

COD removal from textile industry effluent: pilot plant studies

G.M. Walker^{a,*}, L.R. Weatherley^b

^a School of Chemical Engineering, The Queen's University of Belfast,
Stranmillis Road, Belfast BT9 5AG, Northern Ireland, UK

^b Department of Chemical and Process Engineering, University of Canterbury, Christchurch, New Zealand

Abstract

This work involved the treatment of industrial waste water from a nylon carpet printing plant. As dyeing of nylon is particularly difficult, acid dyes, fixing agents, thickeners, finishing agents, are required for successful colouration and cause major problems with the plant's effluent disposal in terms of chemical oxygen demand (COD). Granular activated carbon (GAC) Filtrasorb 400 was used to treat a simulated process plant effluent containing all the pollutants. Equilibrium isotherm experiments were established and experimental data obtained showed good empirical correlation with Langmuir isotherm theory. Column experimental data, in terms of COD were correlated using the bed depth service time (BDST) model. Solid phase loading in the columns were found to approach that in equilibrium studies indicating an efficient use of adsorbent. The results from the BDST model were then used to design a pilot adsorption rig at the plant. The performance of the pilot plant column were accurately predicted by scale-up from the bench scale columns. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

In the design of industrial adsorption towers the effect of many process parameters need to be quantified. The effect of effluent flow rate, effluent concentration and adsorbent particle size have been investigated in pilot scale adsorption towers by many researchers [1,2]. The effluents used in these systems were simulated effluents from process industries containing single component adsorbates including dyes [3], metal ions [4] and trace organic compounds [5]. Results from previous work by these researchers has shown that the removal of the non-coloured agents in textile industry effluent is more problematic, in terms of COD, than visible dye agents [6].

In general granular activated carbon (GAC) processes have attracted interest for municipal wastewater treatment with most of the academic research into this field concentrating on the removal of small concentrations of low molecular weight contaminants, such as phenol, from solution [1]. The first pilot plant scale system developed for the treatment of textile industry wastewater, was the study of Perrotti and Rodman of the Fram corporation into the treatment of dyehouse effluent by biological activated carbon (BAC) systems [7]. Several researchers have since stud-

ied textile wastewater treatment using GAC. Rozzi et al. in comparing several methods of textile effluent treatment concluded that GAC adsorption was the best method for colour removal [8]. Recent reports have also highlighted the effectiveness of GAC treatment for textile wastewater in comparison with other treatment techniques [9,10]. In many cases GAC adsorption is coupled with ozonation [11] or bio-oxidation [12] in order to improve the removal treatment for textile waste. This study however, concerns the use of GAC adsorption as the sole treatment technique for COD removal from textile effluent.

2. Experimental 1 — bench scale

2.1. Effluent characterisation

The work involves the treatment of aqueous effluent from the textile manufacturing plant with COD levels approaching 1400 mg dm^{-3} . Previous work by the authors has indicated that the installation of a GAC system could potentially treat the wastewater [13].

The textile process effluent consisted of not only of dyes but also a thickener (guaranate) surfactants (aphrogen, verolan, thiotan, sand acid) and finishing agents (dyapol, irgadyol), used in the dyeing process (see Table 1). Many of these additives are not adsorbed on the nylon carpet

* Corresponding author. Tel.: +44-28-90274172; fax: +44-28-90381753.
E-mail address: g.walker@qub.ac.uk (G.M. Walker).

Nomenclature

a_L	Langmuir isotherm constant ($l \text{ mg}^{-1}$)
C_0	initial dye concentration (kg m^{-3})
C_b	breakthrough dye concentration (kg m^{-3})
C_e	liquid phase equilibrium concentration (mg l^{-1})
F	linear flow rate (m s^{-1})
K_a	BDST adsorption rate constant ($\text{m}^3 \text{ kg}^{-1} \text{ s}^{-1}$)
K_L	Langmuir isotherm constant ($l \text{ g}^{-1}$)
N_0	BDST adsorption capacity (kg m^{-3})
q_e	solid phase equilibrium concentration (mg g^{-1})
Z	bed height (m)
Z_0	critical bed height (m)
<i>Greek letter</i>	
ρ_b	carbon bulk density in column (kg m^{-3})

and are therefore discharged from the plant in the aqueous effluent.

