

The dynamic adsorption characteristics of phenol by granular activated carbon

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Abstract

The objective of the present work is to determine the operating conditions of an activated carbon filter, based on the characteristics of breakthrough curves. For this we apply the technical developed by Mickaels [1] for the ionic exchange and applied by Luchkis [2] for the adsorption, and which is the mass transfer zone. To reach our goal, an evaluation of the operating conditions (height of the bed, flow and concentration of effluent) on the characteristics of the mass transfer zone was made and an explanation of the mechanism of adsorption was given. Thereafter a modeling of the experimental results was done.

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1. Introduction

Water pollution is a very persistent problem in fact; the intensive throwing up of different toxic substances without control constitutes a real danger for humanity. Each year we assist to the total or partial disappearance of many creations and, or plants species leading to an ecological disaster. Phenolic compounds are common contaminants in wastewater, generated by petroleum and petrochemical, coal conversion, and phenol producing industries. Phenols are widely used for the commercial production of a wide variety of resins including phenolic resins, which are used as a constructions material for automobiles and appliances, epoxy resins and adhesives, and polyamides for various applications [3].

The phenols are considered as priority pollutants since they are harmful to organisms at low concentrations and many of them have been classified as hazardous pollutants because of their potential harm to human health. Stringent US Environmental Protection Agency (EPA) regulations call for a lowering phenol content in wastewater to less than 1 mg/l [4].

A complete removal or in some cases reduction of these persistent organic compounds to an acceptable concentration,

has become a major objective of advanced water and wastewater treatment before being discharged. The efficiency of various treatment processes in removing refractory organic was been largely documented: precipitation/coagulation, chemical oxidation, sedimentation, filtration, adsorption, osmosis, ion-exchange, ultrafiltration, electrodialysis, electrochemical degradation, flotation [5].

New treatment technologies are constantly searched and developed. This research includes chemical oxidation [6–9], solvent extraction [10] biological degradation [11–13]. Although many different adsorbents [14] were tried to remove phenols from wastewater, activated carbon [15,16] was still the most widely used adsorbent to this end.

The adsorption by activated carbon has emerged as the most efficient and the most economical process for removing undesirable organic materials from aqueous solution [17–21].

The concept of an organic compound adsorption on activated carbon is not new, and since the beginning of the century, filters were used to remedy to the objectionable taste and odour of the water. At present, activated carbon systems are in use throughout the world in water treatment. Activated carbons are among the adsorbents, having a high specific surface area; they exhibit a strong adsorption capacity for phenolic compounds. Their systematic use in wastewater treatment is the result of several factors: (a) creation of an efficient technology of carbon activation, (b) decrease in prices of water treatment by reusing

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Nomenclature

A_{Bp}/m	amount of solute eliminated by the activated carbon at breakpoint (mg/g)
A_Z, A_{Bp}	the amount of really eliminated by the filter inside the mass transfer zone and at breakpoint, respectively (mg)
C_{Bp}	(5% of initial concentration), instantaneous concentration of the effluent at the breakpoint (mg/l)
C_E	(95% of initial concentration), instantaneous concentration of the effluent at the exhaustion concentration (mg/l)
C_i	instantaneous concentration of the effluent (mg/l)
C_0	initial concentration of the effluent (mg/l)
F	fractional capacity
H_b	height oh the bed (cm)
H_Z	height of mass transfer zone (cm)
m	mass of granular activated carbon (g)
Q	volumetric flow (l/h)
t	operating time (h)
U_Z	rate of movement of MTZ (cm h^{-1})
V_{Bp}	volume treated at breakthrough point (l)
V_E	volume treated at the exhaustion concentration (l)

carbon after its regeneration and (c) the use of carbonaceous wastes material for the manufacture of activated carbon.

As such, attempts have been made by different workers to develop alternative adsorbents, preferably of low cost, materials such as fly ash corncob wastes; spent bleaching earth, apricot stone shells, rubber seed coat, waste tire rubber, etc. have been utilized for this purpose [22–25].

