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Equilibrium, kinetic and thermodynamic study of acid yellow-34 adsorption onto *Cedrus deodara* sawdust

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ABSTRACT

Sawdust of *Cedrus deodara* was explored as a biosorbent for the removal of acidic textile dye, acid yellow-34. The process was pH sensitive and maximum sorption (92%) was achieved at pH 3 in 12-h agitation time using 750 mg of sawdust for 50 mL of dye solution. The sorption data followed the Freundlich, Dubinin–Radushkevich (D–R) and Langmuir isotherms. Freundlich constants $1/n = 0.890 \pm 0.039$ and $C_e = 51.70 \pm 2.25$ mmol g^{-1} have been estimated. Sorption capacity, $X_m = 0.62 \pm 0.35$ mmol g^{-1}, $\beta = -1.395 \pm 0.569$ kJ mol^{-1} and adsorption energy, $E = 0.60 \pm 0.03$ kJ mol^{-1} have been evaluated using D–R isotherm. The Langmuir constants $Q = 0.0573 \pm 0.0051$ μmol g^{-1} and $b = 1.076 \pm 0.007$ dm^3 mol^{-1} are calculated. Our results show that sorption varies with temperature yielding thermodynamic parameters $\Delta H = -39.00 \pm 2.57$ kJ mol^{-1}, $\Delta S = -116.29 \pm 8.54$ J mol^{-1} K^{-1} and $\Delta G = -456.54 \pm 2.23$ kJ mol^{-1} at 298 K. The negative value of enthalpy and free energy reflect the exothermic and spontaneous nature of sorption, respectively. Our results also show that sorption decreases in the presence of oxalate, bicarbonate, carbonate and Ca^{2+} ions.

Keywords: Wastewater; Acid yellow-34; Textile dye; Biosorption

1. Introduction

More than 0.1 million commercial dyes are available in the market [1,2]. Chemically, dyes include conjugated rings and groups in their structure, which impart different colours. Dyes are extensively used in textile industry for dyeing and are generally used in aqueous solutions.

About 10–15% of dyes are lost during the dyeing process and are discharged as effluents that go to water bodies such as streams, lakes and rivers. They severely affect the aesthetic quality of water and reduce its recreational use. Depending on the exposure time and dye concentration, dyes can have acute or chronic effects on exposed organisms causes diseases such as nausea, allergy, ulceration, haemorrhage, irritation in respiratory tract and eyes, dermatitis and perforation of nasal septum, cancer and mutation [3–6].

Dyes are difficult to remove from wastewater using the tradition methods such as flocculation, precipitation, electro-kinetic coagulation, membrane filtration, electrochemical destruction and ozonation due to their complex structure [7]. Sorption onto activated carbon is a popular method, used widely all over the world for the removal of textile dyes but it is expensive [8,9] and not feasible in the developing countries where the ineffectiveness of the implementation of the

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environmental laws is observed. The use of low cost biosorbents gained popularity in recent years and is effectively used for pollution abatement [10–14], which can also be used for the cleaning of dyes from water.

Textile industry is one of the major industries of Pakistan. It contributes 46% of total industrial manufacturing in Pakistan and employees 38% of total labour. It is necessary to attract the industrialists for their role in the pollution control and sustainable development. The development of a cheaper method, especially according to the demands of an underdeveloped country like Pakistan is the utmost requirement of the day to save the precious commodity of water. Keeping this in mind, the sawdust of *C. deodara* which is easily available and has a lower cost is used as an adsorbent in this study for the abatement of textile dyes from water.

2. Materials and methods

2.1. Dye

Textile dye acid yellow-34 was selected to study its pre-concentration behaviour from aqueous solutions. Its chemical formula is $C_{16}H_{12}ClN_4NaO_4S$, IUPAC name is "sodium-4-chloro-3-(5-hydroxy-3-methyl-4-phenylazopyrazol-1-yl) "benzenesulphonate" and structure is given in Fig. 1.

2.2. Sorbent

The sawdust of *C. deodara* with known properties [10] was obtained from a local sawmill in Vehari city, Pakistan, at a price of PKR 20 per kg. The sawdust was sieved and 40 mesh size (0.42 mm Φ) was separated and washed thoroughly with distilled water and dried in an oven overnight at 105˚C and used as sorbent.

2.3. Chemicals and reagents

Analar grade chemical was used throughout this study. Buffer solution of pH 1–3 was prepared by mixing 0.1 M solution of KCl with 0.1 M solution of HCl, and buffer of pH 4–6 was prepared by mixing 0.1 M solution of CH_3COONa and 0.1 M solution of CH_3COOH.

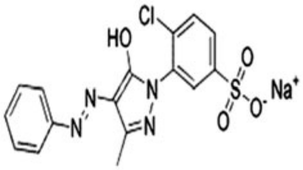

Fig. 1. Structure of dye acid yellow-34.

2.4. Dye determination

A UV–visible spectrophotometer, model UV-1800 made by Shimadzu was used for the determination of dye. The maximum absorbance (λ-max) was noted by sorbate solution at 410 nm, and all the experiments were performed at this wavelength.

