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Fixed bed column study for the removal of crystal violet (C. I. Basic Violet 3) dye from aquatic environment by surfactant-modified alumina

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Abstract

The fixed bed column study was undertaken for the removal of crystal violet (CV), also known as Basic Violet 3, a well-known cationic dye from wastewater using surfactant-modified alumina (SMA) as an adsorbing media. The SMA was found to be a very efficient media for the removal of dye from wastewater. The column with 2 cm diameter, different bed heights such as 10, 20 and 30 cm could treat 3.58 l, 7.94 l and 12.51 l, respectively, of CV bearing wastewater with CV concentration 200 mg/l and flow rate 8.5 ml/min. The adsorption bed was exhausted within a very short time (~1.5 h) after the breakthrough occurred. This indicated that the adsorption zone was almost saturated at the time of exhaustion. Different column design parameters like depth of exchange zone, time required for exchange zone to move its own height, adsorption rate, adsorption capacity etc. were calculated. Effect of flow rate and initial concentration was studied. Theoretical breakthrough curve was drawn from the batch isotherm data and was compared with experimental breakthrough curve. Desorption of CV from the SMA surface was possible using 1 M sodium hydroxide solution, rectified spirit and acetone.

Keywords: Alumina; Sodium dodecyl sulfate; Surfactant-modified alumina; Crystal violet; Adsorption; Column study; Breakthrough curve

1. Introduction

Dyes are the one of the major constituents of the wastewater produced from many industries related to textile, paint and varnishes, ink, plastics, pulp and paper, cosmetics, tannery etc., and also to the industries, which produce dyes. Colored dye effluents pose a major threat to the surrounding ecosystem. Many of the dyes are extremely toxic. Among various dyes, crystal violet (CV), also known as Basic Violet 3, is a well-known cationic dye being used for various purposes: a biological stain, a dermatological agent, a veterinary medicine, an additive to poultry feed to inhibit propagation of mold, intestinal parasites and fungus etc. It is also extensively used in textile dying and paper printing. It is a mutagen and mitotic poison. Various treatment technologies like adsorption [1–5], photodegradation [6–10], coagulation flocculation [11], chemical oxidation [12,13], electrochemical oxidation [14], biological process [15] etc. are available for the removal of dye from the wastewater.

Many of the above mentioned processes are not cost-effective and hence are not suitable for being applied in developing countries. Very recently removal of organic contaminants using surfactant-modified solid surface has drawn much attention. The solid surface can be modified by surfactant to form micelle like structures on its surface having the potential to solubilize organic

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molecules within the structures formed. These micelles are called hemimicelle or admicelle and the phenomenon is called adsolubilization. For the last two decades this new separation technology has been used for the removal of different organic compounds [16–19]. Most of the studies mentioned above have been conducted in batch mode. Reports regarding the removal of pollutants in continuous mode using fixed bed column are only a few. Thus, the present study aims towards the removal of CV from CV bearing wastewater in a fixed bed column of sodium dodecyl sulfate (SDS) treated alumina. This surfactant-modified alumina (SMA) is found to be an efficient adsorbing material for CV even when it is present at a very high concentration in the wastewater and hence can be used as a practical adsorbent for the treatment of CV bearing wastewater. The authors feel that this is the first report where SMA is being used for dye removal from dye bearing wastewater in continuous mode.

2. Materials and methods

2.1. Reagents

Acridine orange (ACO), CV, SDS, glacial acetic acid, toluene, and acetone were from BDH (AR grade) and were used as received. All other chemicals used in this study were of high purity and used without further purification.

2.2. Instrumentation

A high precision electrical balance (Sartorious GMBH) and a digital pH meter (DHP-500, SICO, India) were used for weighing and for pH measurement, respectively. A spectrophotometer (Thermo Spectronic UV1, UK) was used for absorbance measurement.

2.3. Preparation of surfactant-modified alumina (SMA)

Alumina was treated by anionic surfactant (AS) to form micelle like structure on their surfaces. SDS was used as the representative of AS. This SMA was used for the removal of CV from water environment.

