

Adsorption of *o*-Cresol from Its Aqueous Solution on Granular Activated Carbon Columns

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Compliance with stringent discharge limits and the most economic way of achieving it without loss of production has led to continued refinement of existing treatment technologies and the recognition and development of promising emergent technologies. Adsorption on granular activated carbon (GAC) has proved itself as an efficient technology for removal of final traces of a broad spectrum of toxic organic compounds from domestic and industrial wastewaters. In the present investigation, experiments have been carried out to determine the effect of various physico-chemical parameters associated with both adsorbent and adsorbate on the adsorption of *o*-cresol from aqueous phase. Some design parameters such as bed height, adsorbate flow rate, temperature, initial adsorbate concentration and adsorbate pH for efficient designing of fixed bed GAC columns has also been investigated. The commercially available bituminous coal based granular activated carbon Filtrasorb-300 (F 300) was used as adsorbent. The validity of the Michael's mass transfer zone (MTZ) concept for designing adsorption columns was used in this work.

INTRODUCTION

Industrial wastewaters including those from petrochemical, petroleum refining and coking plants contain appreciable amount of *o*-cresol which has been identified as a hazardous compound for many aquatic organisms by Environmental Protection Agencies¹. The Indian Minimum National Standard stipulated for wastewater discharge is not more than 1 ppm of phenols from refineries and pesticide industry; < 5 ppm into sewers followed by thermal treatment and < 1 ppm for discharge in the case of wool industry. The Indian Standard Specifications² for drinking water has set the maximum allowable limit of 0.001 ppm. Compliance with these stringent discharge limits and the most economic way of achieving this without loss of production has thus become a challenge to human ingenuity and responsibility. This has led to continued refinement of existing treatment technologies and the recognition and development of promising emergent technologies like activated carbon adsorption. The economic feasibility

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of GAC treatment has also been successfully evaluated in pilot and industrial plants³⁻⁵. In some of the developed countries inclusion of an adsorption step in physico-chemical wastewater treatment chain has been made mandatory by law⁶.

In the present investigation the effect of various physico-chemical parameters associated with adsorbent and adsorbate and certain column design parameters such as bed height, adsorbate flowrate, temperature, initial concentration of adsorbate and adsorbate pH on adsorption of *o*-cresol in fixed bed granular activated carbon columns has been evaluated.

EXPERIMENTAL

Adsorbent

The commercially available F-300 GAC (bituminous coal based) sample was used as adsorbent. It was sieved to get 12 × 16 mesh (B.S. standard) granules and was washed several times with distilled water until the leachate was free from any suspended impurities. The sample was dried in an oven at 100–110°C and stored in a calcium chloride desiccator until use. The physical properties of GAC are reported in Table-1.

TABLE-1
PHYSICO-CHEMICAL PROPERTIES OF FILTRASORB-300 GAC

N ₂ -BET* surface (m ² /g)	Particle* density (g/cm ³)	Pore* volume (cm ² /g)	wt(%)					
			Moisture content	Ash content	Volatile matter	Fixed carbon	Carbon	Hydrogen
970	0.730	0.850	5.3	5.4	1.7	87.6	97.63	0.18

*Information supplied by the manufacturer.

Adsorbate

o-Cresol (OC) was selected as an adsorbate in the present investigation as it is a priority pollutant¹. *o*-Cresol was procured commercially in its purest form available and its aqueous solutions of desired concentration were used for equilibrium and fixed bed adsorption studies. The physico-chemical properties of *o*-cresol are listed in Table-2. Adsorbate concentration in solutions was measured by UV spectroscopy.

TABLE-2
PHYSICO-CHEMICAL PROPERTIES OF ADSORBATES

Adsorbate	Abbreviation	m.w.	m.p. (°C)	b.p. (°C)	Solubility at 30°C (g/L)	λ _{max} (nm)	ε mol ⁻¹ cm ²
<i>o</i> -Cresol	(OC)	108.13	31	189	27.30	270	1540

Adsorption Equilibrium Studies

To evaluate the adsorption equilibrium isotherm, experiments were carried out

in a batch system. The experimental setup consisted of a thermostated round bottom flask and a motor impeller assembly, which enabled vigorous stirring of the solid-liquid system by a two-bladed teflon stirrer. Temperature of the system was maintained at $33 \pm 0.2^\circ\text{C}$. One litre of the adsorbate solution of known concentration was placed in a 2 L round bottom flask and an accurately weighed quantity of GAC was added into the flask. After the equilibrium was reached (about 5 h of stirring was required) the adsorbate concentration at equilibrium C_e was determined by UV absorption at 270 nm.