2.2. Adsorbent specifications

The adsorbent used in this study was the GAC Filtrasorb 400. The adsorbent was washed in de-ionised water and sieved into 1000–1400 μm particle size range before contacting with the adsorbate solutions. The physical properties of the activated carbon is given in Table 2.

Table 1
Composition and COD of process effluent^a

Component	COD ($\text{mg O}_2 \text{ l}^{-1}$)
Tectilon blue	1200
Tectilon red	570
Tectilon orange	603
Guaranate	1500
Aphrogen	1484
Dyapol	942
Irgadypol	1305
Sand acid	272
Verolan	120
Thiotan	858

^a Total COD = 1000–1300 mg l^{-1} .

Table 2
Physical properties of GAC F400 (from Chemviron Carbon)

Total surface area (N_2 BET method) ($\text{m}^2 \text{ kg}^{-1}$)	$(1.05\text{--}1.20) \times 10^6$
Solid phase density (kg m^{-3})	2100
Particle density (wetted in water) (kg m^{-3})	1300–1400
Porosity	0.4
Iodine number	1000–1100
Methylene blue number	280–300

2.3. Equilibrium isotherm experiments

Batch equilibrium experiments were undertaken using a series of mixed solutions of consecutively increasing initial effluent concentration and of fixed volume, which were placed in vessels where they are brought into contact carbon. The mixed solutions had equal dye concentrations on a mass basis. The mass of carbon was pre-determined to give a constant volume to mass ratio. The jars were sealed and placed in a shaker at a constant temperature of 20°C for 3 weeks until equilibrium was reached. The samples were then analysed using standard COD analysis (Section 2.5) for measurement of the liquid dye concentration, C_e (mg l^{-1}). This was then used to calculate the solid phase dye concentration, q_e (mg g^{-1}) by a material balance on the adsorption system. The particle size of the adsorbent was maintained at 1000–1400 μm . As the effluent was essentially neutral the experiments were conducted at a constant pH of 7.

2.4. Bench scale experimental methods

The equipment used during these fixed bed column studies is described previously by the authors [3,13]. It consisted of a 120 l PVC make-up tank in which the process effluent added at a concentration of 1000 mg l^{-1} COD. The solution was then gravity fed to a feed tank, having a volume of 120 l and from which the effluent was pumped using a peristaltic pump at constant flow rate of 7.5 $\text{cm}^3 \text{ min}^{-1}$ ($2.548 \times 10^{-4} \text{ m s}^{-1}$). The effluent solution was fed through a bed of GAC F400 in up flow mode.

The carbon beds were contained in Perspex columns with diameter of 25 mm (i.d.) to which an end plate with an inlet nozzle was attached. The carbon bed was supported in the column by several layers of glass beads of various sizes located on a support plate, which ensured good liquid distribution. Each column had five 5 mm (i.d.) Perspex sample ports stoppered with an Suba-seal bung. This gave a standard bed height of 250 mm which resulted in beds with a carbon mass of approximately 75 g of 1000–1400 μm particle size. Samples were drawn at regular time intervals from these ports using a syringe with a hypodermic needle capable of taking a sample from the centre of the bed.

2.5. COD analysis

The chemical oxygen demand (COD) is an indication of the overall oxygen load that a wastewater will impose on an effluent stream. COD is equal to the amount of dissolved oxygen that a sample will absorb from a hot acidic solution containing potassium dichromate and mercuric ions. The apparatus used in this case was the Hach 2000 COD reactor and spectrophotometer in which a 2 ml sample was contacted, in a vial, with the oxidising acid solution which was then held at 150°C for 2 h. After cooling the sample was then analysed in the spectrophotometer at 620 nm. The colour of

a sample varied from orange to dark green indicating COD strength in the range 0–1500 mg/l.