Alumina was from SRL, India and was used as such without further grinding and sieving. The granulation of neutral alumina was 70–290 mesh ASTM, molecular weight 101.96 and zero point charge (Z_{pc}) 9.15.

Alumina (200 g) was shaken for 24 h with 2 l of SDS solution having a concentration of 20 000 mg/l. To increase the adsorption capacity of alumina NaCl was added to the solution and the pH was kept low. The NaCl dose was 2500 mg/l and the pH was 4.4 ± 0.1. After shaking, the supernatant was discarded and the alumina was washed thoroughly initially with tap water and finally with distilled water. Then the material was dried at 60 °C for 24 h. The alumina thus obtained is called surfactant-modified alumina (SMA) and was used as an adsorbent for CV removal. The SDS coverage on alumina surface was found to be 111.6 mg/g.

2.4. Analytical method

A rapid and reliable solvent extraction spectrophotometric method has been developed for the determination of anionic surfactant (AS). Acridine orange (ACO) chemically known as 3,6-bis(dimethylamino) acridine having a colour ($\lambda_{max} = 467$ nm) has the potential for being used as an ion-pairing agent with AS. Sample solution (10 ml) containing SDS (a representative of AS) in the range of 0.1–6.0 mg/l was transferred into a 25 ml separating funnel. ACO (5×10^{-3} M) and glacial acetic acid 100 μl each was added. Then 5 ml of toluene was added to it and shaken for 1 min. The aqueous layer was then discarded and the toluene layer was used for absorbance measurement at 467 nm.

CV was measured by spectrophotometric method at its λ_{max} 591 nm at neutral pH.

2.5. Experimental studies

Fixed bed column studies were conducted using columns having 2 cm diameter and 55 cm length. The column was packed with SMA between two supporting layers of glass wool. The bed depths were taken as 10, 20 and 30 cm. The schematic diagram of the column study is shown in Fig. 1. The column was charged with CV bearing wastewater in the up flow mode with a volumetric flow rate of 8.5 ml/min (~ 1.63 $m^3/m^2/h$). The initial concentration of CV was 200 mg/l. The samples were collected at certain time intervals and were analysed for remaining CV concentration. Experiment was also conducted with an initial CV concentration of 400 mg/l, bed depth 10 cm and flow rate 8.5 ml/min. Effect of flow

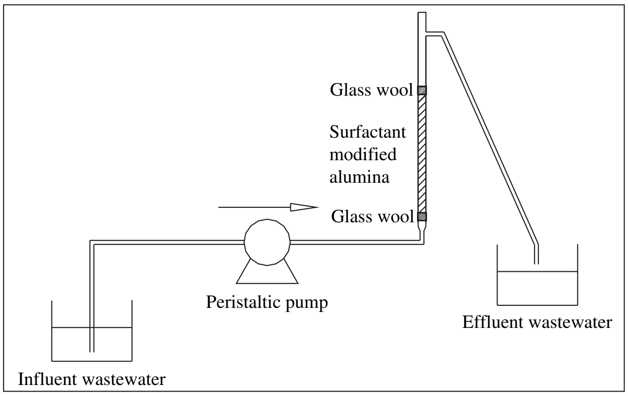

Fig. 1. Schematic diagram of lab-scale column study.

rate was also studied, where columns were run with flow rates of 5 and 12 ml/min, with the bed depth of 10 cm and initial CV concentration of 200 mg/l. In all the cases the temperature was 27 ± 2 °C and the pH was 6.9 ± 0.1.

After exhaustion of the SMA by CV it is necessary to regenerate it for further use. Aqueous NaOH, rectified spirit and acetone were used for the regeneration of the exhausted SMA.