These new adsorbents prepared from carbonaceous materials have been evaluated and compared to standard activated charcoal. The results indicate that the phenols removal efficiency of carbonaceous adsorbent is about 45%. Consequently they can be used for the removal of phenols as a low-cost alternative (US\$ 0.1/kg) [26].

The aim of this work is to study the influence of the various operating parameters on the removal performances of the filter, to understand the adsorption mechanisms, and thereafter treat

the experimental data mathematically, in order to determine the relations governing a filter use.

Phenol has been selected: (a) they are the main responsible agents of the objectionable taste of water and its nauseous odor, (b) they are considered to be very toxic compounds and supposed carcinogenic and (c) In addition, in nature, their adverse effects have also been found on some useful bacteria (nitrification bacteria, bacteria in sediment, etc.).

2. Theoretical

Normally the adsorption isotherms are used for the preliminary screening of an adsorbent before running more costly tests. The procedures are now known and give indications on effectiveness of adsorption for removing specific impurities as well as the maximum quantity that can be adsorbed by a particular unit. However the adsorption isotherms are unable to give us information concerning filter behavior because adsorption in carbon column is not at equilibrium and granular carbon rarely becomes totally exhausted in commercial processes [27].

Therefore practical experiments have been investigated to obtain a factual model. In order to properly design and operate fixed-bed adsorption processes. We apply the concept of the mass transfer zone (MTZ) that helps to obtain the evolutions of the operating parameters of the fixed-bed. This concept was developed by Mickaels [1] for the ionic exchange and applied by Luchkis [2] for the adsorption.

The MTZ is the portion of the filter where major adsorption is occurring, where adsorbate is actively removing from the liquid phase at any given moment. Three characteristics determine the mass transfer zone, noticing that these characteristics can be calculated in referring to either the solid or the liquid phases.

2.1. Fractional capacity (F)

It determines the elimination efficiency of the granular activated carbon. Based on the solid phase, F is defined by the ratio of the quantity of carbon who really participates in transfer process compared to the total quantity of carbon used by the height of the mass transfer zone. It may be defined also by the ratio of the real quantity eliminated of solute on the potential capacity of elimination of carbon within the mass transfer zone.

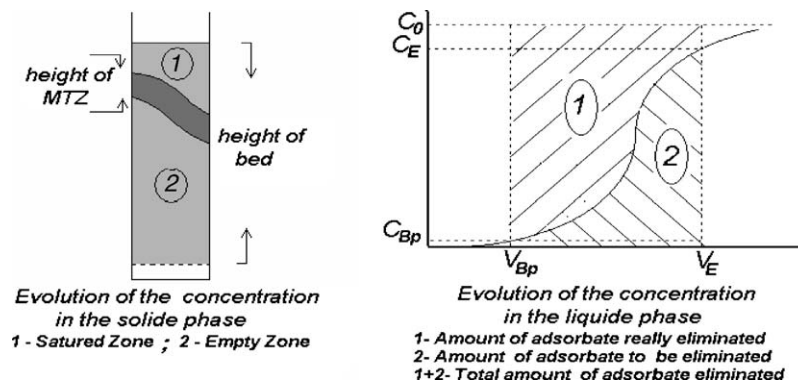


Fig. 1. Evolution of the concentration in both phases.

The fractional capacity may be calculated graphically from Fig. 1.

- The maximum amount of adsorbate which can be eliminated by the filter inside the mass transfer zone is:

$$A_{\max} = C_0(V_E - V_{Bp}) \quad (1)$$

- V_E corresponds at the volume of effluent treated whose the instantaneous concentration C_E at the exhaustion time t_E is equal or more than 80% of the initial concentration C_0 . This concentration is chosen arbitrarily depending on the working mode of the filter, from economic point of view.
- V_{Bp} corresponds at the volume of effluent treated whose the instantaneous concentration C_{Bp} at the breakthrough time t_{Bp} is less than 5% of the initial concentration.