3. Results and discussion 1 — bench scale

3.1. Equilibrium isotherm analysis

Data obtained from the equilibrium isotherm experiments are illustrated in Fig. 1 as a plot of solid phase equilibrium concentration, q_e versus liquid phase equilibrium concentration, C_e . The experimental data were empirically modelled with the Langmuir isotherm using Eq. (1).

$$q_e = \frac{K_L C_e}{1 + a_L C_e} \quad (1)$$

Results of the best-fit Langmuir analysis are illustrated in Fig. 1 as a solid line. The empirically modelled data showed excellent correlation with experimental data over the entire concentration range investigated ($r^2 = 0.990$), with an empirical Langmuir monolayer capacity (a_L/K_L) of approximately 540 mg g^{-1} .

3.2. Breakthrough curves

The experimental data obtained from the bench scale adsorbers are illustrated as a breakthrough curves in Fig. 2 (0.05 and 0.25 m bed height). The results indicate that the activated carbon column system is successful in removing COD from textile wastewater. The breakthrough curve for 0.05 m (near column inlet) is typically steeper than 0.25 m (column outlet), this can be attributed to the relatively large adsorption zone [1,13]. As the complete adsorption zone is established, represented by the 0.25 m data, a typical “s” shaped curve is found, which is indicative of effective use of adsorbent. To aid the design of the pilot plant adsorber, the experimental breakthrough data were modelled using the bed depth service time (BDST) analysis.

It should be noted that the linear liquid velocity in the adsorbers was quite low ($2.548 \times 10^{-4} \text{ m s}^{-1}$). The decision

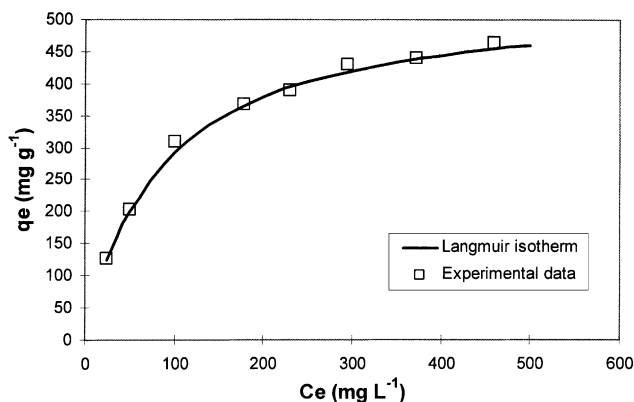


Fig. 1. COD equilibrium isotherm plot for adsorption of textile effluent onto GAC F400, $a_L = 0.01197 \text{ (l mg}^{-1}\text{)}$, $K_L = 6.431 \text{ (l g}^{-1}\text{)}$, $r^2 = 0.990$.

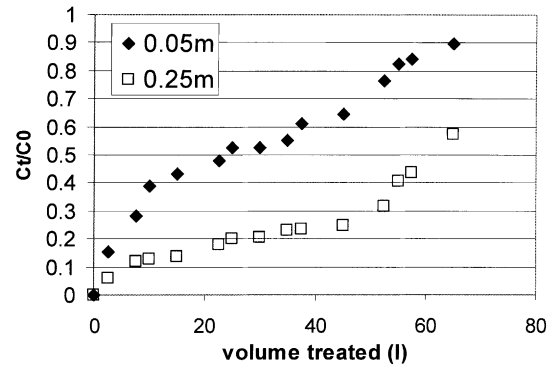


Fig. 2. Adsorption breakthrough data for bench scale adsorber for COD removal at heights of 0.05 and 0.25 m. Concentration = 1000 mg l^{-1} COD, flow rate = $2.548 \times 10^{-4} \text{ m s}^{-1}$, column diameter = 0.025 m , GAC 1000–1400 μm .

to operate the columns at a low linear flow rate was based on previous results by the authors [3] which indicated that, due to low diffusivity of the adsorbate species, low linear flow rates greatly extended the operational life of the adsorber.

