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Fixed bed column study for Cd(II) removal from wastewater using treated rice husk

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Abstract

A fixed bed of sodium carbonate treated rice husk was used for the removal of Cd(II) from water environment. The material as adopted was found to be an efficient media for the removal of Cd(II) in continuous mode using fixed bed column. The column having a diameter of 2 cm, with different bed depths such as 10, 20 and 30 cm could treat 2.96, 5.70 and 8.551 of Cd(II) bearing wastewater with Cd(II) concentration 10 mg/l and flow rate 9.5 ml/min. Different column design parameters like depth of exchange zone, adsorption rate, adsorption capacity, etc. was calculated. Effect of flow rate and initial concentration was studied. Theoretical breakthrough curve was drawn from the batch isotherm data and it was compared with experimental breakthrough curve. An amount of 0.01 mol/l HCl solution was used for desorption of adsorption column. Column regeneration and reuse studies were conducted for two cycles of adsorption–desorption.

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Keywords: Cadmium; Sodium carbonate treated rice husk (NCRH); Adsorption; Column study; Breakthrough curve

1. Introduction

Cadmium is a metal widely used in industries such as cadmium plating, alkaline batteries, copper alloys, paints and plastics. It is a non-essential and non-beneficial element to plants and animals. Its toxic effects are well documented. Diseases such as renal damage, anemia, hypertension and itai-itai are associated with excess cadmium [1]. Hence it is important to eliminate trace of cadmium from drinking water, or to remove cadmium from wastewaters before they are discharged into receiving bodies. Various treatment techniques have been employed to eliminate or reduce cadmium in wastewater including precipitation, adsorption, ion exchange and reverse osmosis. As of now, adsorption by activated carbon is accepted to be the best available technology for the reduction of heavy metals, except that its manufacturing cost is quite high. Hence, a search is on world wide for a low-cost alternative. Research in the recent years has indicated that some natural biomaterials including agricultural products and by-products can accumulate high concentration of heavy metals. Adsorbent generated from these biomass are

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cost effective and efficient. Low-cost agricultural products and by-products have been reported to be effective in removing cadmium. Corncob [2], Sugar beet pulp [3] and Petiolar felt-sheath of palm [4] to name a few.

Rice husk, an abundant biomaterial, is capable of removing heavy metals and can be considered as an efficient and low-cost adsorbent for heavy metals. The earlier studies on rice husk has also been reported to be effective in removing cadmium, such as rice husk ash [5], phosphate-treated rice husk [6], rice husk [7] and sodium carbonate treated rice husk (NCRH) [8]. Most of the studies mentioned above have been conducted in batch mode, which are usually limited to the treatment of small quantities of wastewater. The sorption capacity parameter obtained from a batch experiment is useful in providing information about effectiveness of metal-biosorbent system. However, the data obtained under batch conditions are generally not applicable to most treatment system (such as column operations) where contact time is not sufficiently long for the attainment of equilibrium [9]. Hence, there is a need to perform equilibrium studies using columns. Volesky and Prasetyo [10] studied the cadmium removal by Ascophyllum nodosum in a biosorption column. Column studies for the adsorption of chromium(VI) using sphagnum moss peat have been reported by Sharma and Foster [11].

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Nomenclature

a, a_1, a_2	slope (h/cm)
$b, b_1 b_2$	intercept (h)
cb	effluent concentration at influent concentration C_1
	(mg/l)
c_{f}	effluent concentration at influent concentration C_2
	(mg/l)