3. Results and discussion

3.1. Behaviour of adsorption column

Fixed bed column studies were conducted using columns having 2 cm diameter and 55 cm length. The columns were packed with SMA at different depths – 10, 20 and 30 cm. The column was charged with CV-spiked distilled water in the up flow mode with a volumetric flow rate of 8.5 ml/min (~1.63 $m^3/m^2/h$). The initial concentration of CV was 200 mg/l. The breakthrough curves are shown in Fig. 2. The adsorption bed was exhausted within a very short time (~1.5 h) after the breakthrough occurred, which indicated that the adsorption zone was almost saturated at the time of exhaustion. These types of breakthrough curves are highly desirable. The breakthrough times (corresponding to $C/C_0 = 0.001$) were found to be 7.02, 15.58 and 24.53 h for 10, 20 and 30 cm bed depth, respectively. The exhaust times (corresponding to $C/C_0 = 0.9$) were 8.25, 16.81 and 25.80 h, respectively. The corresponding volumes of wastewater treated at breakthrough point were 3.58, 7.94 and 12.51 l, respectively, and at exhaust point 4.20, 8.57 and 13.15 l, respectively. Different parameters for column have been calculated from the above data.

The time required for the exchange zone to move the length of its own height up/down the column once it has become established is

$$t_z = \frac{V_E - V_B}{Q_w} \tag{1}$$

where, V_E = total volume of wastewater treated to the point of exhaustion (l); V_B = total volume of wastewater treated to the point of breakthrough (l); Q_w = wastewater flow rate (l/h).

The time required for the exchange zone to become established and move completely out of the bed is

$$t_E = \frac{V_E}{Q_w} \tag{2}$$

Rate at which the exchange zone is moving up or down through the bed is

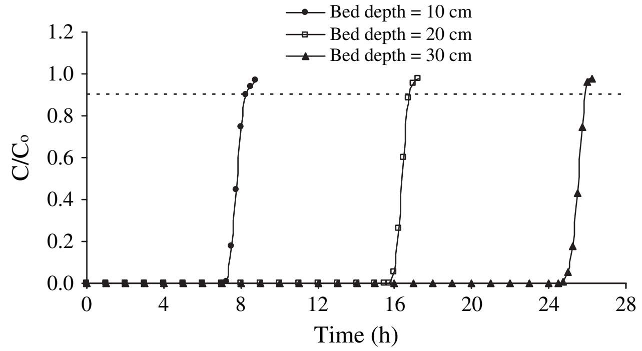

Fig. 2. The breakthrough curves of CV removal by SMA packed columns of different bed depth.

$$U_z = \frac{h_z}{t_z} = \frac{h}{t_E - t_f} \tag{3}$$

where, h_z = height of exchange zone (cm); h = total bed depth (cm); t_f = time required for the exchange zone to initially form (h).

Rearranging Eq. (3) provides an expression for the height of the exchange zone as given below.

$$h_z = \frac{h(t_z)}{t_E - t_f} \tag{4}$$

The value of t_f can be calculated as follows:

$$t_f = (1 - F)t_z \tag{5}$$

At breakthrough the fraction of adsorbate present in the adsorption zone still possessing ability to remove solute is

$$F = \frac{S_z}{S_{max}} = \frac{\int_{V_B}^{V_E}(C_0 - C)\,dV}{C_0(V_E - V_B)} \tag{6}$$

where, C_0 = initial solute concentration (mg/l); S_z = amount of solute that has been removed by the adsorption zone from breakthrough to exhaustion; S_{max} = amount of solute removed by the adsorption zone if completely exhausted.

The percentage of the total column saturated at breakthrough is

$$\%\ \text{saturation} = \frac{h + (F - 1)h_z}{h} \times 100 \tag{7}$$

The values of the important design parameters for 10, 20 and 30 cm bed depth were calculated and are shown in Table 1.

Table 1
Important column behaviour parameters

Parameter	Bed depth = 10 cm	Bed depth = 20 cm	Bed depth = 30 cm	Average value
t_z (h)	1.30	1.23	1.27	1.27
h_z (cm)	1.60	1.50	1.52	1.54
U_z (cm/h)	1.30	1.21	1.20	1.23
Bed saturation (%)	92.4	97.60	96.65	

3.2. Evaluation of adsorption column design parameters

Data collected during laboratory and pilot plant tests serve as the basis for the design of full-scale adsorption columns. A number of mathematical models have been developed for the use in design. Among various models, model proposed by Bohrat and Adams [20] is widely used. The equation, which is based on surface reaction rate theory, can be represented as follows:

$$\ln\left(\frac{C_0}{C_B}-1\right)=\ln\left(e^{KN_0\frac{x}{V}}-1\right)-KC_0t \tag{8}$$

where, C_0 = initial concentration of solute (mg/l); C_B = desired concentration of solute at breakthrough (mg/l); K = adsorption rate constant (l/mg-h); N_0 = adsorption capacity (l/mg); x = bed depth of column; V = linear flow velocity of feed to bed (cm/h); t = service time of column under above conditions (h).

