositions.

EXPERIMENTS

1. Working Solutions

Three kinds of waters were used as source of working solutions: (i) a domestic wastewater with DOC comprising the majority of the organic matter, (ii) a biological process effluent from a wastewater treatment plant in Sydney, Australia, and (iii) a wastewater treatment water and a surface water (Gwangju, Korea). Three PACs supplied by James Cummings & Sons PTY LTD in Australia were used as adsorbents in this study. Equilibrium was obtained after shaking for 5 days at 25 °C, and then the carbon particles were filtered by 0.45 μm membranes. The physical properties of these adsorbents measured via a Micromeritics ASAP 2000 automatic analyzer are shown in Table 1.

2. Batch Test Procedure

Batch adsorption experiments were performed at different RPM (50, 100, 150, and 200) to obtain the data required for the design and operation of a membrane-PAC hybrid system for the treatment of wastewater and surface water. The batch reactors were 1 L conical glass flask. In each flask, a known concentration of water was mixed with the known amount of adsorbent. The purpose of these experiments is to investigate the DOC removal process with time and to find the equilibrium DOC concentration. For the determination of liquid phase concentration, the PAC particles were filtered, and all samples taken during experiments were analyzed for DOC by using a UV-Persulfate TOC analyzer (DOHRMANN Phoenix 8000). Liquid samples were taken periodically by using an airtight precision syringe, withdrawing the solution from the vessel directly. All runs were carried out at a temperature of 25 °C.

3. Membrane-PAC Hybrid System

The schematic diagram of the microfiltration set-up used in this study is shown in Fig. 1. Wastewater and PAC were delivered from stock tanks (equipped with a stirrer) to the CFMF (Crossflow micro-

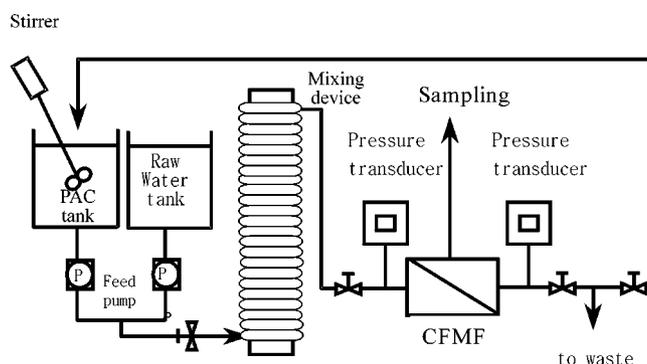


Fig. 1. Experimental set-up for PAC-membrane hybrid system.

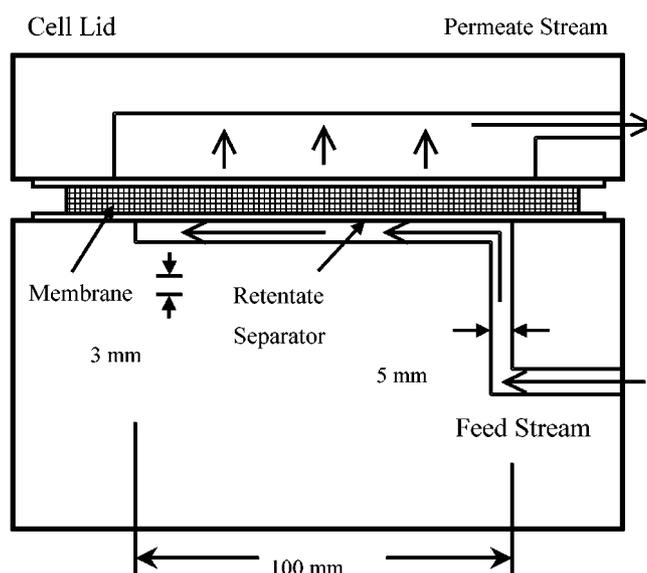


Fig. 2. Schematic diagram of the membrane cell.