- C effluent concentration at any time t (mg/l)
- $C_{\rm B}$ desired concentration of solute at breakthrough (mg/l)
- *C*_e equilibrium concentration (mg/l)
- $C_{\rm E}$ concentration of solute at exhaust point (mg/l)
- C_0 initial concentration (mg/l)
- C^* equilibrium concentration of solute in solution corresponding to an adsorbed concentration, q_e (mg/l)
- $F_{\rm w}$ wastewater flow rate (l/h)
- *h* total bed depth (cm)
- $h_{\rm z}$ height of exchange zone (cm)
- *K* adsorption rate constant (l/mg h)
- $K_{\rm F}$ constant in the Freundlich isotherm model
- $K_{\rm m}$ overall mass transfer coefficient (cm²/min)
- *m* weight of adsorbent (g) NCRH sodium carbonate treated rice husk
- N_0 adsorption capacity (mg/l)
- $q_{\rm e}$ adsorption capacity (mg/g)
- *t* service time of column (h)
- *V* linear flow velocity of feed to bed (cm/h)
- *V*_B total volume of wastewater treated to the point of breakthrough (l)
- $V_{\rm E}$ total volume of wastewater treated to the point of exhaustion (l)
- *x* amount of adsorbate adsorbed (mg)
- *X* bed depth of column (cm)
- X_0 minimum column height necessary to produce an effluent concentration C_B (cm)
- *1/n* constant in the Freundlich isotherm model representing intensity of sorption

Reports regarding the removal of pollutants in continuous mode using fixed bed column are only a few [7,9–15]. Thus, the present study aims towards the removal of Cd(II) from Cd(II) bearing wastewater in fixed bed column filled with sodium carbonate treated rice husk (NCRH). The present research work basically deals with the design parameters of fixed bed adsorption column, so that it may find practical application to the real world.

2. Materials and methods

2.1. Reagents

All chemicals used were of analytical grade (E. Merck, India). Stock solutions of 1000 mg/l was prepared for Cd(II) using cadmium nitrate. In order to avoid hydrolysis as well as high adsorption of species in the flask wall the stock solution was prepared with HNO₃ 2% (v/v), which were diluted with distilled water to prepare working solutions. pH of the working solutions were adjusted to 6.0 ± 0.2 for all studies using dilute NaOH solution.

2.2. Instrumentation

A atomic absorption spectrophotometer (AA-6650, Shimadzu, Japan) was used for Cd(II) measurement. A high precision electrical balance (Sartorious GMBH) was used for weighing and a digital pH meter (DHP-500, SICO, India) was used for pH measurement. A peristaltic pump (Miclins India Limited, PP 30) was also used for providing constant flow of metal and desorbing solution in fixed bed column.

2.3. Preparation of NCRH

Fresh rice husk was obtained from a local rice mill and was passed through different sieve size. The fraction of particle between 425 and 600 μ m (geometric mean size: 505 μ m) was selected [8]. Rice husk was washed thoroughly with distilled water and was dried at 60 °C. This was treated with 0.1 mol/l sodium carbonate solution at room temperature for 4 h. Excess of sodium carbonate was removed with water and the material was dried at 40 °C. Sodium carbonate treated rice husk was designated as NCRH [8].

2.4. Experimental methods

Fixed bed column studies were conducted using columns of 2 cm diameter and 55 cm length. The column was packed with NCRH between two supporting layers of preequilibrated 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 Cd(II) bearing wastewater in the up flow mode with a volumetric flow rate of 9.5 ml/min (\sim 1.81 m³/m²/h). The initial concentration of Cd(II) was 10 mg/l. The samples were collected at certain time intervals and were analysed for Cd(II) using atomic absorption spectrophotometer (AA-6650, Shimadzu). Experiment was also conducted with initial Cd(II) concentration of 20 mg/l keeping a bed depth of 10 cm and flow rate of 9.5 ml/min. Effect of flow rate was also studied, with flow rates of 5 and 15 ml/min, with the bed depth of 10 cm and initial Cd(II) concentration of 10 mg/l. The temperature was 28 ± 2 °C and the pH was 6.0 ± 0.2 for all studies. This was in accordance with the earlier study [8]. The optimum pH should be within the adsorption edge and should have the lowest solution pH for maximum metal ion adsorption and also should be near to pH of the solution prepared with metal salts. It is also well known that the pH of wastewater is generally in a slightly acidic range. It should also be such that the precipitation of metal ions should not occur.