The equation can be rearranged to yield an expression for service time, t. Realizing that $e^{KN_0\frac{x}{V}}\gg 1$, the above equation simplifies to

$$\ln\left(\frac{C_0}{C_B}-1\right)=\ln e^{KN_0\frac{x}{V}}-KC_0t \tag{9}$$

Solving the above equation for t,

$$t=\frac{N_0}{C_0V}x-\frac{1}{C_0K}\ln\left(\frac{C_0}{C_B}-1\right) \tag{10}$$

The form of the Bohart–Adams equation, shown as Eq. (10) can be used to determine the service time, t, of a column of bed depth, x, given the values of N_0, C_0 and K which must be determined for laboratory columns operated over a range of velocity values, V.

Setting $t = 0$ and solving Eq. (10) for x yields

$$x_0=\frac{V}{KN_0}\ln\left(\frac{C_0}{C_B}-1\right) \tag{11}$$

where, x_0 is the minimum column height necessary to produce an effluent concentration C_B.

At least nine individual column tests must be conducted to collect the laboratory data required for the Bohart–Adams approach, an expensive and time-consuming task. A technique has been presented by Hutchins [21], which requires only three column tests to collect the necessary data. In this technique, called the bed depth service time (BDST) approach, the Bohart–Adams equation is expressed as

$$t=ax+b \tag{12}$$

where,

$$a=\text{slope}=\frac{N_0}{C_0V} \tag{13}$$

$$b=\text{intercept}=\frac{1}{KC_0}\ln\left(\frac{C_0}{C_B}-1\right) \tag{14}$$

As it is already mentioned, the breakthrough times (corresponding to $C/C_0 = 0.001$) were found to be 7.02, 15.58 and 24.53 h for 10, 20 and 30 cm bed depth, respectively, and the exhaust times (corresponding to $C/C_0 = 0.9$) were found to be 8.25, 16.81 and 25.80 h, respectively. Fig. 3 showed the depth vs. service time plot for 0.1% and 90% saturation of column. The equations of these lines were as follows:

For 90% saturation

$$t=0.8775x-0.5967 \tag{15}$$

For 0.1% saturation

$$t=0.8754x-1.796 \tag{16}$$

From the slope it was found that these lines were parallel and the horizontal distance between the lines was found to be 1.45 cm. This horizontal distance is called the height of exchange zone. Theoretically this value was found to be 1.54 cm (Table 1).

From the slope and the intercept of the 0.1% saturation line design parameters like K and N_0 could be found using Eqs. (13) and (14). The minimum column height (x_0) necessary to produce an effluent concentration C_B was calculated using Eq. (11). The values of K, N_0 and x_0 were found to be 0.019228 1/mg-h, 28410 mg/l

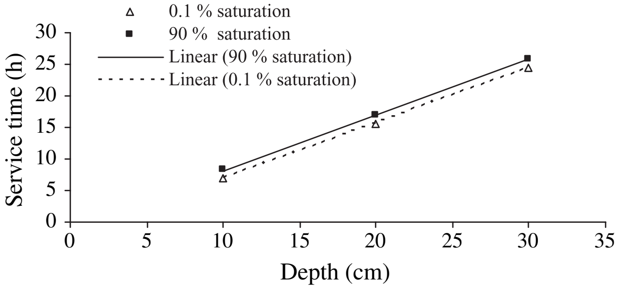

Fig. 3. BDST curve of CV removal.

and 2.05 cm, respectively. The values of K and N_0 indicated that the SMA is highly efficient for the removal of CV from water environment.