Table 1. Characteristics of powdered activated carbons (PACs) tested

Specification	PAC-WB	PAC-CB	PAC-HA
Iodine number, mg/g min*	900	800	900
Ash content*	6% max	5% max	3% max
Moisture content*	5% max	8% max	10% max
Bulk density, kg/m^3 *	290-390	300-100	350-500
Surface area, m^2/g **	882	915	1199
Nominal size*	80% min finer than 75 micron	55% to 65% finer than 45 micron	75% min finer than 75 micron
Type*	Wood based	Coal based	Coconutshell based
Product code*	MD3545WB Powder	MD3545CB Powder	MD3545CO Powder
Mean pore dia., Å **	30.61	24.20	30.41
Micropore vol., cc/g **	0.34	0.192	0.067
Mean diameter, μm **	19.72	10.9	34.15

*From the manufacture's report.

**From the Micromeritics ASAP 2000 automatic analyzer.

filtration) as shown in Fig. 2 by a variable speed tubing pump. Both the permeate and retentate lines were returned to the stock tank to maintain constant inlet concentration. The pressure of membrane was controlled at 20 kPa by two valves and the transmembrane pressure drop was monitored by using two pressure transducers in order to measure the permeate flux.

The dimensions of the filtration channel in the CFMF cell are 6 cm, 0.6 cm and 0.036 cm of length, width, and thickness, respectively. The CFMF cell has nine filtration channels and the total membrane area is $3.24 \times 10^{-3} \text{ m}^2$.

A spiral-mixing device was made by winding a PVC tube with diameter of 6 mm around a column 110 mm in diameter. A coconut shell based PAC with nominal size of 80% finer than $75 \mu\text{m}$ was used. The PAC was fed into a mixing device along with the feed solution. After mixing with PAC, the solution was then delivered to the CFMF cell. The membrane used was hydrophilic PVDF (poly-vinylidene fluoride) membrane (MILLIPORE: Catalogue no. GVLP OMS 10) supplied by Millipore Corp. with nominal size of $0.65 \mu\text{m}$.

RESULTS AND DISCUSSION

1. Wastewater

Adsorption equilibrium experiments for simulation data at different kinds of PAC were conducted in a batch adsorber. As can be seen in Figs. 3 and 4, the overall batch adsorption isotherm and IAE experiments of wastewater on the influent concentration of 3.92 ppm in terms of the different PACs were represented. As shown in Fig. 4, predictions based on the characterization results were in good agreement with experimental data within the accuracy of experiment. The characterization procedure based on IAST was applied in the IAE experiments with wastewater in Korea. Fig. 5 shows the DOC fraction depending on Freundlich constant k . Using this binomial distribution, the adsorption isotherm on various influent concen-

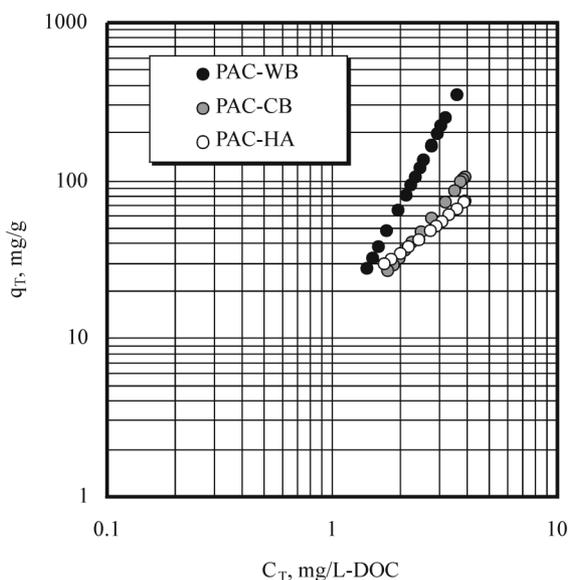


Fig. 3. The overall batch adsorption isotherms of wastewater (Gwangju, Korea) with the initial concentration of 3.92 ppm at different PACs.