Fixed Bed Adsorption Studies

The experimental set-up for column studies consisted of a glass bottle of capacity about 5 L for feed solution, feed pump, thermostat, adsorption column and a sampler for collecting the effluent from the adsorbent column. The feed pump was a Swiss make Ismatec MC-MS-3 with flow rate range 0.005–75.2 mL/min. The adsorption column consisted of two co-centric glass tubes. The inner column served the purpose of a fixed bed adsorber while the outer glass column served the purpose of a jacket for circulating water from a constant temperature thermostat to maintain the adsorber unit at a constant temperature. The thermostat was provided with a contact thermometer and relay to maintain constant temperature in the bath.

The effluent from the fixed bed GAC adsorber was collected at desired intervals of time with the help of a fraction collector (212 Redirac fraction collector made in Sweden). These samples were later analysed on a UV spectrophotometer for the determination of adsorbate concentration. Silicon tubings were used for internal connections.

Procedure for Breakthrough Curve Determination

1. 4 L of adsorbate solution of desired concentration was prepared by diluting the stock solution and transferred to the feed bottle.
2. GAC sample was soaked in distilled water and the carbon slurry was poured into the column, avoiding air entrapment by lightly tapping the column.
3. The peristaltic pump was adjusted to obtain the desired flow rate of the influent stream flowing upwards.
4. The adsorbate solution was now introduced into the adsorbent column and the contact time of the feed solution in the column was determined.
5. The column effluent was collected at regular intervals using the fraction collector.
6. Collected samples were analysed on UV spectrophotometry.

RESULTS AND DISCUSSION

The adsorbent used in the present study was characterised with respect to proximate and ultimate analysis, surface area, pore size distribution, surface groups and pore structure by instrumentation techniques like N_2 -BET, scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). The SEM micrograph (Plate No. 1) of F-300 GAC sample shows a layered,

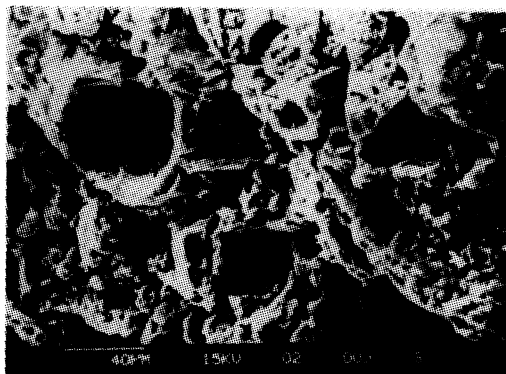


Plate 1. SEM Photograph of GAC Filtrasorb 300

loosely packed structure with a lot of cavities, cracks, irregular protrusions and widely dispersed pores⁷. The high mineral matter content may be causing the loose packing as can be seen from tiny particulate material in the micrographs.

The FTIR spectra of F-300 (Fig. 1) shows the presence of free phenolic —OH stretch vibrations around 3500 cm^{-1} . The bands around $3000\text{--}2900\text{ cm}^{-1}$ are probably due to the symmetric and asymmetric C—H vibrations. The asymmetric