The amount of adsorbate really eliminated by the filter inside the mass transfer zone is:

$$A_Z = \int_{V_{Bp}}^{V_E} (C_0 - C_i) dV \quad (2)$$

The fractional capacity is giving by the ratio:

$$F = \frac{A_Z}{A_{\max}} = \frac{\int_{V_{Bp}}^{V_E} (C_0 - C_i) dV}{C_0(V_E - V_{Bp})} \quad (3)$$

2.2. The height of MTZ (H_Z)

It is the area where practically takes place all the phenomenon of adsorption. It determines the rate of elimination of the adsorbate by the adsorbent, and gives indications about the diffusion resistances.

Weaker is the resistance of transfer, faster is the kinetic of adsorption and smaller is the depth of the zone of mass transfer. Therefore, it is an effective parameter for quantifying the overall kinetics of the adsorption process.

The height of the mass transfer zone, H_Z is calculated as follows:

$$H_Z = U_Z \cdot t_Z \quad (4)$$

U_Z is the rate of movement of MTZ

$$t_Z = t_E - t_{Bp} \quad (5)$$

The time required for the movement of the mass transfer zone down its own length in the column, t_Z is determined experimentally, and the only unknown is U_Z .

On the other hand, the time necessary for the mass transfer zone H_Z to move the bed is:

$$t_E - t_f = \frac{H_b}{U} \quad (6)$$

The adsorption not being an instantaneous phenomenon, t_f is the time required for the formation of the adsorption zone. Like the rate of the movement of the zone of mass transfer is constant,

consequently:

$$U_Z = \frac{H_Z}{t_Z} = U = \frac{H_b}{t_E - t_f} \quad (7)$$

So the only unknown is the formation time of the mass transfer zone, which may be estimated, based on the solid phase:

- If $F = 1$, So that the adsorbent is entirely saturated, the formation time (t_f) of the zone at the top of the bed should be substantially the same as the time t_Z , time required for the zone to move a distance equal to its own depth.
- On the other hand, if $F = 0$, so that the adsorbent contains no adsorbate, the zone-formation time should be very short, at the limit equal to 0. These limiting conditions are described by one relation:

$$t_f = (1 - F)t_Z = (1 - F)(t_E - t_{Bp}) \quad (8)$$

and

$$H_Z = \frac{H_b t_Z}{t_E - t_f} = \frac{H_b(t_E - t_{Bp})}{t_{Bp} + F(t_E - t_{Bp})} \quad (9)$$

Finally knowing that $t = V/Q \Rightarrow$

$$H_Z = \frac{H_b(V_E - V_{Bp})}{V_{Bp} + F(V_E - V_{Bp})} \quad (10)$$

2.3. The rate of the movement of the MTZ (U_Z)

The rate of the movement of the MTZ is primarily a function of adsorption capacity of the adsorbent, and thus, is able to determine the equilibrium nature of the adsorption process under continuous flow conditions. It permits the calculation of rate of bed saturation. It is directly related to the height of mass transfer zone. Small is the depth of MTZ, more quickly is the rate of transfer, and rapid is the saturation of the bed.

The rate of the movement of the mass transfer zone is given:

$$U_Z = \frac{H_Z}{t_Z} = \frac{H_b \cdot Q}{V_{Bp} + F(V_E - V_{Bp})} \quad (11)$$

The calculation of F , H_Z and U_Z will permit us to understand and to explain how the system is evolving. The different variables depend implicitly and/or explicitly upon these three parameters that manage whole adsorption process.