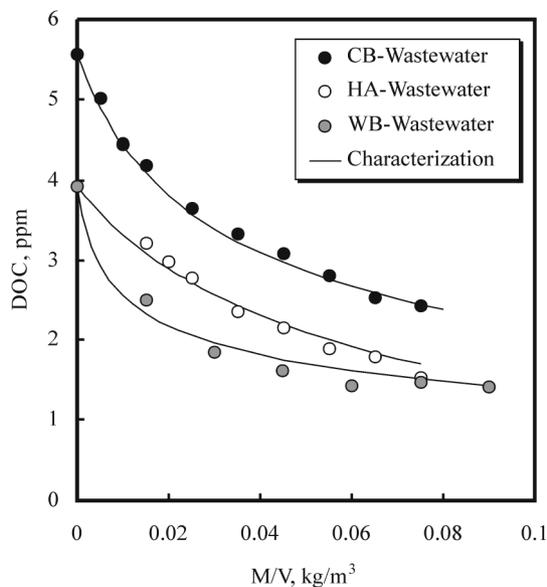


Fig. 4. Calculated and experimental IAE data at different PACs (wastewater in Gwangju, Korea).

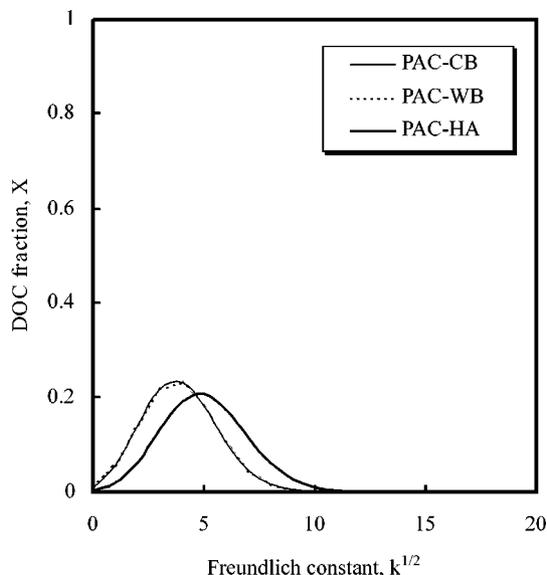


Fig. 5. DOC fraction of wastewater and Freundlich constant at different PACs (wastewater in Gwangju, Korea).

trations can be predicted. Tables 2 and 3 show the results of wastewater solution on the characterization procedure with $N=12$ and characterization results of wastewater in Korea on PAC. The values of X , calculated according to Eq. (18), and pseudospecies number (N) were chosen to be 12. The scale factor of the k value was determined to be 1.0 and the optimum values of n in terms of three PACs were 0.73, 1.68 and 0.4. PAC-WB was found to be the best for DOC removal in the range of high organic concentration. However, in the range of low organic concentration, PAC-HA is a better adsorbent for removing DOC in wastewater solution. The effect of mixing (in terms of rotational speed of the mechanical stirrer) in the batch adsorber is shown in Fig. 6. The mass transfer coefficients estimated directly from the concentration decay curve by minimiz-

Table 2. Search results of wastewater solution on the characterization procedure with N=12

Pseudospecies N	K_f	X			Average, X
		PAC-CB	PAC-HA	PAC-WB	
0	0	0.0101	0.0027	0.0100	0.0076
1	1	0.0548	0.0189	0.0547	0.0428
2	4	0.1385	0.0625	0.1383	0.1131
3	9	0.2154	0.1297	0.2153	0.1868
4	16	0.2304	0.1892	0.2304	0.2166
5	25	0.1792	0.2061	0.1792	0.1882
6	36	0.1045	0.1737	0.1046	0.1276
7	49	0.0465	0.1158	0.0465	0.0696
8	64	0.0158	0.0620	0.0158	0.0312
9	81	0.0041	0.0268	0.0041	0.0117
10	100	0.0008	0.0094	0.0008	0.0037
11	121	0.0001	0.0027	0.0001	0.0010
12	144	0.0000	0.0006	0.0000	0.0002
F (%)		2.0	4.2	5.1	3.8

ing the mean percent deviation between experimental and calculated concentrations are between 2.0×10^{-5} m/s to 1.0×10^{-5} m/s in terms of stirring speed in batch adsorber. The adsorption kinetics on various mixing RPM can be predicted by using this binomial distribution by the IAST-Freundlich model. That is the reason why we already know the components according to the difference in adsorbability. Regardless of the number of the pseudospecies, the DOC fraction was exactly the same in the optimum range and applied to predict the adsorption kinetic data during adsorption experiments under the unchanged mass transfer.