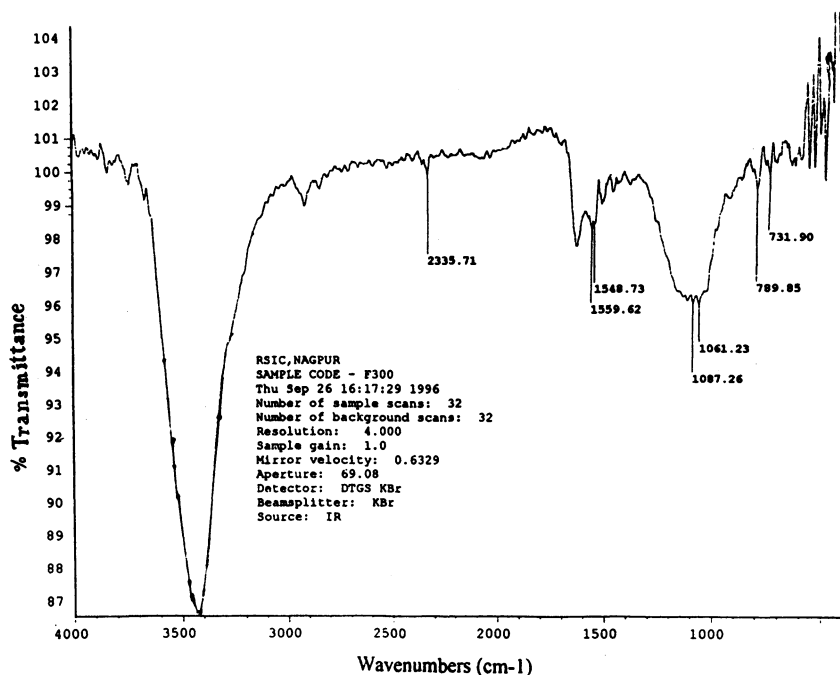


Fig. 1. FTIR Spectra of GAC Filtrasorb 300

O=C=O stretch vibration of CO₂ adsorbed in pores of adsorbent are represented by bands around 2336 cm⁻¹. Peaks around 1548 cm⁻¹ to 1622 cm⁻¹ which correspond to the vibrations of C=O in the quinone configurations are also present⁸. The peaks around 1087 cm⁻¹ probably indicate the moisture inclusions in between the layers as it has 5.3% moisture.

Adsorption equilibrium studies carried out in the present work for the *o*-cresol-F 300 system show typical type 1 favourable isotherm (Fig. 2) as generally referred in the literature. The fixed bed adsorption studies were carried out for the optimisation of various physico-chemical parameters associated with adsorbent, adsorbate and design parameters. Tables 3 to 7 give the variation in the characteristics of the mass transfer zone (MTZ) with depth of adsorbent flow rate, temperature, initial feed concentration and pH respectively. The values of Q_b, Q_c, φ, Z_a and U, reported in these tables were estimated using the relations proposed by Michaels⁹ and Hutchins¹⁰.

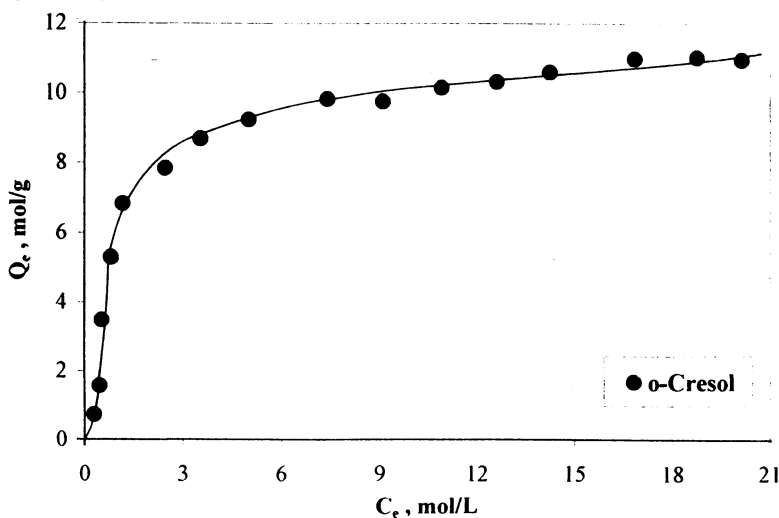


Fig. 2. Adsorption isotherm of *o*-cresol-F300 system.

Effect of Bed Height

Experimental breakthrough curves were determined at 10, 12 and 15 cm column heights keeping other parameters constant. These column heights correspond to adsorbate-adsorbent contact times 9, 11 and 14 minutes respectively. The breakthrough curves are shown in Fig. 3 and the obtained results are summarised in Table-3.

The height and rate of movement of mass transfer zone for bed height of 10 cm are higher than these values for 12 and 15 cm, whereas the fractional capacity of the bed for 10 cm height is smaller as compared to the longer bed heights 12 and 15 cm. The length and rate of movement of MTZ remains constant once a constant pattern is established, for which the adsorbent column height should be $\geq 2Z_a$.¹¹⁻¹³ Obviously the bed height of 10 cm is not enough for the constant

pattern of the MTZ to be established. For column heights of 12 and 15 cm, parameters Q_b , Q_t , Φ , Z_a , and U are nearly constant indicating that these parameters are independent of the adsorbent bed height once the adsorbent bed is long enough to allow the establishment of constant pattern of MTZ. The % S of the bed shows a slight increasing trend with the bed height. It justifies the Michael's concept⁷ that for uniform and stable MTZ zone the bed height should be $\geq 2Z_a$.