3.3. Design of adsorption column

The column design parameters as obtained earlier could be used for the design of adsorption column for practical purpose. According to BDST approach, if a value is determined for one flow rate, values for the other flow rates can be calculated by multiplying the original slope a by the ration of the original and the new flow rates. It is not necessary to adjust b value, since this term is assumed to be insignificantly affected by changing flow rates.

It is also proposed that the data collected for one influent solute concentration can be adjusted by the BDST technique and used to design systems for treating other influent solute concentrations. If a laboratory test is conducted at solute concentration C_1, yielding an equation of the form

$$t = a_1 x + b_1 \tag{17}$$

then it is possible to predict the equation for concentration C_2 as follows:

$$a_2 = a_1 \frac{C_1}{C_2} \tag{18}$$

$$b_2 = b_1 \left(\frac{C_1}{C_2}\right) \frac{\ln\left(\frac{C_2}{C_F} - 1\right)}{\ln\left(\frac{C_1}{C_B} - 1\right)} \tag{19}$$

where, a_1 = slope at concentration C_1; a_2 = slope at concentration C_2; b_1 = intercept at concentration C_1; b_2 = intercept at concentration C_2; C_F = effluent concentration at influent concentration C_2; C_B = effluent concentration at influent concentration C_1.

3.3.1. Design of adsorption column for different flow rates

Columns were run with flow rates of 5 ml/min and 12 ml/min, whereas original flow rate was 8.5 ml/min. The initial CV concentration was kept constant at 200 mg/l. The bed depth and column diameter were 10 and 2 cm, respectively. The breakthrough times (corresponding to 0.2 mg/l effluent concentration) were found to be 12.70 and 4.25 h for 5 and 12 ml/min flow rates, respectively (Fig. 4). The revised values of a were calculated from flow rate ratio and the values were found to be 1.4881 and 0.6200 for 5 and 12 ml/min flow rate, respectively. The value of intercept was −1.796. From these vales of a and b, the service times for 10 cm column were calculated and these are 13.08 and 4.40 h for 5 and 12 ml/min, respectively. These values were comparable with the experimental values.

3.3.2. Design of adsorption column for different initial concentrations

Column was run with initial CV concentration of 400 mg/l, whereas original CV concentration was 200 mg/l. The flow rate was kept at 8.5 ml/min. The bed depth and column diameter were 10 and 2 cm, respectively. The breakthrough time was found to be 3 h (Fig. 5). The effluent concentration was taken as 0.2 mg/l, which was taken for 200 mg/l initial CV concentration. The values of a_2 and b_2 calculated from Eqs. (16) and (17) were 0.4377 and 0.9881, respectively. From these vales of a and b, the service time for 10 cm column was calculated and this was 3.35 h. This value was comparable with the experimental values.

3.4. Theoretical breakthrough curve

Using the data obtained from the batch isotherm studies, it is possible to predict the theoretical breakthrough curve, which can be well compared with the experimental curve. The theoretical breakthrough curve was generated following the concepts of Michaels [22]. The detail calculations for 200 mg/l initial CV

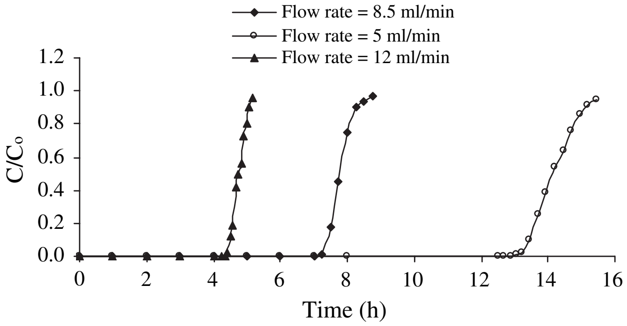

Fig. 4. Breakthrough curves of CV removal by SMA packed column for different flow rate.