Figs. 7 and 8 show the overall batch adsorption isotherms of domestic wastewater by PAC-HA in Australia depending on the initial concentrations and IAE experiments on PAC-HA. Since the overall adsorption isotherm depends on influent concentrations, the DOC fraction in terms of Freundlich constant k in terms of pseudospecies number should be known (Fig. 9). DOC Concentrations distribution functions for PAC-HA were obtained comparing those pseudospecies numbers in the optimum range. This showed that the availability of simple characterization techniques offers the potential for predicting the adsorbability of DOC without precise knowledge of the chemical structures present in the water and wastewater. Table 4 shows the characterization results of secondary effluent from a wastewater treatment plant in Australia using PAC-HA as adsorbent. The scale factor of the k value was determined to be 1.0 and the optimum values of n was 1.0.

2. Water

Table 3. Characterization results of wastewater in Korea on PAC

k_f	N	n			s			F(%)*		
		CB	HA	WB	CB	HA	WB	CB	HA	WB
		5.57 ppm	3.92 ppm	3.92 ppm	5.57 ppm	3.92 ppm	3.92 ppm	5.57 ppm	3.92 ppm	3.92 ppm
1.0	16	0.73	1.68	0.4	0.28	0.28	0.28	9.8	11.4	8.6
	18	0.73	1.68	0.4	0.28	0.28	0.28	17.3	4.2	15.7
	20	0.73	1.68	0.4	0.28	0.28	0.28	20.0	7.1	21.9

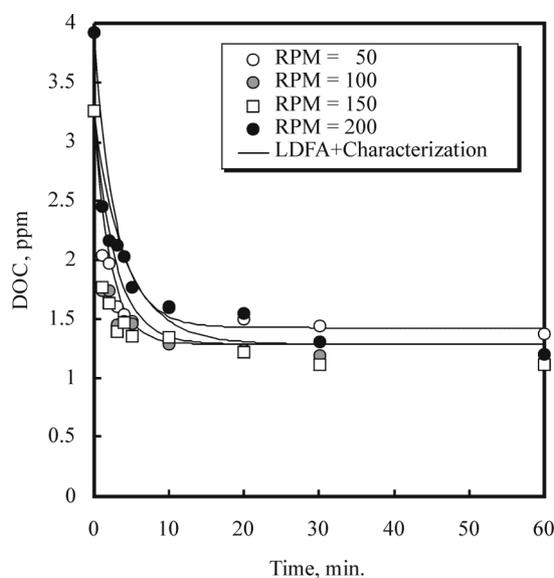
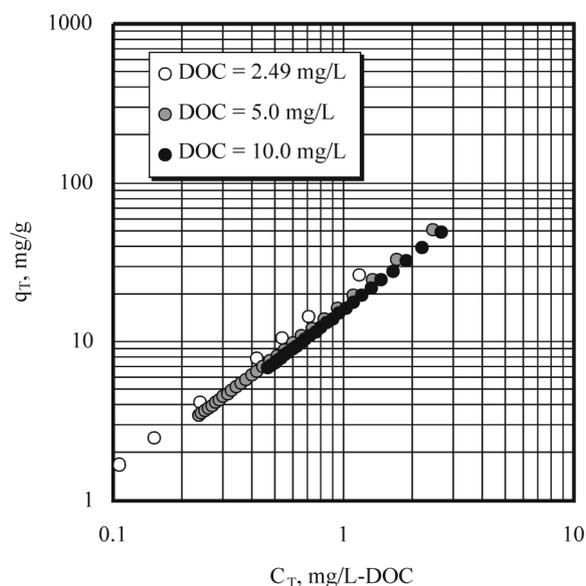
**Fig. 6. Effect of DOC concentration on mass transfer rate in wastewater in Gwangju, Korea (PAC-WB=150 mg/L).****Fig. 7. The overall batch adsorption isotherms of secondary effluent from a wastewater treatment plant in Sydney, Australia different initial concentrations.**

Fig. 10 shows the overall batch adsorption isotherm at the influent concentration of 2.24 ppm. The results show that PAC-WB and