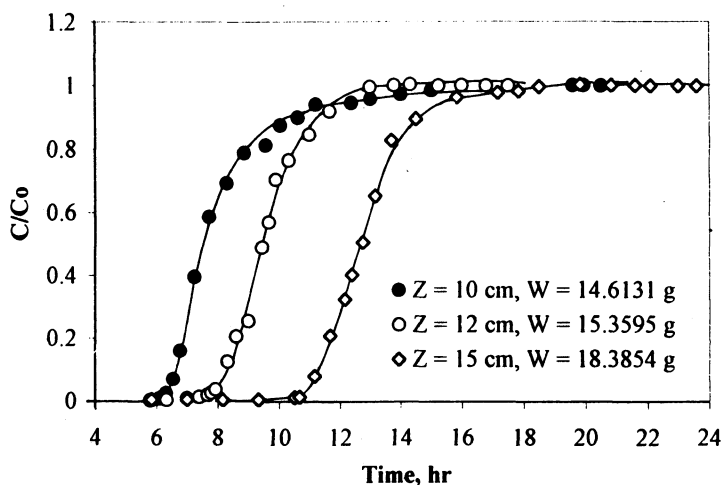


Fig. 3. Influence of bed height on breakthrough profile.

TABLE-3
EFFECT OF GAC BED HEIGHT ON BREAKTHROUGH PROFILE

Flow rate	1.7 mL/min	GAC particle size	12 × 16 mesh									
Feed concentration	0.036 mol/L	Temperature	33°C									
Bed diameter	1.8 cm											
No.	Z	W	ρ_b	CT	t_b	t_c	Q_b	Q_t	Φ	Z_a	U	S (%)
1.	10	14.6131	0.57	9.0	6.40	11.10	0.17	0.21	0.33	5.93	1.26	80.4
2.	12	15.3595	0.50	11.0	8.05	12.00	0.22	0.25	0.39	4.90	1.24	85.1
3.	15	18.3854	0.48	14.0	10.85	14.70	0.23	0.27	0.40	4.67	1.21	87.5

From the results it has been observed that the volume of effluent treated to the initial breakthrough is a direct function of the depth of adsorbent. This is obvious from Fig. 4a, which shows the plot of volume of the effluent treated to the 0.05 and 0.95 of the breakpoints *versus* the adsorbent column height. The linear relationship found can be expressed mathematically as:

$$V_{0.05} = (-263.72) + (91.129)Z \quad (2)$$

$$V_{0.95} = (361.56) + (74.889)Z \quad (3)$$

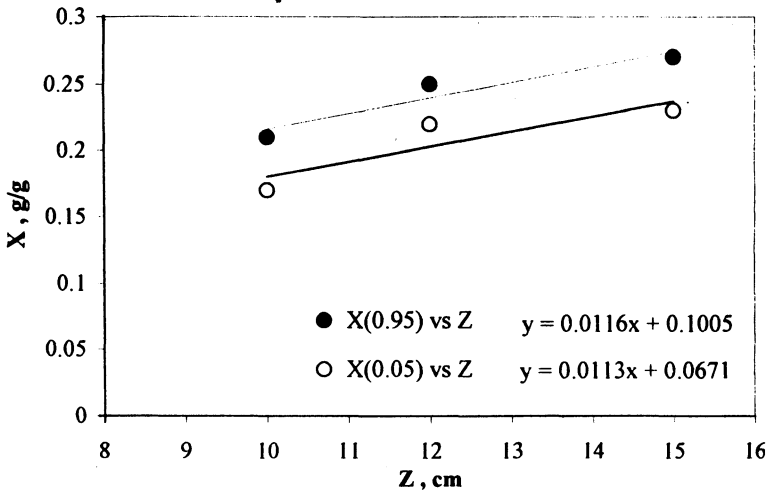


Fig. 4a. Influence of bed height on the adsorbate volume treated

where $V_{0.05}$ = vol. of adsorbate solution treated to 0.05 breakthrough point, mL

$V_{0.95}$ = vol. of adsorbate solution treated to 0.95 breakthrough point, mL

The actual amount of *o*-cresol adsorbed at each depth of adsorbent was determined and is summarised in Table-3 and shown graphically in Fig. 4b. As expected, the total amount of adsorbate removed increases with increasing depth of adsorbent. The linear equations describing the relationships presented in Fig. 4 are:

$$X_{0.05} = (0.0671) + (0.0113)Z \quad (4)$$

$$X_{0.95} = (0.1005) + (0.0116)Z \quad (5)$$

where $X_{0.05}$ = the amount of adsorption up to the 0.05 breakthrough point, in g/g;