If we look for only the efficiency of the column, we can calculate the number of unit transfer equivalent, NUT, like in absorption by:

$$N_Z = \frac{H_b}{H_Z} = \frac{H_b[V_{Bp} + F(V_E - V_{Bp})]}{H_b(V_E - V_{Bp})} = \frac{V_{Bp} + F(V_E - V_{Bp})}{V_E - V_{Bp}} \quad (12)$$

Fast is the adsorption mechanism, small is the height of MTZ and great is the number of transfer unit. But in the dynamic adsorption, having a high NUT is not interesting, because fast is the exhaustion of the bed.

2.4. The amount of pollutant eliminated at breakthrough point

The amount of pollutant eliminated by the bed of activated carbon at the breakthrough point can be calculated graphically:

$$A_{Bp} = \int_0^{V_{Bp}} (C_0 - C_i)dV \tag{13}$$

And by the activated carbon:

$$\frac{A_{Bp}}{m} = \int_0^{V_{Bp}} \frac{C_0 - C_i}{m} dV \tag{14}$$

3. Materials and methods

To characterize the adsorption ability of the activated carbon in the wastewater treatment, dynamic adsorption tests were carried out. Activated carbon used in this study is commercially available but its physical and chemical properties were unknown, therefore a characterization was done.

To eliminate impurities, the activated carbon is washed by hot distillate water and then was dried at 105 °C for 48 h. Finally the activated carbon was sieved to a uniform size (30 μm) and stored in closed bottles. The phenol stocks was prepared and suitable diluted to the required initial concentration. The concentration was obtained by measuring the absorbency at wavelength maximal, using a UV–vis spectrophotometer Shimadzu mini 1240.

The columns used in the experiments were Pyrex tube of 10 mm inner diameter and 50 cm in length.

The rate of flow was regulated by a peristaltic pump (Gilson’s Minipuls 3). The Concentration of solution passing through the columns was monitored continually by collecting manually the samples in volumetric flasks. Measurement of the concentration of adsorbate was made by direct ultra-violet absorbency. Before analyzing, a centrifugation of the sample was done at 10,000 rpm during 10 min. A preliminary scan was conducted to determine the wavelength of maximum absorbency (λ_{max} = 271 nm).

The rate of influent was changed from 0.30 to 0.80 l/h while the bed depth was varied from 5.7 to 17.5 cm and the concentration from 50 to 300 mg/l.

4. Results and discussion

The main characteristics of activated carbon used in this study were determined and resumed in Table 1

The evolutions of the concentration of effluent with the time are displayed in Figs. 2 and 3, respectively.

Table 1
Main characteristics of granular activated carbon

Real density	1.48
Apparent density	0.73
Pore volume	1.29 cm ⁻³ g ⁻¹
Specific surface	950 m ² g ⁻¹
Grain size	30 mm
Porosity	0.64
Surface function	pH = 5.70

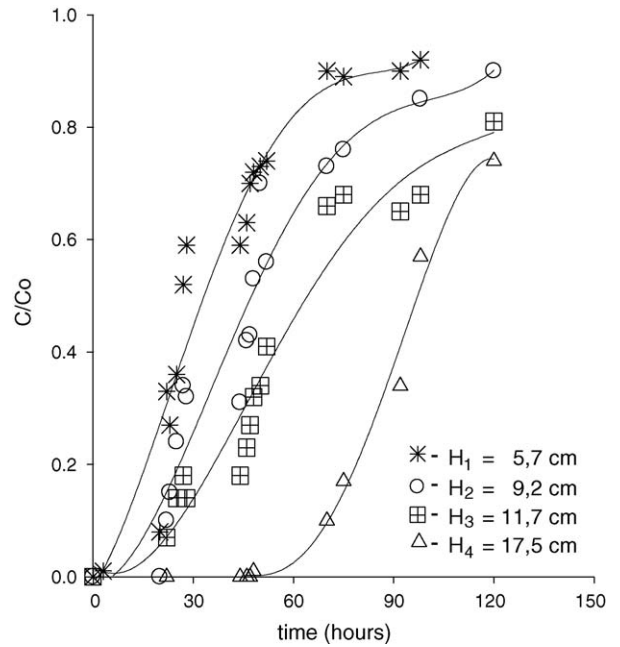


Fig. 2. Breakthrough curves for several depths of bed, Q = 0.30 l/h; C₀ = 50 mg/l.