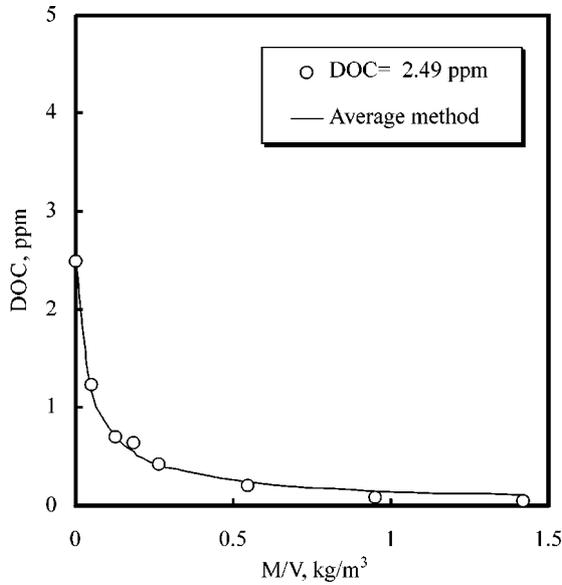


Fig. 8. Calculated and experimental IAE data at PAC-HA (wastewater in Sydney, Australia).

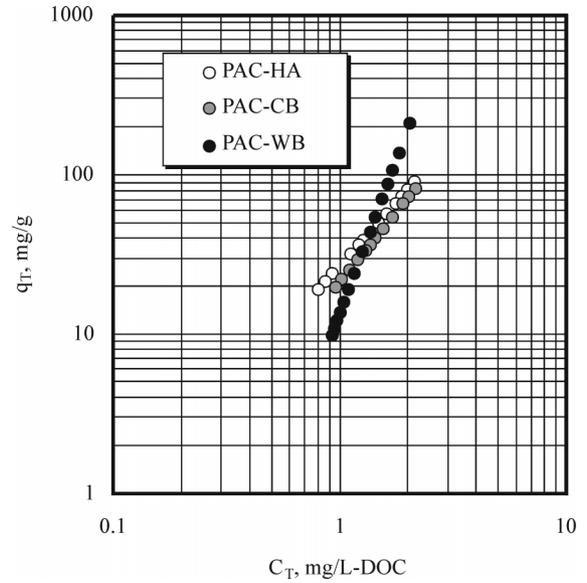


Fig. 10. The overall batch adsorption isotherms of surface water at different PACs (the initial DOC concentration=2.24 ppm).

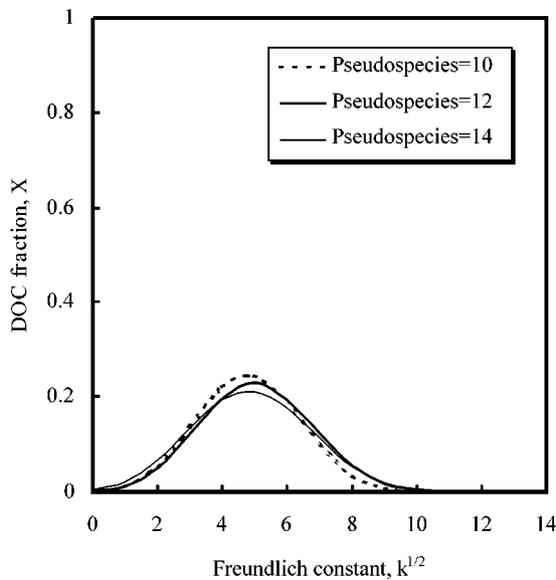


Fig. 9. DOC fraction of secondary effluent from a wastewater treatment plant in Sydney, Australia versus Freundlich constant at PAC-HA.

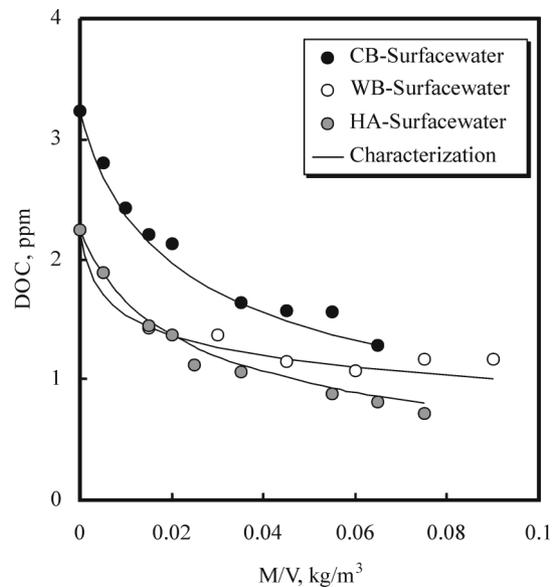


Fig. 11. Calculated and experimental IAE data at different PACs (surface-water in Korea).