3.3. Bed depth service time model

The BDST model is well established for dye adsorption in fixed bed systems [13] therefore only a brief description is included. In the operation of fixed bed adsorbers, the objective is to reduce the concentration in the effluent so that it does not exceed a pre-defined breakthrough value, C_b . Initially, when the activated carbon is unsaturated the actual effluent concentration is lower than C_b , but as the effluent is pumped through the bed the carbon becomes saturated and the effluent concentration approaches C_b , i.e. the break-point is reached. The original work on the BDST model was carried out by Bohart and Adams [14], who proposed a relationship between bed depth, Z , and the time taken for breakthrough to occur. The service time, t was related to the process conditions and operating parameters, see Eq. (2).

$$\ln\left(\frac{C_0}{C_b} - 1\right) = \ln(e^{K_a N_0 Z/F} - 1) - K_a C_0 t \quad (2)$$

Hutchins [15] proposed a linear relationship between the bed depth and service time, see Eq. (3).

$$t = \frac{N_0}{C_0 F} Z - \frac{1}{K_a C_0} \ln\left(\frac{C_0}{C_b} - 1\right) \quad (3)$$

Eq. (3) enables the service time, t of an adsorption bed to be determined by a specified bed depth, Z , of adsorbent. The service time and bed depth are correlated with the process parameters and initial pollutant concentration, solution flow rate and the adsorption capacity.

The analysis of the experimental breakthrough data using the BDST theory, yielded a plot of bed depth versus service time, illustrated in Fig. 3. The linearisation of the experimental data using this technique proved quite successful, for the textile waste activated carbon system ($r^2 = 0.995$).

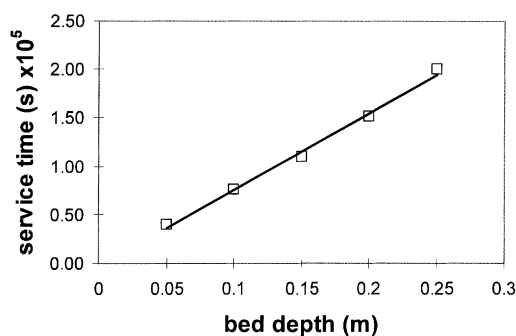


Fig. 3. Adsorption BDST plot for bench scale adsorber for COD removal at heights of 0.05–0.25 m, for 20% breakthrough. Concentration = 1000 mg l^{-1} COD, flow rate = $2.548 \times 10^{-4} \text{ m s}^{-1}$, column diameter = 0.025 m, GAC 1000–1400 μm .

The BDST parameters, namely, BDST adsorption capacity, N_0 , and rate constant, K_a were calculated from the linearised experimental data and are presented in Table 3, for 10, 20, 40, and 60% breakthrough. The critical bed depth, Z_0 (m) is the theoretical depth of adsorbent sufficient to prevent the adsorbate concentration exceeding C_b at time $t = 0$ (calculated from Eq. (3), letting $t = 0$). The critical bed depths shown in Table 3 are less than 10% of the total bed height in the bench scale columns (for 10 and 20% breakthrough), indicating that the columns are operating efficiently, and enabling BDST scale-up to the pilot rig. The BDST adsorption capacity, N_0 , was calculated as kg of adsorbate per m^3 of adsorbent. The value of N_0 has also been calculated in terms of mg of adsorbate per g of adsorbent, using the bulk density of the carbon bed. This enables a direct comparison with the equilibrium isotherm data, which can be used to establish the efficiency of the adsorber.

As an example, an equilibrium liquid phase concentration of 200 mg l^{-1} (i.e. 20% breakthrough using initial concentration of 1000 mg l^{-1}), would result in an equilibrium solid phase concentration of 380 mg g^{-1} , from the Langmuir equation. The adsorber capacity calculated using the BDST data resulted in a solid phase concentration of 250 mg g^{-1} , which is approximately 65% of the equilibrium value.

Results of the comparison of solid phase loading in equilibrium and column studies are presented in Table 3. These data indicate that an increase in extent of breakthrough results in an increase in solid phase loading of the columns and an increase in percentage of equilibrium capacity. The

high solid phase loading approaching equilibrium values is indicative of efficient use of adsorbent for COD removal from textile wastewater. The adsorption capacity expressed as percentage of equilibrium capacity for COD removal, is very favourable compared to previous work (typically 5%), by the authors for acid dye adsorption [13].

