CHAPTER III
EXPERIMENTS

3.1 Materials

In the present work, both jar tests and fixed bed adsorption experiments were carried out to investigate the adsorption isotherms and breakthrough characteristics of 4 single-component, 2 binary, 1 tertiary aqueous systems. In addition, an unknown multi-solute system and an industrial wastewater were also investigated.

o-xylenol(XLN), p-chlorophenol(PCN), benzoic acid(BA) and cyclohexanol(CHN) were chosen as representatives of industrial organic pollutants in the cases of single-component aqueous systems. Phenol compound was organic contaminants frequently found in wastewater from chemical and refinery plant. Furthermore, cyclohexanol and benzoic acid were chosen to investigate the effects of structural stericality and lypophilicity (adsorption affinity) on the adsorption isotherms.

The two binary systems were aqueous solutions of PCN+CHN and PCN+XLN and the tertiary aqueous system was BA+PCN+XLN.

As an unknown multi-solute substance, natural humic substance(NHS : collected by Y.Sudo, Tokyo National College of Technology, Dept. of Industrial Chemistry) and tapwater at Institute of Industrial Science, University of Tokyo were used. The latter contained 97-98% wt. inorganic carbon compared to total carbon. The intial TOC concentration of the natural humic substance was about 5-6 ppm. In addition, wastewater from the process line of an oil refinery plant was also collected for investigation.

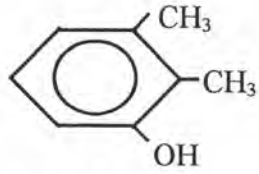
Activated carbon fiber-15,(-20) (ACFs-15, ACFs-20) , pitch- based ACF produced by Osaka Gas Co., were used as adsorbent. Its pore size distribution was analyzed using Belsorp-36 (Japan Bell Corp.) and its specific surface area was determined using the method of B.E.T. adsorption isotherm. As analytical instruments for concentration measurements , UV-visible spectrophotometer, TOC analyzer (Shimadzu Model TOC-500) and steam gas chromatography with FID detector

(column packing : Chromosorb PAW#60/80 , 3wt% phosphoric acid coating , Shimadzu) were used.

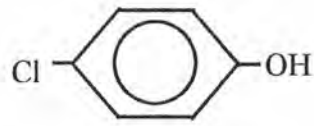
Table 3-1, 3-2 and **3-3** , respectively, list the properties of each chemical species, the type of analytical instrument used and the basic properties of ACF-15,20. The experimental operating conditions are listed in **Table 3-4**.

Table 3-1: Properties of Organic Chemical Species

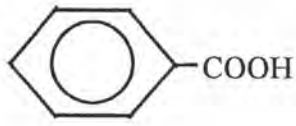
Species	**M.Wt.**	**Sp.Gr.[1]**	**Solubility in water[2]**
o-Xylenol(**XLN**)	122.17	-	-
p-Chlorophenol(**PCN**)	128.56	1.306 (20/4)	2.71 (20°C)
Benzoic acid (**BA**)	122.12	1.266 (15/4)	0.2 (17°C)
Cyclohexanol(**CHN**)	100.16	0.962 (20/4)	3.6 (20°C)



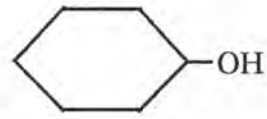
o-Xylenol



p-Chlorophenol



Benzoic acid



Cyclohexanol

Note : 1) 1.306 (20/4) : a density of 1.306 at 20 °C relative to water at 4 °C

2) Solubility : grams of substance in 100 gm of solvent

Table 3-2 : Analytical instrument used

Species	Analytical instrument	
	UV [1](nm.)	SGC [2](min.)
o-Xylenol	275	5.6
p-Chlorophenol	280	4.4
Benzoic acid	225	-
Cyclohexanol	-	3.2

Note : 1) The wavelength for maximum absorption of each species

2) Steam Gas Chromotography : Retention time in the packed column

Table 3-3 : Property of Activated Carbon Fiber-15,20

Note :- (**values** : ACFs-15 / ACFs-20)

Pore size distribution			*BET isotherm*		
S_t :	1831 / 1931	m^2/g	V_m :	402.8 / 425.2	ml./g
S_{ex} :	15.3 / 26.8	m^2/g	S_{BET} :	1753 / 1851	m^2/g
V_p :	0.806 / 0.994	ml./g.			
R_{peak} :	0.45 / 0.50	nm.			
d_p	15 / 20 μm. (7~20μm.)				

3.2 Methods

Adsorption equilibrium and isotherm measurement

The equilibrium adsorption isotherm for each organic aqueous solution was determined from the relationship between the amount of the adsorbent and the equilibrium concentration of the solution phase in each glass flask, as shown in **Fig. 3-1**. The adsorption isotherms were obtained by experiments at room temperature (25°C).