Table 4. Characterization results of wastewater in Australia on PAC-HA IAE data with $C_{T0}=2.49$ ppm DOC

k_s	N	n	s	F(%)
1.0	10	1	0.48	37
	12	1	0.42	32
	14	1	0.36	35

PAC-HA could be used in range of high and low influent concentration (Figs. 10 and 11). The concentration DOC fraction was found to be dependent on Freundlich constant k in relation of skewness s (Fig. 12). The results of surface water solution at the characteriza-

tion procedure with $N=14$ and characterization results of surface water on PAC are shown in Tables 5 and 6. The scale factor of the k value was determined to be 1.0 and the optimum values of n in terms of three PACs were 0.67, 0.75 and 0.27. The value of X_i calculated according to Eq. (18) and pseudospecies number (N) was chosen to be 14.

From the batch experimental data and adsorption isotherm information, we could apply the adsorption process to removal DOC in water and wastewater such as membrane-PAC hybrid system. As the above explanation, in range of low concentration, PAC-HA was the best adsorbent to remove the DOC in aqueous solution. Membrane-PAC hybrid experimental data is shown in Fig. 13. The contact time and membrane pore size was 4 min and $0.65 \mu\text{m}$. In these

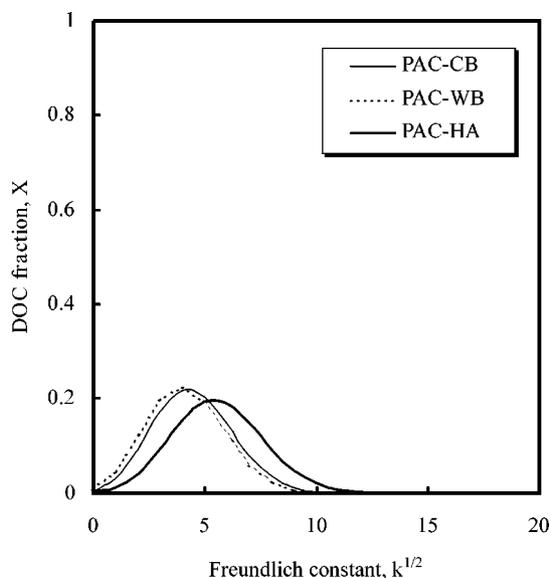


Fig. 12. DOC fraction of surface water and Freundlich constant at different PACs.

Table 5. Search results of surface-water solution on the characterization procedure with N=14

Pseudospecies N	K_i	X			Average, X
		PAC-CB	PAC-HA	PAC-WB	
0	0	0.0052	0.0014	0.0081	0.0049
1	1	0.0324	0.0109	0.0454	0.0296
2	4	0.0946	0.0403	0.1197	0.0848
3	9	0.1717	0.0940	0.1963	0.1540
4	16	0.2171	0.1553	0.2242	0.1989
5	25	0.2027	0.1933	0.1892	0.1951
6	36	0.1446	0.1879	0.1219	0.1515
7	49	0.0804	0.1462	0.0612	0.0959
8	64	0.0352	0.0924	0.0242	0.0506
9	81	0.0122	0.0479	0.0076	0.0225
10	100	0.0033	0.0205	0.0019	0.0086
11	121	0.0007	0.0072	0.0004	0.0028
12	144	0.0001	0.0021	0.0001	0.0008
13	169	0.0000	0.0005	0.0000	0.0003
14	196	0.0000	0.0001	0.0000	0.0000
F (%)		4.2	2.7	6.3	4.4

experiments, the experimental data and prediction data were not in good agreement because the phenomenon might be interpreted as