It is postulated that this extreme variation can be attributed to a number of factors: (i) the lower linear flow rates used in this work allowed more time for intraparticle diffusion; (ii) the equilibrium isotherms for the dyes reached the “monolayer” capacity at low liquid phase concentrations (i.e. Langmuir constant, K_L very much higher for dye adsorption); (iii) the intraparticle diffusivity of the colourless components which make-up the bulk of the COD could be much higher than that of the dyes, which were noted in previous work of having a very low effective diffusivity [3].

4. Experimental 2 — pilot plant scale

4.1. Effluent selection

The inlet feed to the pilot GAC treatment was taken from the discharge from the carpet washing line. This is a combined effluent from two vacuum dryers and the two cold water rinses which produces an effluent with a high COD content ($1000\text{--}1300 \text{ mg l}^{-1}$). As the process line is in semi-continuous operation and the large volume of effluent discharged by the vacuum dryers occurs periodically a holding tank was necessary for the pilot plant.

4.2. Pilot plant layout

Fig. 4 illustrates the general layout of the pilot plant at the printing plant. It consists of two 2 m high PVC columns (A + B), supported by a tubular steel rig, fed in a up flow direction by a centrifugal pump (D) from a feed tank (E), see Table 4 for details. This tank consists of three interconnected 1000 dm^3 PVC tanks and is feed by a centrifugal pump from the pump with an overflow system returning effluent to the drain. Flow rate was maintained in the unit by valved rotameters (R_{1-2}) which allowed the flow rate to be varied from 10 to $100 \text{ dm}^3 \text{ h}^{-1}$. The outlet from the columns was analysed before being sent to drain.

Table 3

Comparison of BDST capacity constants and equilibrium data^a

Breakthrough (%)	N_0 (kg m^{-3})	N_0 (mg g^{-1})	Equilibrium (%)	Z_0 (m) ^b	K_a ($\text{m}^3 \text{ kg}^{-1} \text{ s}^{-1}$) ^b
10	0.46×10^5	56.8	19.4	0.0278	4.39×10^{-7}
20	2.02×10^5	249	65.7	0.0040	4.33×10^{-7}
40	3.26×10^5	402	90.5	—	—
60	3.31×10^5	408	86.6	—	—

^a Concentration = 1000 mg l^{-1} COD, flow rate = $2.548 \times 10^{-4} \text{ m s}^{-1}$, column diameter = 0.025 m, GAC 1000–1400 μm , $\rho_b = 810 \text{ kg m}^{-3}$.

^b Values of Z_0 and K_a could not be computed for 40 and 60% breakthrough due to positive intercept on BDST plot.