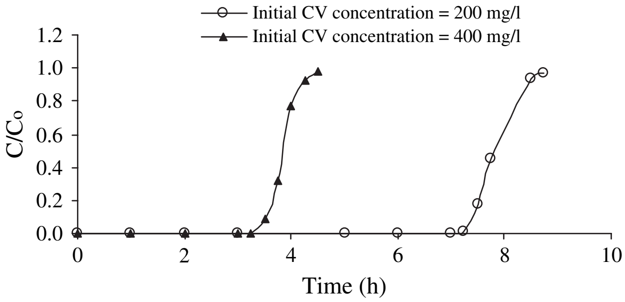

Fig. 5. Breakthrough curves of CV removal by SMA packed column for different initial concentration of CV.

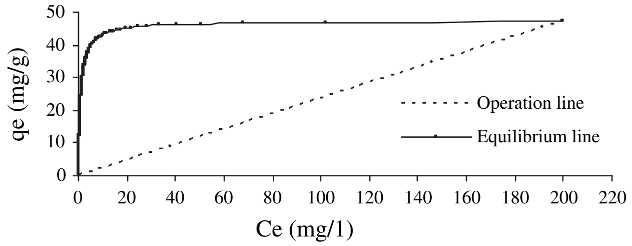

Fig. 6. Equilibrium and operating lines for determination of theoretical breakthrough curve of CV removal by SMA packed column.

concentration and 0.2 mg/l breakthrough CV concentration are as follows:

1. The equilibrium line was prepared (Fig. 6) using Langmuir adsorption isotherm as follow:

$$\frac{1}{q_e} = \frac{0.0216}{C_e} + 0.0211 \tag{20}$$

   where, q_e is the amount of CV (mg) adsorbed per unit weight of SMA (g) and C_e is the equilibrium concentration of CV remaining in the solution (mg/l).
2. An operating line was drawn which was passing through the origin and the point given by C_0 and corresponding q_e. The q_e value corresponding to 200 mg/l CV concentration was 47.15 (mg/g). The significance of this line was that the data of continuously mixed batch reactor (CMBR) and the data of fixed bed reactor (FBR) are identical at these two points, first at the initiation and other at the exhaustion of the reaction.
3. According to Weber [23], the rate of transfer of solute from solution over a differential depth of column, dh, is given by

$$F_w \, dC = K_a (C - C^*) \, dh \tag{21}$$

   where, F_w = wastewater flow rate; K_a = overall mass transfer coefficient, which includes the resistances offered by film diffusion and pore diffusion; C^* = equilibrium concentration of solute in solution corresponding to an adsorbed concentration, q_e. The term $(C - C^*)$ is the driving force for adsorption and is equal to the distance between the operating line and equilibrium curve at any given value of q_e. Integrating Eq. (21) and solving for the height of the adsorption zone,

$$h_z = \frac{F_w}{K_a} \int_{C_B}^{C_E} \frac{dC}{(C - C^*)} \tag{22}$$

   The plot of $(C - C^*)^{-1}$ vs. C is shown in Fig. 7. The area under the curve represented the value of the above integration.
   For any value of h less than h_z, corresponding to a concentration C between C_B and C_E, Eq. (22) can be written as

$$h = \frac{F_w}{K_a} \int_{C_B}^{C} \frac{dC}{(C - C^*)} \tag{23}$$

   Dividing Eq. (23) by Eq. (22) results in

$$\frac{h}{h_z} = \frac{\int_{C_B}^{C} \frac{dC}{(C - C^*)}}{\int_{C_B}^{C_E} \frac{dC}{(C - C^*)}} = \frac{V - V_B}{V_E - V_B} \tag{24}$$

   where V_B and V_E are total volume of water treated till breakthrough and up to exhaust point, respectively, and V is the volume of water treated within V_E for effluent concentration C within C_E. Dividing the values of $\int_{C_B}^{C} dC/(C - C^*)$ by the value of $\int_{C_B}^{C_E} dC/(C - C^*)$ the term $(V - V_B)/(V_E - V_B)$ was evaluated.
4. Now the plot of C/C_0 vs. $(V - V_B)/(V_E - V_B)$ represents the theoretical breakthrough curves.