$X_{0.95}$ = the amount of adsorption up to the 0.95 breakthrough point in g/g;

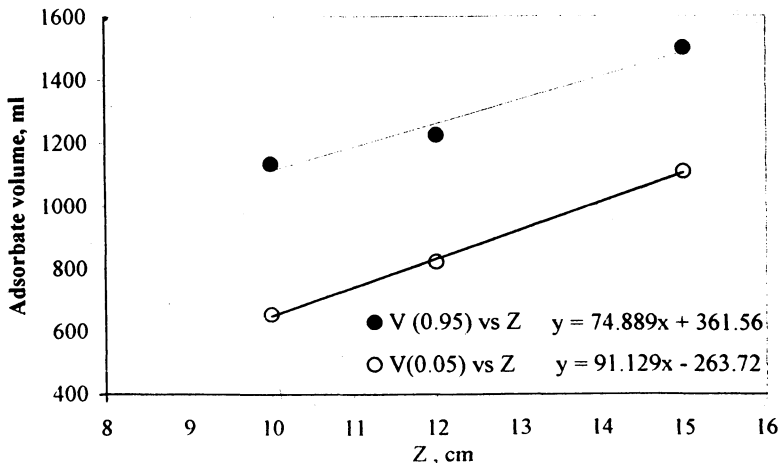


Fig. 4b. Influence of bed height on the amount of adsorbate treated

The linearity of the plot in Fig. 4b had the linear regression coefficient value of 0.95. This may be due to insufficient bed height, 10 cm, for the formation of stable mass transfer zone and its uniform movement through the adsorbent column.

Effect of Temperature

In the present investigation adsorption breakthrough curves were obtained at different temperatures for OC-F300 system and the breakthrough curves are shown in Fig. 5 and the results are summarised in Table-4.

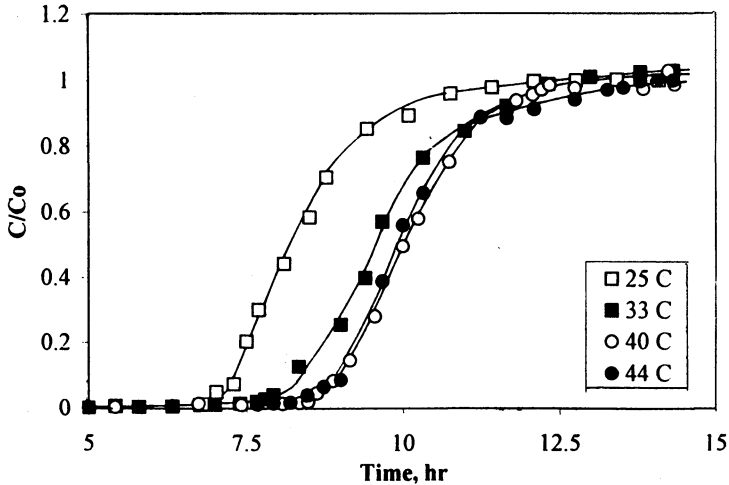


Fig. 5. Influence of temperature on breakthrough profile

TABLE-4
EFFECT OF TEMPERATURE ON BREAKTHROUGH PROFILE

Flow rate	1.7 mL/min	GAC particle size	12 × 16 mesh									
Feed concentration	0.036 mol/L	Bed diameter	1.8 cm									
Bed height	12 cm											
No.	T(°C)	W	ρ_b	CT	t_b	t_c	Q_b	Q_t	Φ	Z_a	U	% S
1.	25	15.3596	0.50	11.0	7.10	11.51	0.20	0.26	0.40	5.97	1.35	80.1
2.	33	15.3594	0.50	11.0	8.00	12.00	0.21	0.25	0.39	4.90	1.23	84.1
3.	40	15.3595	0.50	11.0	8.69	11.83	0.22	0.26	0.47	3.71	1.18	85.4
4.	45	15.3595	0.50	11.0	8.70	12.40	0.22	0.26	0.39	4.58	1.18	85.1

The equilibrium adsorption capacity of phenols on GAC is reported to decrease at higher temperatures but the adsorption and desorption rates increase as the temperature increases¹⁴. Thus, it is evident that the kinetics of the adsorption is an endothermic process, while the equilibrium position of adsorption is an exothermic process. That is, as the temperature of the system increases the rate of removal of adsorbate increases but adsorption capacity decreases. However, the effect of temperature was not very significant. Snoeyink¹⁵ noted that

temperature exhibits more influence on the adsorption process at lower equilibrium concentrations and that the temperature effects can generally be neglected for actual design work over a narrow temperature range.