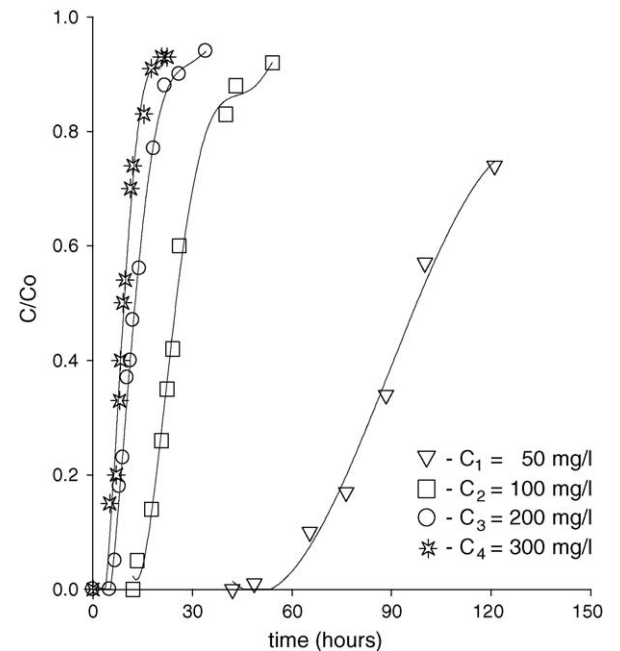


Fig. 3. Breakthrough curves for several Initial concentrations; Q = 0.30 l/h; H = 11.7 cm.

As we can notice it, the breakthrough times increase with the bed depth, and decrease with the influent concentration. This supposes that the treated volumes are changing in the same way, but in reality they are varying unexpectedly.

The breakthrough curves obtained, the characteristics *F*, *H_Z*, *U_Z*, and their evolutions are determined as function of the rate of flow and of the bed depth.

These three parameters enable us to estimate the behavior of the filter, to understand how the process of adsorption is done and how to act for an improvement.

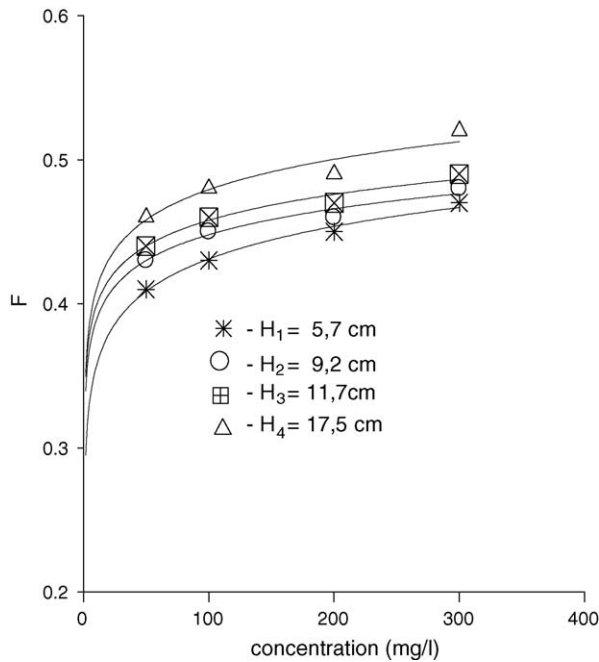


Fig. 4. Variation of fractional capacity of MTZ with the initial concentration for several bed depths.

In order to determine filter efficiency, we will calculate specific parameters (quantities of adsorbate eliminated or volumes of effluent treated . . .) and establish their variations with operating conditions.