Fig. 8 showed the experimental and theoretical breakthrough curves. The experimental and theoretical breakthrough curves followed the same trend, but there was difference between these. The reason behind this was that it was assumed that the isotherm followed the

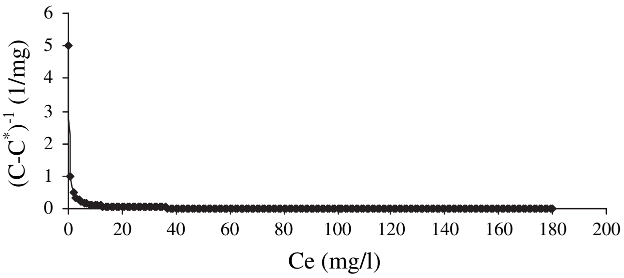

Fig. 7. Curve to evaluate $\int_{C_B}^{C_E} dC/(C - C^*)$ for drawing theoretical breakthrough curve of CV removal by SMA packed column.

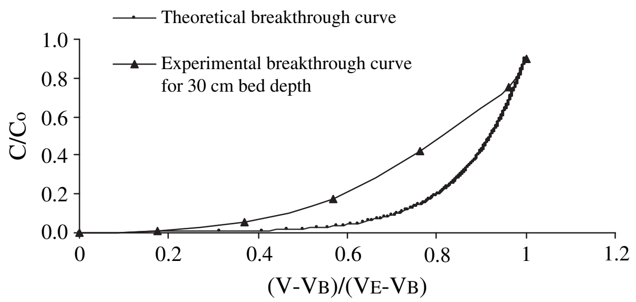

Fig. 8. Experimental and theoretical breakthrough curve of CV removal by SMA packed column.

Langmuir model, but originally it was not so (the R^2 value for liner fit was 0.9513).

3.5. Regeneration of exhausted SMA

CV could be desorbed from exhausted alumina surface using aqueous NaOH, rectified spirit and acetone. It was observed that when 4 g of exhausted SMA was shaken with 20 ml (i.e. 200 g/l) of 1 M NaOH for 1 h at a temperature of 25 °C, 90. 2% of CV and around 95% SDS were desorbed from exhausted SMA. When rectified spirit and acetone was tried separately on the exhausted SMA it was observed that neither rectified spirit nor acetone desorbed SDS from the exhausted SMA but they could desorb CV only. This is because the pH of the solution was lower than Z_{pc} (9.15). In these cases, 2 g of exhausted SMA was shaken with 8 ml of organic solvent (i.e. 250 g/l) for 60 min at a temperature of 25 °C. The regeneration efficiency was found to be 86% and 65% for rectified spirit and acetone, respectively. The regenerated CV could be collected and reused. The organic solvent could be distilled off using a Soxhlet apparatus and the same solvent could be recycled for desorption i.e. for regenerating SMA from CV-adsorbed SMA. Regenerated SMA was again used for CV removal and was found to have the efficiency in the range of 58–70% (whereas the removal efficiency was 99% using fresh SMA). Rectified spirit is a better choice for regenerating SMA.

4. Conclusion

SMA was found to be very efficient media for the removal of dye from wastewater. The column with 2 cm diameter, and having bed depths of 10, 20 and 30 cm could treat 3.5817, 7.9458 and 12.5103 l, respectively of CV bearing wastewater (CV concentration 200 mg/l) at breakthrough. The adsorption bed was exhausted within a very short time (~1.5 h) after the breakthrough occurred, which indicated that the adsorption zone was almost saturated at the time of exhaustion. The height of the adsorption zone was 1.41 cm and the rate at which it moved its own height was found to be 1.15 cm/h. The bed was almost 95% saturated at the time of breakthrough. The adsorption rate constant (K) and adsorption capacity (N_0) and minimum bed depth (x_0) were found to be 0.019228 1/mg-h, 28410 mg/l and 2.05 cm, respectively. Effect of flow rate and initial concentration was studied. The increase (or decrease) in flow rate decreased (and increased) the breakthrough time. The initial CV concentration also had the similar effect. Theoretical breakthrough curve was drawn from the batch isotherm data and it followed the same pattern of experimental breakthrough curve. Desorption of CV from the SMA surface was possible using 1 M sodium hydroxide solution, rectified spirit and acetone.

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