After exhaustion of the NCRH by Cd(II) it is necessary to regenerate it for further use. The column desorption was studied by using 0.01 mol/l HCl (1200 ml) and the column were regen-



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

erated and reactivated by washing the bed with distilled water (700 ml) followed by 0.01 mol/l NaOH (650 ml) and then again washing with distilled water (350 ml).

3. Results and discussion

3.1. Behavior of adsorption column

The column experiments were conducted with 10 mg/l Cd(II) solutions at bed depths of 10, 20 and 30 cm, respectively maintaining a constant flow rate of 9.5 ml/min. The breakthrough curves of S-shaped as obtained were shown in Fig. 2. The break-



Fig. 2. Breakthrough curves for Cd(II) at bed depth of 10, 20 and 30 cm.

Table 1							
Volume of effluent	treated and	the mass	of NCRH	required	upto	breakth	rough

Bed depth (cm)	10% Breakthrough time (h)	Treated volume (l)	Total mass of NCRH (g)	Mass of NCRH per liter (g/l)
10	5.2	2.96	8.39	2.83
20	10	5.70	16.78	2.94
30	15	8.55	25.16	2.94

through times and the exhaust times corresponds to $C/C_0 = 0.1$ and 0.9, respectively. The volume of metal effluent treated and the requirement of NCRH upto breakpoint have been shown in Table 1. It was observed that time to achieve breakthrough was increased with the increase of bed depths. The presence of carboxylate, siloxane and lignin groups in the surface adsorbent were responsible for adsorption of Cd(II).

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 design approach, bed depth service time (BDST) approach based on Bohrat and Adams [16] equation is widely used. The equation of Bohrat and Adams, which is based on surface reaction rate theory, can be represented as follows:

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

Since $e^{KN_0(X/V)}$ is usually much greater than unity Eq. (1) can be simplified to

$$\ln\left(\frac{C_0}{C_{\rm B}} - 1\right) = \ln e^{KN_0(X/V)} - KC_0t \tag{2}$$

Solving the above equation for t

$$t = \frac{N_0}{C_0 V} X - \frac{1}{C_0 K} \ln\left(\frac{C_0}{C_B} - 1\right)$$
(3)

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

The form of the Bohart–Adams equation, shown as Eq. (3) 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. (3) for X yields

$$X_0 = \frac{V}{KN_0} \ln\left(\frac{C_0}{C_{\rm B}} - 1\right) \tag{4}$$

where X_0 is the minimum column height necessary to produce an effluent concentration C_B , also known as critical bed depth.

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. Hutchins [17] presented a modification of Bohart–Adams equation, which requires only three fixed bed tests to collect the necessary data instead of nine columns. This is called the bed depth service time (BDST) approach. The Bohart–Adams Eq. (3) can be expressed as

$$t = aX + b \tag{5}$$

where

$$a = \text{slope} = \frac{N_0}{C_0 V} \tag{6}$$

and

$$b = \text{intercept} = -\frac{1}{\text{KC}_0} \ln\left(\frac{C_0}{C_B} - 1\right)$$
(7)

In order to develop a BDST correlation, the data of breakthrough curves plotted for each bed depth of 10, 20 and 30 cm are used to plot a BDST correlation by recording the operating time to reach a certain removal at each bed depth. The slope of the BDST line is equal to the reciprocal velocity of the adsorption zone and the intercept on abscissa is the critical depth defined as the minimum bed depth required to obtain the desired effluent quality at time zero. If the adsorption zone is arbitrarily defined as the adsorbent layer through which the effluent concentration varies from 90 to 10% of the feed concentration, then this zone is defined as the horizontal distance between these two lines in the BDST plot. The BDST plot of Cd(II) is shown in Fig. 3.