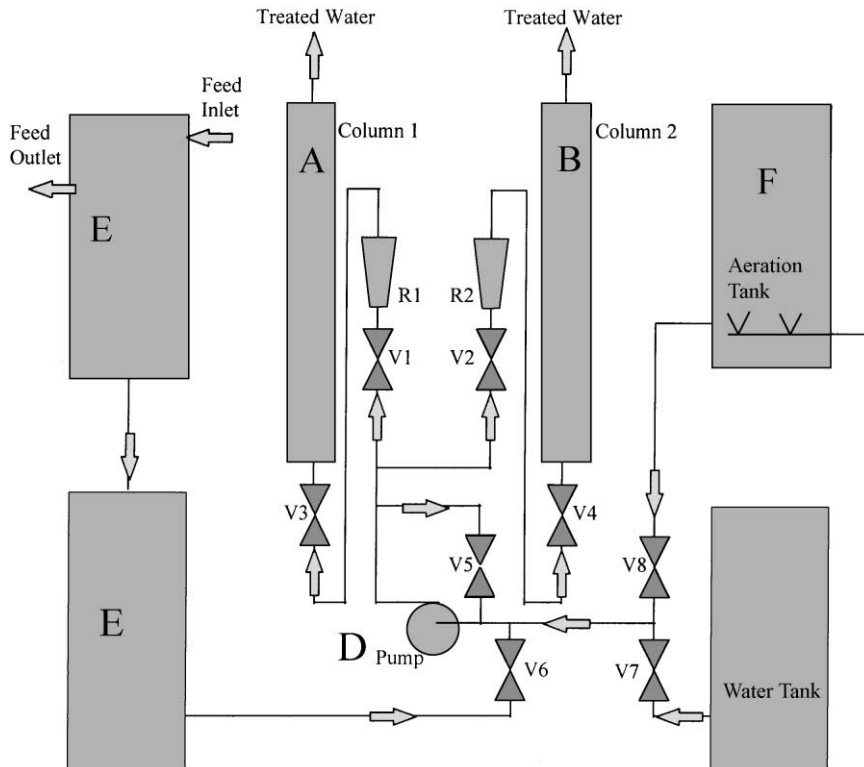


Fig. 4. Layout of GAC pilot plant at Carpets International (10801 per day).

4.3. Column design

The major design parameter for any adsorber system is the residence or empty bed contact time (EBCT). Due to the diffusion controlling nature of acid dye adsorption a long contact time is required for the efficient use of the activated carbon [3]. This can be achieved by two methods.

1. Increasing the diameter of the columns, this serves to increase the contact time. The dimensions of the column however, were directly scaled-up from the 25 mm i.d. bench scale columns. Therefore, the pilot scale 250 mm column had a bed height of 1000 mm.
2. Decreasing the volumetric flow rate in the columns also serve to increase the contact time and as with the column

diameter a linear flow rate was chosen from a scale-up from the bench scale experiments ($45 \text{ m}^3 \text{ h}^{-1}$). The linear flow rate for the bench scale and pilot plant were identical and effluent inlet concentration was on average 20% higher in the pilot plant. The BDST parameters, N_0 and K_a calculated from the bench scale work were used in Eq. (3) to predict the service time on the pilot plant. It was found, especially at higher extent of breakthrough, that N_0 was the major parameter in predicting service time of the columns.

The grade of activated carbon used in the pilot plant was GAC F400, identical to the bench scale studies, although in the pilot plant the carbon was used as supplied by Chemviron Carbon. This carbon was of a broader particle size

Table 4
Pilot plant specifications

Item	Description
A	Column
B	Column
C	Support rig
E	Feed tank
F	Aeration tank
R ₁₋₂	Rotameters
V ₁₋₂	Control valves
V ₃₋₈	Bail valves
	Pipework

250 mm i.d. × 2000 mm height × 4 mm thick PVC
detailed design in Section 4.2

Kee Klamp (5) tubular galvanised steel

3 × 1000 dm³ PVC with calibration

1000 dm³ PVC with calibration plus 750 mm PVC tubular aerator
(for BAC not required for this study)

2 × GF type SK rotameters (10–100 dm³ min⁻¹)

2 × GF type 215 diaphragm valves (PVC)

10 × GF type 346 ball valves

Assorted 1/2", 3/4" and 1" (class C) GF PVC pipes/fittings

Table 5
Particle size distribution of GAC F400

Size range (µm)	wt.%
<150	0.1
150–250	0.2
250–355	0.5
355–500	2.9
500–710	11.2
710–1000	18.1
1000–1400	65.9

distribution than carbon used in the bench scale studies (see Table 5).

Fig. 5 illustrates the detailed design of the column with the 1000 mm carbon bed supported above and below by layers of plastic rings (10 mm × 10 mm by 200 mm height) and sand ((0.1–0.2) mm × 100 mm height) and separated from the carbon bed by an aluminium gauze. This packing provides good liquid distribution for adsorption with a small pressure drop. The packing is supported by flanged end plates above and below the bed. These plates are 5 mm thick circular discs with 16 mm × 0.6 mm bolts equally spaced along the circumference holding it to the column shell with a 3 mm thick Neoprene flange between the end plate and a PVC rim at the column ends.