These results show that the MTZ height decreases slightly as the temperature increases from 25 to 40°C. The decrease of MTZ height with increase in temperature could be attributed to the effect of temperature on bulk diffusion in solution, liquid viscosity and the intraparticle diffusivity. The bulk diffusivity is directly proportional to temperature and is inversely proportional to the viscosity of liquid; thus a higher temperature favours the mass transfer of solute from solvent to adsorbent phase thereby reducing the MTZ height which is a measure of resistance to mass transfer. The slight increase in MTZ height at 45°C may be due to experimental error in concentration measurements as at higher temperature evaporation losses of solute and solvent will be more.

Effect of flow rate

Adsorbate flow rate is an important parameter which influences the overall performance of the adsorbent column as the contact time between the adsorbate and adsorbent gets affected and in turn affects the mass transfer parameters. In this investigation adsorption breakthrough curves were obtained at different adsorbate flow rates for OC-F300 system. The breakthrough curves are shown in Fig. 6 and the results are given in Table-5.

These results show an inverse relationship between the adsorbate flow rate and the initial breakthrough time as well as the bed exhaustion time. The breakthrough curves become flatter at higher flow rates^{11, 12}. Thus, the MTZ height increases with the increase in adsorbate flow rate or decrease in the contact time. This would mean that the adsorbate in the fluid stream travels faster than it is transferred into the adsorbent particle and increasing the flow would mean increasing the adsorbate volume between initial breakthrough and saturation.

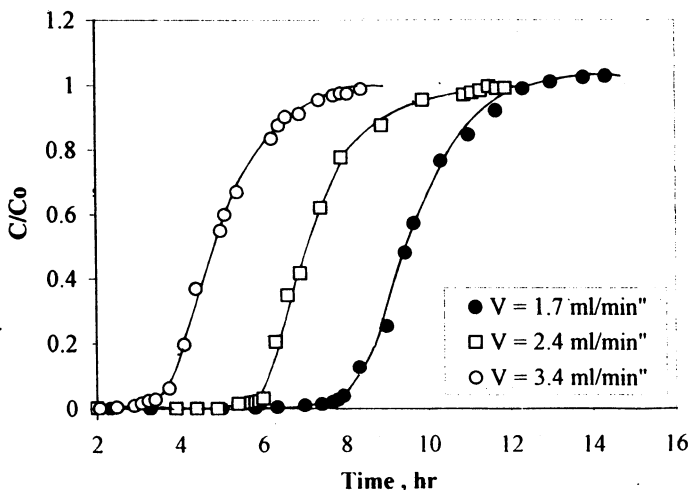


Fig. 6. Influence of flow rate on breakthrough profile.

TABLE-5
EFFECT OF ADSORBATE FLOW RATE ON BREAKTHROUGH PROFILE

GAC particle size	12 × 16 mesh	Temperature	33°C									
Feed concentration	0.036 mol/L	Bed diameter	1.8 cm									
Bed height	12 cm											
No.	V	W	ρ_b	CT	t_b	t_c	Q_b	Q_t	Φ	Z_a	U	S(%)
1.	1.7	15.3596	0.50	11.0	8.05	12.01	0.21	0.25	0.39	4.90	1.24	84.1
2.	2.4	15.3594	0.50	8.0	6.10	9.70	0.22	0.26	0.38	5.84	1.64	83.8
3.	3.4	15.3595	0.50	5.0	3.65	7.27	0.19	0.26	0.39	8.60	1.38	72.2

Effect of initial adsorbate concentration on the breakthrough profile

To study the effect of variation of feed concentration, breakthrough experiments were carried out for F300-OC system, in which initial concentration of the adsorbate was the only variable parameter. The initial concentrations of *o*-cresol taken were 0.020, 0.027, 0.036 and 0.047 mol/L. The breakthrough curves obtained for these feed concentrations are presented in Fig. 7 and the breakthrough data is summarised in Table-6.