4.1. Fractional capacity of the bed (within the mass transfer zone) F (Fig. 4)

MTZ established the changes with the hydrodynamic and the physicochemical characteristics of the system remain insignificant. A variation in the operating conditions does not affect (or slightly) the fractional capacity, that we can say than F is a characteristic of studied system.

This work is confirmed by that carried out by Gupta et al. [27] for substituted phenols. He finds that the fractional capacity is of same order of magnitude and is specific to each studied system.

4.2. The height of mass transfer zone: H_Z (Fig. 5)

We can study the height of the MTZ as well as the number of unit of transfer. It is one of the most important parameters in adsorption column since it determines the elimination efficiency of the adsorbent and the exchange rate between phases. The faster the exchange is, small is the MTZ height, and more important is the number of transfer unit developed. An increase of the feed flow and/or the concentration (more molecules of solute), is leading to an increase in the residence time of molecules in the column, due to the steric effect or/and in the difficult choice of an adsorption site. For remedying this state, the mass transfer zone increases its Height and offers more sites to the molecules, i.e. it compensates the resistance to the transfer by increasing

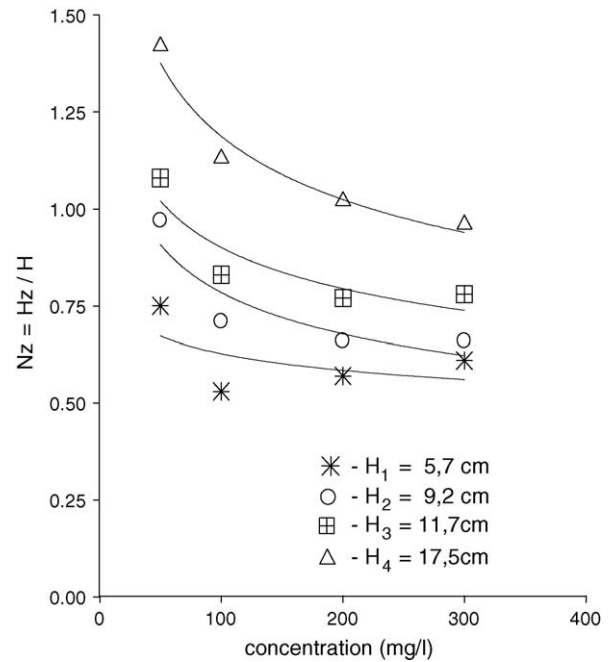


Fig. 5. Variation of N_Z with the initial concentration for several bed depths.

his depth and in both cases we have a reduction in rate of mass transfer between the both phases.

On the contrary, the fact of increasing the bed depth gives a greater number of adsorption sites for an equivalent number of molecules, therefore an easier choice of the site, as a result, an acceleration of the process. Consequently increasing the bed depth improves the adsorption kinetic.

In comparison, the adsorption of phenol onto the activated carbon fibers, Brasquet and Le Cloirec [28] found that H_Z does not depend on the volumetric flow. But on the other hand the eliminated quantity decreases with the flow i.e. a loss of efficiency.

4.3. The rate of movement of the mass transfer zone U_Z (Fig. 6)

Another characteristic of breakthrough curve as important as the mass transfer zone height is its rate of the movement which determines the working time of the filter. When increasing the height of the bed, hydrodynamic conditions remain unchanged, but the specific characteristics of the MTZ changes (H_Z increases). The distance to be traversed by the MTZ decreases its rate of travel increases. Of same the increasing the volumetric flow, or/and the concentration of effluent, increase particularly the height of MTZ and consequently, her rate of movement, the result, the granular activated carbon bed is rapidly exhausting (Figs. 7 and 8).