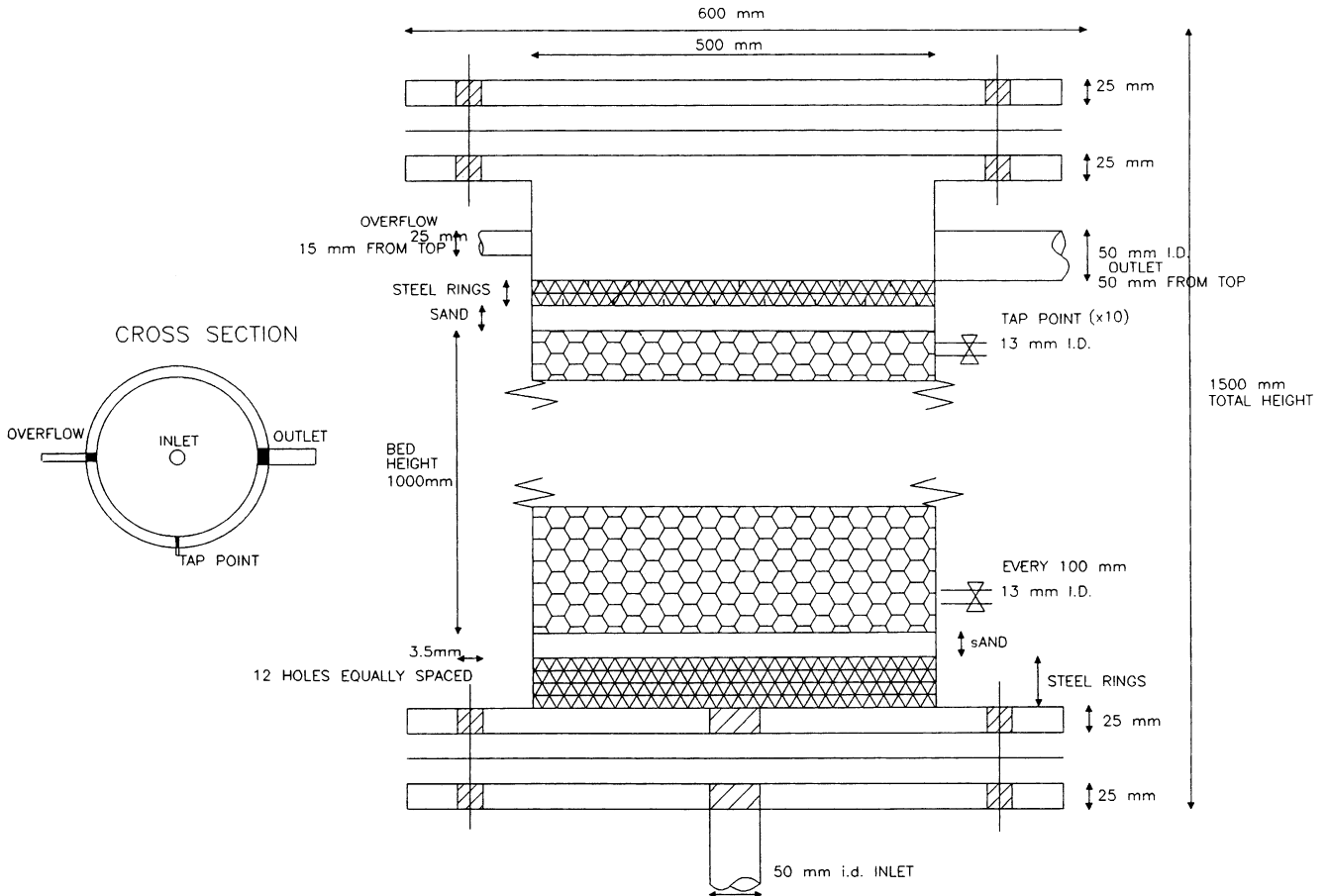


Fig. 5. Detailed design of pilot scale adsorption column.