The increase in initial concentration of the adsorbate leads to an early breakthrough and exhaustion of the bed. The mass transfer zone height decreases while rate of movement of MTZ increases with the increase of feed concentration. This is because of the increase in mass flow rate of adsorbate applied to the columns. However, the adsorption capacity at break point and exhaustion point does not change. Thus the adsorbent capacity is achieved earlier at higher adsorbate concentrations indicating that the fluid to adsorbent mass transfer rate of the solute increases. This is expected since the concentration gradient across the fluid film surrounding the adsorbent particle will be higher at higher concentration of the adsorbate.

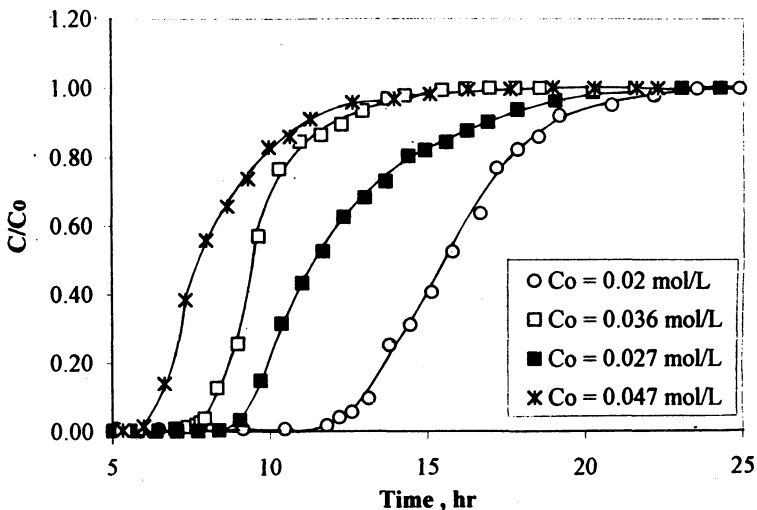


Fig. 7. Influence of concentration on breakthrough profile

TABLE-6
EFFECT OF ADSORBATE CONCENTRATION ON BREAKTHROUGH PROFILE

GAC particle size 12 × 16 mesh				Temperature		33°C					
Bed diameter		1.8 cm		V		1.7 mL/min					
Bed height		12 cm		W		15.3596 g					
No.	C ₀	ρ _b	CT	t _b	t _c	Q _b	Q _t	Φ	Z _a	U	S(%)
1.	0.020	0.50	11.0	13.56	22.69	0.20	0.26	0.48	6.10	0.67	75.6
2.	0.027	0.50	11.0	10.77	16.78	0.21	0.26	0.45	5.36	0.89	79.9
3.	0.036	0.50	11.0	8.05	12.00	0.21	0.25	0.39	4.90	1.24	84.1
4.	0.047	0.50	11.0	6.32	9.16	0.21	0.25	0.37	4.62	1.63	85.8

Effect of hydronium ion concentration

Column studies were conducted with *o*-cresol as adsorbate at pH values 5.23, 7.20 and 8.38 to investigate the influence of pH on breakthrough profile. The adsorbate pH was adjusted to required value by adding the phosphate buffer. The breakthrough curves for these experiments are shown in Fig. 8, while a summary of the characteristics of the curves is presented in Table-7.