The depth of adsorption zone also known as mass transfer zone (MTZ) was obtained as 12 cm. From the slope and intercept of the 10% saturation line design parameters like *K* and N_0 could be found out using Eqs. (6) and (7). The minimum column height (X_0) necessary to produce an effluent concentration C_B (10 mg/l) was calculated using Eq. (4). The values of *K*, N_0 and X_0 were found to be 823.85 cm³/mg h, 0.889 mg/cm³ and 0.544 cm, respectively. The value of *K* and N_0 indicated that the NCRH is highly efficient for removal of Cd(II) from water environment.



Fig. 3. BDST plot for Cd(II).

3.3. Design of adsorption column

The column design parameters as obtained earlier could be used for the design of adsorption column in practical use. According to BDST, if the value of a is determined from one flow rate, values for other flow rates can be computed by multiplying the original slope by the ratio of the original and new flow rates, and the change of b value is insignificant with respect to the changing flow rates [17].

It is also proposed that data collected at one influent solute concentration be adjusted by 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

$$t = a_1 X + b_1 \tag{8}$$

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

$$a_2 = a_1 \frac{C_1}{C_2} \tag{9}$$

$$b_2 = b_1 \left(\frac{C_1}{C_2}\right) \frac{\ln\left(C_2/C_{\rm F} - 1\right)}{\ln\left(C_1/C_{\rm B} - 1\right)} \tag{10}$$

where a_1 is the slope at concentration C_1 , a_2 the slope at concentration C_2 , b_1 the intercept at concentration C_1 , b_2 the intercept at concentration C_2 , C_f the effluent concentration at influent concentration C_2 , C_b is the effluent concentration at influent concentration C_1 .

3.3.1. Design of adsorption column for different flow rate

Columns were run with flow rates of 5 and 15 ml/min, whereas original flow rate was 9.5 ml/min. The initial Cd(II) concentration was kept constant at 10 mg/l. The bed depth and column diameter were 30 and 2 cm, respectively. The break-through times (corresponding to 0.1 mg/l effluent concentration) were found to be 27.1 and 8.7 h for 5 and 15 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 0.931 and



Fig. 4. Breakthrough curves of Cd(II) removal by NCRH packed column for different flow rate.



Fig. 5. Breakthrough curves of Cd(II) removal by NCRH packed column for different initial concentration.

0.310 for 5 and 15 ml/min flow rate, respectively. The value of intercept was 0.2667. From these vales of a and b, the service times for 30 cm column were calculated and these are 27.67 and 9.03 h for 5 and 15 ml/min, respectively. These values were comparable with the experimental values.

3.3.2. Design of adsorption column for different initial concentration

Column was run also with initial Cd(II) concentration of 20 mg/l (whereas original Cd(II) concentration was 10 mg/l). The flow rate was kept at 9.5 ml/min. The bed depth and column diameter were 10 and 2 cm, respectively. The breakthrough time was found to be 2.45 h (Fig. 5). The effluent concentration was taken as 1 for 10 mg/l initial Cd(II) concentration. The values of a_2 and b_2 calculated from Eqs. (9) and (10) were 0.245 and 0.133, respectively. From these vales of a and b, the service time for 10 cm column was calculated and this was 2.58 h. This value was comparable with the experimental values.

3.3.3. Design of numbers of stages and its bed depth

In order to develop a BDST correlation, one can also use three columns of equal depth (10 cm) operated in series instead of three column of bed depth 10, 20 and 30 cm.

For a multistage system, the number of stages and the depth in each stage are related to the depth of the adsorption zone by the following equation [18]:

$$N = \frac{D}{d} + 1 \tag{11}$$

where N is the number of stages (columns) in series, D the depth of adsorption zone, d is the bed depth of single stage.

The depth of single stage (column) was kept fixed at 10 cm, as explained earlier than the numbers of stages in series were obtained as 2.2. This gives an idea to design the number of stages in series and the bed depth of its each single stage. For example, three stages (columns) of equal bed depth of 6 cm can be used.