As it is noted the quantities eliminated depend on the characteristics from the MTZ, especially from the number of transfer unit (N_Z). In other words the height (H_Z) is an explicit and an implicit function of the operating conditions. The eliminated quantities decrease with the flow and the concentration of the

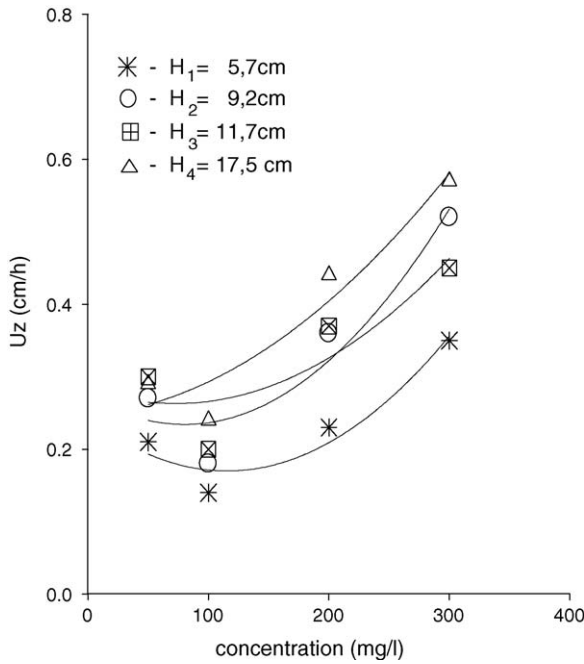


Fig. 6. Variation of U_Z with the initial concentration for several bed depths.

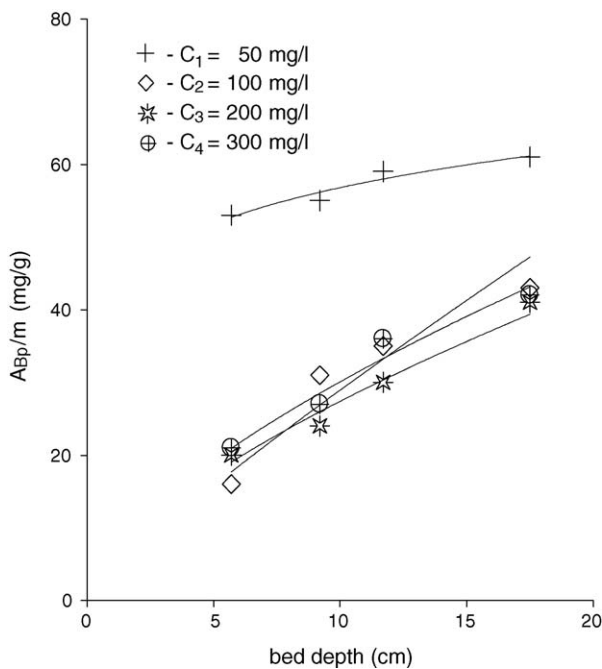


Fig. 7. Variation of (A_{Bp}/m) with bed depth for several initial concentrations.

influent and increase with the height of the bed for the same reasons quoted previously.

The behavior of these characteristics are resumed in Table 2.

5. Mathematical exploitation

For predicting the working of fixed bed, many parameters are needed, some are determined by an independent batch kinetic study whereas others are by suitable correlations. There