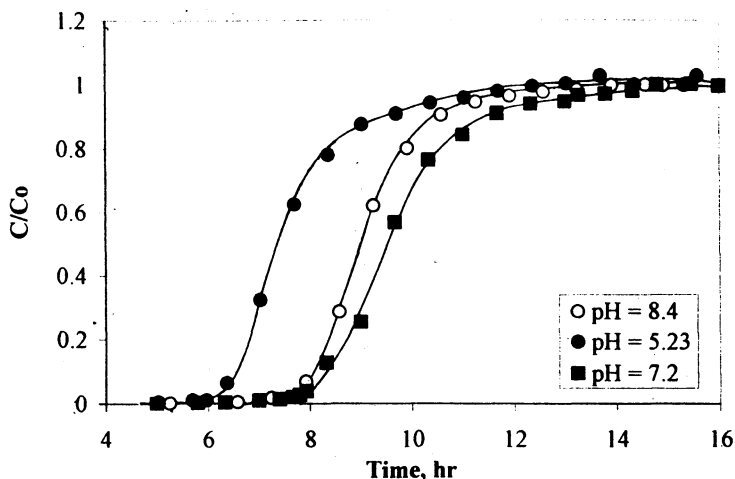


Fig. 8. Influence of adsorbate pH on breakthrough profile

TABLE-7
EFFECT OF HYDRONIUM ION CONCENTRATION ON BREAKTHROUGH PROFILE

GAC particle size		12 × 16 mesh		Temperature		33°C						
Feed concentration		0.036 mol/L		Bed diameter		1.8 cm						
Bed height		12 cm		Flow rate		1.7 mL/min						
No.	pH	W	ρ _b	CT	t _b	t _c	Q _b	Q _t	φ	Z _a	U	S(%)
1.	5.23	15.3596	0.50	11.0	6.30	10.70	0.16	0.20	0.33	6.81	1.55	81.3
2.	7.20	15.3594	0.50	11.0	8.05	12.00	0.21	0.25	0.39	4.90	1.21	84.8
3.	8.38	15.3595	0.50	11.0	7.80	11.40	0.20	0.26	0.52	4.46	1.24	80.7

These results show that the MTZ height increases as the pH decreases. The initial breakthrough times for pH = 7.20 and 8.38 are closer to each other but at lower pH of 5.23, the initial breakthrough time is about 22% lower. The adsorption capacity was observed to increase as the pH of the adsorbate solution increased from 5.23 to 8.40. It supports the studies carried out by Faust *et al.*¹⁶ that the adsorption capacity for an adsorbate increases up to the pH equal to its (pKa-2).

The effect of the pH on adsorption from the solution must be considered to result from the combined effects of pH on the nature of the adsorbate molecule, the adsorbent surface and the inorganic acids, bases and salts used to prepare the buffer for adjusting the pH. The effect of pH may alter the chemical nature of the adsorbent surface. The hydronium ion concentration affects the associated or dissociated state of the adsorbate which in turn will affect the extent of adsorption. Phenols being acidic in nature are likely to get dissociated at higher pH above their pKa value forming ionic species which are adsorbed to lesser extent by the covalent activated carbon surface. The pKa value¹⁵ of *o*-cresol is 10.2. The weak acidic functional groups present on the adsorbent surface are also affected, in turn influencing the adsorption to a lesser degree than the adsorbate.

Conclusions

The present work has led to some important conclusions as stated below:

1. Pore size distribution, scanning electron microscopy, Fourier transform infrared spectroscopy and proximate and ultimate analysis show that the source material used for preparation of GAC has significant effect on the adsorbent pore structure, surface characteristics, resistance to fragmentation and adsorption capacity.
2. FTIR spectra show presence of various carbon-oxygen complexes on the granular activated carbon surface which makes the surface slightly polar.
3. The mass transfer zone height and the rate of movement of mass transfer zone along with the other parameters Q_b , Q_t , and ϕ are independent of adsorbent column height provided the column is long enough for the formation of a stable mass transfer zone, whereas the mass transfer zone height increases with the increase in adsorbate flow rate or decrease in the contact time. At smaller bed contact time the % saturation of the GAC column also decreases.
4. The decrease of MTZ height with increase of temperature indicates that rate of adsorption is an endothermic process. The adsorption capacity of the adsorbent increases with the decrease of pH in the range below the pKa value of the adsorbate.
5. The increase in initial concentration of the adsorbate leads to an early breakthrough and exhaustion of the bed. The mass transfer zone height decreases while rate of movement of MTZ and % saturation of the GAC increases with the increase of feed concentration but the break point and the exhaustion point adsorption capacity Q_b and Q_t do not change significantly.

Nomenclature

- C = concentration of adsorbate in liquid phase, mol/L
- C_0 = initial concentration of adsorbate, mol/L
- C_e = equilibrium concentration of adsorbate, mol/L
- ϕ = fractional capacity of the bed
- Q^0 = monolayer capacity of the adsorbent, mol/g
- Q_b = amount adsorbed on adsorbent, mol/g
- Q_e = amount adsorbed at equilibrium, mol/g
- %S = per cent saturation of bed
- t_b = breakthrough time, min (breakthrough concentration is taken as 5% of C_0)
- t_e = bed exhaustion time, min (bed exhaustion concentration is taken as 95% of C_0)
- U = rate of movement of mass transfer zone, cm/min
- V = flow rate, mL/min
- $V_{0.05}$ = volume of adsorbate treated upto t_b , mL
- $V_{0.95}$ = volume of adsorbate treated upto t_e , mL
- Z = Adsorbent bed height, cm
- Z_a = height of mass transfer zone, cm
- W = weight of the adsorbent, g
- CT = adsorbate contact time in the bed, min
- ρ_b = adsorbent bed density, g/cm³

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