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 was generated with the initial concentration of 10 mg/l, following the concepts of Michaels [19] as follows:

(1) The equilibrium line was prepared using Freundlich adsorption isotherm

$$q_{\rm e} = K_{\rm F}(C_{\rm e})^{1/n} \tag{12}$$

$$\therefore q_{\rm e} = \frac{x}{m} = 7.563 (C_{\rm e})^{0.1936}$$
(13)

where q_e is the amount of adsorbate adsorbed (*x*, mg) per unit weight of adsorbent (*m*, g) and C_e is the equilibrium concentration of the adsorbate remaining in solution (mg/l). The equilibrium and operating lines were then plotted on an arithmetic graph as shown in Fig. 6.

- (2) An operating line was drawn which is passing through the origin and through the point given by the co-ordinates C_0 and $(x/m)_0$. The significance of this operating line was that the data of continuously mixed batch reactor (CMBT) 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 [19].
- (3) According to Weber [20], the rate of transfer of the adsorbate from the solution over a differential depth of column d*h* is given by

$$F_{\rm w} \mathrm{d}C = K_{\rm m} (C - C^*) \mathrm{d}h \tag{14}$$

where F_w is the flow rate, K_m the overall mass transfer coefficient which includes the resistances offered by film and pore diffusions, C_e is the equilibrium concentration of the adsorbate in the solution corresponding to a adsorbed concentration (*x/m*) and *C* is the concentration of the adsorbate at a given instant of time *t*.

The term $(C - C^*)$ is the driving force for adsorption and is equal to the difference between the operating line and



Fig. 6. Plot of equilibrium and operating lines for determination of theoretical breakthrough curve of Cd(II).



Fig. 7. Curve to evaluate $\int_{C_B}^{C_E} dC/(C - C_e)$ for determination of theoretical breakthrough curve of Cd(II).

equilibrium curve at any given (x/m) value. Integrating the Eq. (14) and solving for the height of adsorption zone at saturation

$$h_{\rm z} = \frac{F_{\rm w}}{K_{\rm m}} \int_{C_{\rm B}}^{C_{\rm E}} \frac{\mathrm{d}C}{(C - C^*)}$$
(15)

where $C_{\rm B}$ and $C_{\rm E}$ are the concentrations of adsorbate in effluent at breakthrough and at exhaustion, respectively.

(4) The plot of $(C - C^*)^{-1}$ versus *C* is shown in Fig. 7. The area under the curve represented the value of the above integration.

Since $(C - C^*)^{-1}$ approaches infinity as *C* approaches C_0 , It is necessary to terminate the plot of *C* at a value somewhat less than C_0 . Generally, a value of $C_E = 0.90C_0$ is selected, which is frequently considered to be the point of exhaustion for adsorption beds. For any value of *h* less than h_z corresponding to a concentration *C* between C_B and C_E , Eq. (15) can be written as

$$h = \frac{F_{\rm w}}{K_{\rm m}} \int_{C_{\rm B}}^{C} \frac{\mathrm{d}C}{(C - C^*)} \tag{16}$$

dividing Eq. (16) by Eq. (15) results in

$$\frac{h}{h_z} = \frac{\int_{C_B}^{C} dC/(C - C^*)}{\int_{C_B}^{C_E} dC/(C - C^*)} = \frac{V - V_B}{V_E - V_B}$$
(17)

 h/h_z is equal to the ratio $V - V_B/V_E - V_B$. Where V_B and V_E are total volume of water treated till breakthrough and up to the point of exhaustion, respectively and *V* is the volume of water treated within V_E for an effluent concentration *C* within C_E .

(5) The values of $(V - V_B)/(V_E - V_B)$ were calculated by dividing the values of $\int_{C_B}^{C} dC/(C - C^*)$ by the value of $\int_{C_B}^{C_E} dC/(C - C^*)$. Finally the theoretical breakthrough curves that were generated by plotting $(V - V_B)/(V_E - V_B)$ versus (C/C_0) , are shown in Fig. 8. The experimental and theoretical breakthrough curves followed the same trend.