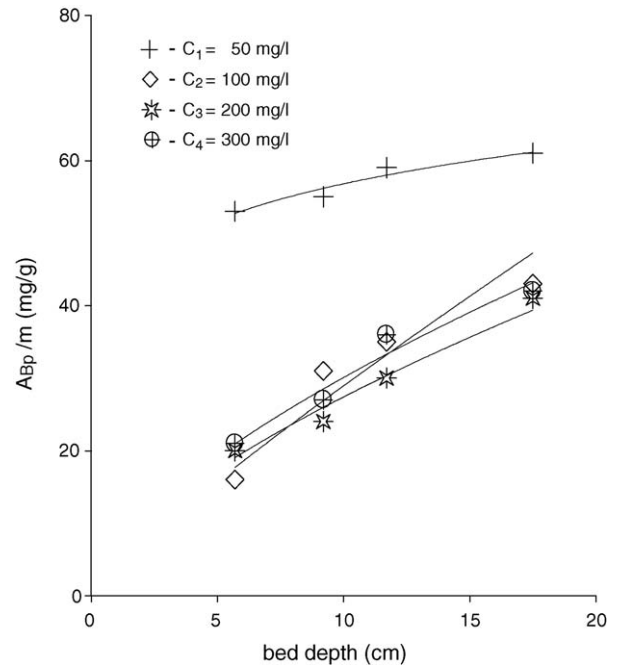


Fig. 8. Variation of (A_{Bp}/m) with bed depth, for several volumetric flows, $C_0=200$ mg/l.

is a simple and effective method making it possible to connect the operating conditions to the working parameters (breakthrough time, volumes treated, quantities of pollutant eliminated ...).

About the quantity of adsorbate eliminated by the activated carbon, its evolution with the different operational variables can be calculated as follows.

From graph no. 7 for each curve, we can deduce a relation of the linear form:

$$\left(\frac{A_{Bp}}{m}\right)_i = aH + \beta_i \tag{1*}$$

where β_i the ordinate variable, is function of the initial concentration: it can be calculated in this manner, from a layout plot of the intercept at the origin against the initial concentration, we deduced a form function:

$$\beta_i = \frac{b}{cQ + d} \Rightarrow \left(\frac{A_{Bp}}{m}\right)_i = aH + \beta_i = aH + \frac{b}{cQ + d} \tag{2*}$$

From graph no. 8 we deduce the same function

$$\left(\frac{A_{Bp}}{m}\right)_i = a'H + \gamma_i = a'H + \frac{b'}{c'Q + d'} \tag{3*}$$

Table 2
Behavior characteristics of MTZ

Parameters	Bed depth	Volumetric flow	Concentration
F	±	±	±
N_Z	+	-	-
U_Z	+	+	+
A_{Bp}	+	-	-
A_{Bp}/m	+	-	-

(±) Increase slightly; (+) increase greatly; (=) no influence; (-) decrease.

a, a' are the coefficients which depend of the bed depth; b, b' ; c, c' ; d, d' , of the concentration and volumetric flow. For the three parameters, we propose (because γ_i vary in the same way that β_i) a global equation which is a combination between (1*), (2*), and (3*):

$$\frac{A_{Bp}}{m} = aH + \frac{\alpha}{\lambda C + \delta Q + \chi} \quad (4^*)$$

The resolution of this equation can easily be done, by using a graphic method. With the current software (grapher1.09-2.D) we can obtain directly the parameters incriminated. This technique of calculation that is in reality very simple is able to determine any operating parameter of the filter, like, the treated volume, the breakthrough time . . .

Of course, the found equations are valid only in a limited operating field.

However, we can use the same method to determine equivalent relations governing any processes. The technique is valid for any treatment of experimental data, and permits to found relations between the operating conditions and the evaluation parameters.

For the quantity eliminated of solute by activated granular carbon at the breakthrough concentration the relation for the three operating conditions is:

$$\frac{A_{Bp}}{m} = 1.50H + \frac{5.75}{0.001C + Q}, \quad (\chi = 0)$$

This equation confirms more than 80% of experimental data with an error less than 10%.

6. Conclusion

Before, the design of an activated carbon filter was based on hydrodynamic considerations without regard of physicochemical characteristics of which both the kinetic and equilibrium of adsorption are depending and cannot be neglected.

If the uptake capacities of activated carbon must to be exploited to its fullest extent, we have to understand clearly the chemistry, and the physics inherent in the process.

The present study has permitted the estimation and the understanding of the influence of parameters interfering in the process of the adsorption, and proposes a simple method to correlate them with the operating conditions.

The intrinsic parameters of the mass transfer zone can explain the elimination efficacy and the variability of the adsorption phenomenon.

The study allowed the establishment of a correlation which is concordant with experimental data: $Y = aH + b/cC + dQ$.

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