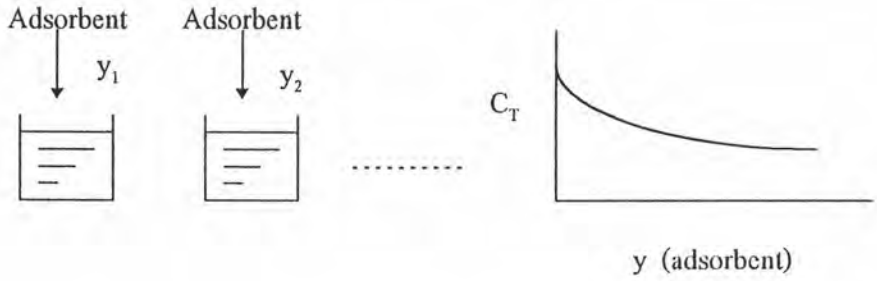


FIGURE 3-1 : INTEGRAL ADSORPTION EQUILIBRIUM CURVE

Prior to each experiment, the ACF was pretreated to remove the fine dust and chemical impurities by boiling 3-4 times in pure water and then dried overnight at 120°C in an electric oven.

Breakthrough curve (Column test)

The experimental apparatus for the fixed bed column test is shown schematically in **Fig. 3-2**.

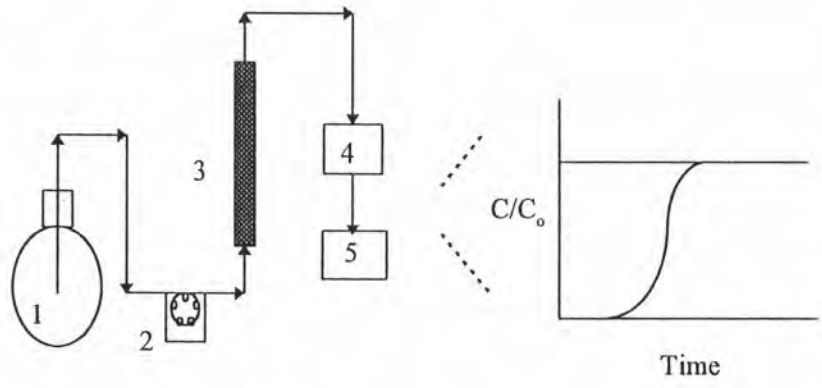


FIGURE 3-2 : COLUMN TEST APPARATUS : 1. Solution sample
2. Peristaltic pump 3. Fixed bed column of adsorbent
4. Fraction collector 5. Analytical instrument

Table 3-4 : The experimental operating conditions in this work

Type	Run No.	Solution	IAEC		Column Test					
			Initial Conc. (mg-C/l)	Temp. (°C)	Initial Conc. (mg-C/l)	Column dia. (cm.)	Bed length (cm.)	Temp. (°C)	*Superf. Vel. (m/hr.)*	*Packing dens. (gm/cm^3)*
Single	1	**XLN**	**11.11**	**25**	10.05	0.4	10	25	14.32	0.159
	2	XLN			10.05	0.4	10	25	4.77	0.159
	3	XLN			10.05	0.4	10	25	4.77	0.080
	4	XLN			10.05	0.4	5	25	4.77	0.159
	5	**PCN**	**11.06**	**25**	10.05	0.4	10	25	14.32	0.159
	6	PCN			10.05	0.4	10	25	4.77	0.159
	7	PCN			10.05	0.4	10	25	4.77	0.080
	8	PCN			10.05	0.4	5	25	4.77	0.159
	9	**BA**	**10.95**	**25**	10.50	0.4	10	25	14.32	0.159
	10	BA			10.50	0.4	10	25	4.77	0.159
	11	BA			10.50	0.4	10	25	4.77	0.080
	12	BA			10.50	0.4	5	25	4.77	0.159
	13	**CHN**	**11.06**	**25**	11.00	0.4	10	25	14.32	0.159
	14	CHN			11.00	0.4	10	25	4.77	0.159
	15	CHN			11.00	0.4	10	25	4.77	0.080
	16	CHN			11.00	0.4	5	25	4.77	0.159
Binary	17	**XLN +PCN**	**11.09**	**25**	11.67	0.4	10	25	14.32	0.159
	18	XLN+PCN			11.67	0.4	10	25	14.32	0.080
	19	**PCN + CHN**	**11.02**	**25**	10.30	0.4	10	25	14.32	0.159
	20	PCN +CHN			10.30	0.4	10	25	14.32	0.080
Tertiary	21	**XLN+PCN+BA**	**10.50**	**25**	10.02	0.4	10	25	14.32	0.159
	22	XLN+PCN+BA			10.02	0.4	10	25	14.32	0.080
Unknown	23	**Tapwater**	-	-	0.60	0.4	10	25	4.77	0.159
	24	**NHS**	**5.84**	**25**	5.80	0.4	5	25	4.77	0.159
	25	NHS			5.66	0.4	10	25	4.77	0.159
	26	NHS			5.54	0.4	10	25	14.32	0.159
	27	NHS			5.78	0.4	10	25	23.86	0.159
	28*	NHS	**6.02**	**25**	5.64	0.4	10	25	4.77	0.159
	29*	NHS			5.64	0.4	10	25	4.77	0.080
Industrial wastewater	30	**Oil Refinery**	**20.54**	**25**	21.85	0.4	5	25	14.32	0.170
	31	Oil Refinery			21.85	0.4	10	25	14.32	0.160
	32	Oil Refinery			21.85	0.4	10	25	14.32	0.312
	33	Oil Refinery			21.85	0.4	20	25	14.32	0.161

Note : Run No. 28,29 (*) used ACFs-20 as adsorbent, another one used ACFs-15