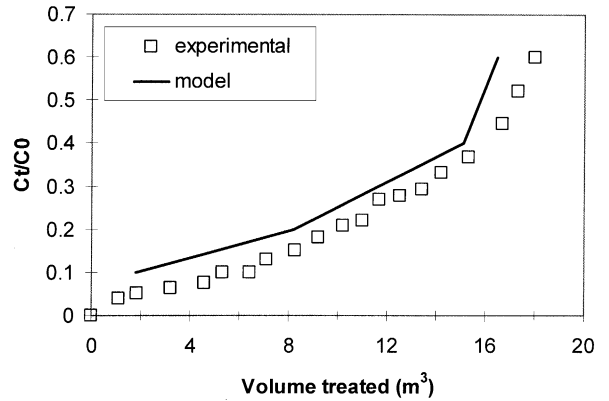


Fig. 6. Pilot plant performance for COD removal, predicted and actual data. Concentration = 1200 mg l⁻¹ COD, flow rate = 2.548 × 10⁻⁴ m s⁻¹, column diameter = 0.25 m, GAC F400 as supplied.

5. Results and discussion 2 — pilot plant scale

Results from the pilot plant studies indicate that GAC systems can successfully treat the textile wastewater (Fig. 6). The performance of the pilot plant column were fairly accurately predicted by the BDST model from 10 to 60% breakthrough. A plot of pilot plant experimental data versus

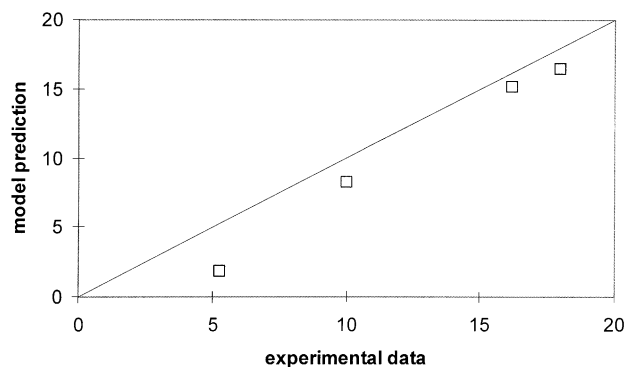


Fig. 7. Model prediction vs. plant data, $r^2 = 0.930$. Concentration = 1200 mg l^{-1} COD, flow rate = $2.548 \times 10^{-4} \text{ m s}^{-1}$, column diameter = 0.25 m, GAC F400 as supplied.

BDST model prediction is shown in Fig. 7, and indicates a good prediction over the course of the plant trial, with regression coefficient of $r^2 = 0.930$. It was noted that the error between the experimental and predicted data was more significant at lower dimensionless concentrations, and that the pilot scale column outperformed the bench scale column in COD removal over the dimensionless concentrations investigated. This deviation can be attributed to:

1. fines present in the coarse F400 used in the pilot plant tests would increase the expected performance of the bed, due to a higher bed bulk density, and a lower mean particle size;
2. the flow rate in the pilot plant columns could not be maintained as accurately as for the bench scale tests because: (a) increased pressure drop in the pilot plant columns; (b) the flow rate was monitored and adjusted every 1 to 2 days in the pilot plant rather than every few minutes with the smaller columns;
3. the effluent wastewater fed to the columns did not remain at a constant concentration due to: (a) each carpet being manufactured produced an effluent of different concentrations; (b) the simulated plant effluent used in laboratory scale tests was a rough average of the actual plant effluent; (c) biological degradation of guaranate (thickener) occurred in the feed tanks due to the long holding time, which did not occur in the smaller columns.

6. Conclusions

COD was chosen as the parameter to characterise effluent from a high volume textile producer. High solid phase loading in equilibrium isotherm experiments indicated that GAC adsorption may provide a treatment for the effluent. Equilibrium results were validated in bench scale fixed bed column studies in which COD from the textile effluent was removed on a continuous basis. The bench scale experimental data as

modelled using the BDST approach with adsorption capacity and adsorption rate parameters calculated. Solid phase loading in the columns, calculated from the BDST adsorption capacity, were found to approach that in equilibrium studies indicating an efficient use of adsorbent. The BDST data was used to design a pilot plant adsorption column which was used to treat effluent from textile plant. Results from the pilot plant studies showed good correlation with the BDST model and indicated that GAC adsorption could successfully reduce the COD of textile effluent in fixed bed columns.

Acknowledgements

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