Fig. 8. Theoretical and experimental breakthrough curve of Cd(II).

3.5. Column regeneration and reuse studies

The column with a bed depth of 10 cm was selected for this study, for which the adsorption breakthrough curve has been shown in Fig. 2. It was observed that at about 13 h the column was exhausted. The extra Cd(II) solution inside the column was pumped out leaving only solid NCRH loaded with Cd(II). Desorption was carried out by 0.01 mol/l HCl solution through the bed in the downward direction at a flow rate of 8.5 ml/min, slightly less that the sorption flow rate 9.5 ml/min so that volume of regenerant is less which helps in easy handling and high in concentration so that economical metal recovery is possible. The regenerations were carried in the counter-current mode, that is, in the downflow mode. Counter-current operation generally reduces regeneration costs and regenerant volume and increases effluent quality [21]. The concentration of Cd(II) was monitored after different time interval as shown in Fig. 9. It was observed that desorption cycle took 1 h, after which further desorption was negligible. The total volume of this eluent at 1 h was 510 ml. The maximum concentration of Cd(II) was obtained at a contact time of 7 min and recorded as 328 mg/l, which is 32.8 times higher than influent Cd(II) concentration. The eluting solution was low



Fig. 9. First cycle desorption profile of Cd(II).

in volume and high in concentration, which could help in easy handling and recovery and reuse of Cd(II).

The performance of FBR was also studied for second adsorption-desorption cycles. The desorbed column was washed with 500 ml distilled water at a flow rate of 35 ml/min followed by reactivation with 0.01 mol/l NaOH with a flow rate of 10 ml/min and finally washing it again with 500 ml distilled water at a flow rate of 35 ml/min, all in upflow direction of the column. The reactivated and regenerated column was reused for second cycle of adsorption-desorption of Cd(II) keeping the parameters same as above, for first cycle. In the first cycle 45.9 mg of Cd(II) was adsorbed from which 44.5 mg could be desorbed, indicating an excellent desorption efficiency of 97%. The performance of the reactivated and regenerated column, that is, second cycle was also highly efficient in adsorption and desorption, where, out of 43.5 mg adsorbed, 41.5 mg i.e. about 95.4% Cd(II) was desorbed. The results indicated that the sorption process for Cd(II) uptake is almost reversible without loss of binding efficiency.

The mineral acids are proton-exchange agents, which dislodge high valance metal ions from biomass [22]. Excessive amount of H^+ ions can reduce the metal sorptive capacity. Washing the biomass with deionized water can remove H^+ ions and can be regenerated. It was observed that NCRH regenerated with NaOH regained its initial metal removal capacity to a good extent and the efficiency was increased as compared to first method, where the desorbed NCRH was only regenerated by washing with distilled water. Yan and Viraraghavan [22] observed similar phenomena and indicated that biomass could be repeatedly subjected to alkaline treatment without losing its adsorption properties.

4. Conclusions

NCRH was found to be efficient media for the removal of Cd(II) from wastewater. The column with 2 cm diameter, and bed depth 10, 20 and 30 cm could treat 2.96, 5.70 and 8.551 of Cd(II) bearing wastewater (Cd(II) concentration 10 mg/l) at breakthrough. About 2.94 g of NCRH was required per liter of Cd(II) treatment. The depth of adsorption zone was 12 cm. The adsorption rate constant (K) and adsorption capacity (N_0) and minimum bed depth (X_0) were found to be 823.85 cm³/mg h, 0.889 mg/cm^3 and 0.544 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 Cd(II) 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 Cd(II) from the NCRH surface was possible using 0.01 mol/l HCl solution. The performance of the reactivated and regenerated column (with 0.01 mol/l NaOH and distilled water) was highly efficient in adsorption and desorption.

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