Table 6. Characterization results of surface-water in Korea (PAC as adsorbent)

k_s	N	n			s			F(%)*		
		CB	HA	WB	CB	HA	WB	CB	HA	WB
		3.23 ppm	2.24 ppm	2.24 ppm	3.23 ppm	2.24 ppm	2.24 ppm	3.23 ppm	2.24 ppm	2.24 ppm
1.0	16	0.67	0.75	0.27	0.28	0.37	0.26	4.2	2.7	6.2
	18	0.67	0.75	0.27	0.28	0.37	0.26	4.5	9.1	10.7
	20	0.67	0.75	0.27	0.28	0.28	0.26	11.3	5.9	15.2

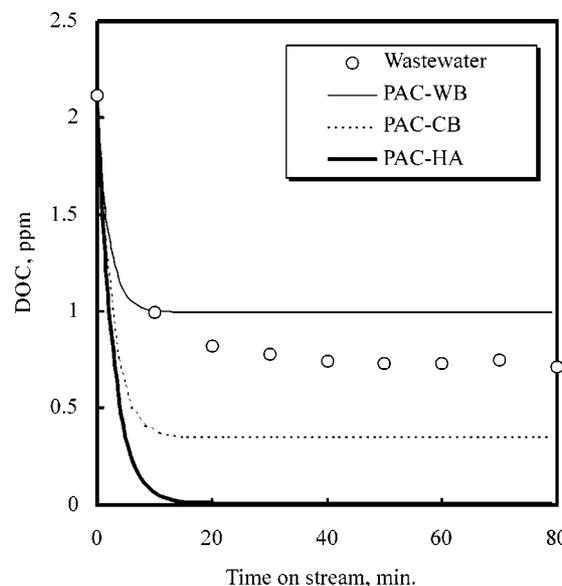


Fig. 13. Membrane-PAC hybrid experimental data and simulation in wastewater in Gwangju, Korea (Contact time=4 min, MPS=0.65 μ m).

coagulation rather than adsorption and diluted by solution of wastewater and PAC tank during the membrane-adsorption hybrid experiments.

This adsorption kinetic data was predicted under the unchanged overall mass transfer coefficient by using the binomial concentration distribution by the IAST-Freundlich model.

CONCLUSIONS

In order to interpret the multicomponent adsorption in water and wastewater, the characterization method explained in this study can be used to fractionate the background organics into groups or components according to the difference in adsorbability. The background organics were fractionated in terms of the adsorptive strength described by the Freundlich isotherm constants k and n with the assumption that the fractionated components differ in the value of k but have the same value of n based on IAST (Ideal Adsorption Solution Theory) using binomial concentration distribution. The composition of each organic mixture was successfully evaluated to describe the IAE (Integral adsorption experiments) data of the total organic carbon using this characterization procedure. It seems that the application of the characterization procedure will be very beneficial in designing and simulating any adsorption process systematically to remove DOC from water and wastewater.

ACKNOWLEDGEMENT

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NOMENCLATURE

A	: area of the solution-adsorbent interface [m ² /g]
C	: concentration in solution [ppm or mg/L]
C _T	: total adsorbate concentration in solution [ppm or mg/L]
F	: object function or average error defined on Eq. (1)
k	: Freundlich isotherm coefficient
k _f	: overall mass transfer coefficient [m/s]
k _m	: mass transfer coefficient [1/s]
k _s	: scale factor of k value
M	: mass of adsorbent [kg]
n	: Freundlich isotherm exponent
N	: number of pseudospecies
ND	: number of data
q	: concentration in adsorbed phase [ppm or mg/L]
q _T	: total adsorbate concentration in adsorbed phase [ppm or mg/L]
R	: radius of adsorbent
s	: skewness of binomial distribution function
V	: volume of solution [L]
x	: DOC fraction in solution
z	: DOC fraction in adsorbed phase

Greek Letters

π	: spreading pressure
Π	: modified spreading pressure defined in Eq. (5)
ρ _p	: density of particle [kg/m ³]

Abbreviations

DOC	: dissolved organic carbon
IAE	: integral adsorption equilibrium
IAST	: ideal adsorbed solution theory
PAC	: powdered activated carbon

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