2.5. Sorption measurements

Sorption was measured in terms of difference between absorbance before and after shaking the dye solution with sorbent. The following equation was used for this purpose:

$$\% \text{ Sorption} = \frac{A_0 - A}{A_0} \tag{1}$$

where "A_0" is the amount of dye before shaking and "A" is the amount of dye after shaking, and the distribution coefficient "K_d" was calculated as:

$$K_d = \frac{\text{Amount of dye sorbed}}{\text{Amount of dye in solution}} \times \frac{\text{Volume of solution (V)}}{\text{Weight of sorbent (W)}} = (\text{cm}^3/\text{g}) \tag{2}$$

The % sorption and the distribution coefficient can be correlated as:

$$\% \text{Sorption} = \frac{100\, K_d}{K_d + V/W} \tag{3}$$

3. Results and discussion

3.1. Effect of pH

The selection of electrolyte plays an important role in separation system. The pH of a system may increase or decrease the rate of sorption process. Initially, the sorption was tested in deionized water but no appreciable sorption was occurred. Therefore, the effect of buffer on sorption was studied. Sorption increased as the pH of buffer solution increased up to pH 3. Then, it showed slight decrease from pH 4–6. And at pH 7 and above, there was no significant sorption. The results are shown in Fig. 2. The increase in sorption of the dye up to pH 3 is due to the acidic nature of the dye. At lower pH, it oxidizes well and it is expected that it should adsorb up to the maximum extent but this is not the situation. The reason is that H^+ ions are in competition with the dye. The smaller size and abundance of H^+ ion affects the sorption of dye from aqueous solution. The sorption at pH 3 should be due to the cellulose where site binding sorption occurs. It may also be due to surface complexation phenomenon facilitated by dissociation of acidic functional groups, i.e. carboxyl, hydroxyl and carbonyl presented on sawdust surface [10]. At slightly higher pH, it adsorbs well. The H^+ ion competition is reduced and the dye is adsorbed easily, while at higher pH, the surface activation due to acidic dissociation of the functional groups is reduced. pH 3 was selected as an optimum pH for further studies. Our results are in agreement with the earlier reported results of the sorption of acid green-25 and acid red-14 onto untreated sawdust of Nara wood occurred at pH 2 [15] and sorption of acid blue-25 onto *Shorea dasyphylla* sawdust at pH 2 that decreased with the increase in pH values [16].

3.2. *Influence of amount of sorbent*

The sorption was studied as a function of amount of sorbent in the range of 0.5–5 g from buffer of pH 3. The results are presented in Fig. 3. Initially, sorption increased with the increase in amount of sawdust because greater number of sorbent particles provides greater surface area. Maximum sorption took place at 0.75 g of sorbent but when the amount of sorbent was increased beyond 0.75 g, sorption decreased gradually. This was due to the fact that as the amount of sorbent increased, particles of sorbent coagulated with each other and decreased the surface area available for sorbate molecules or ions to adsorb, and at the same time, the effective distribution of sorbent in the medium was not possible. Increase in the sorption of acidic dyes with increase in amount of adsorbent is also reported earlier [17].

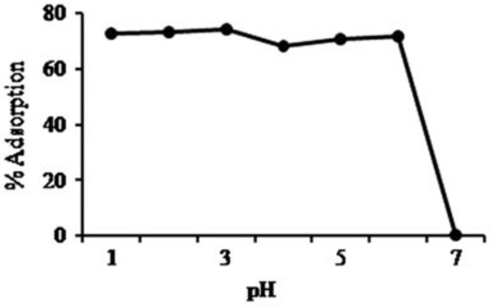

Fig. 2. Effect of pH on the sorption of acid yellow-34 onto sawdust of *C. deodara*.

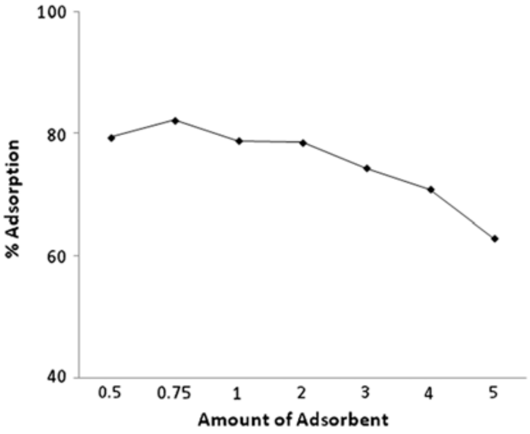

Fig. 3. Effect of amount of sorbent on sorption of acid yellow-34 onto sawdust of *C. deodara*.

3.3. *Effect of shaking time*

Removal of dyes from water is greatly influenced by its shaking time because it affects the distribution of sorbate between sorbent and electrolyte. Shaking time was varied from 4 to 12 h. The sorption gradually increased with increasing agitation time. The increase in sorption with the increase in shaking time was up to 10 h. Beyond that the sorption remained constant. 12-h shaking time was considered optimum and used for further experiments. Sorption of acid black-1 onto potato peel was also reported with similar trend [17].

Results obtained from sorption data were also analysed through Morris–Webber equation [18] which is given as:

$$q_t = R_D\sqrt{t} \tag{4}$$

where "q_t" is the adsorbed concentration at time "t", and "R_D" is the rate constant of intra-particle transport. By plotting q_t vs. $t^{1/2}$, a straight line with a correlation coefficient of 0.971 was obtained which shows that Morris–Webber equation was followed by sorption data. Sorption of acid red-14 and acid green-25 onto untreated sawdust of Narra wood also follows Morris–Webber equation with a correlation coefficient of 0.976 and 0.991, respectively [15].

Reichenburg equation [19] was also applied to sorption data to know the mechanism of sorption either it takes place by film diffusion or by intra-particle diffusion mechanism. It was tested as:

$$F = \left(1 - \frac{6}{\pi^2}\right) e^{-B_t} \tag{5}$$

where "$F = q/q_e$" and "B_t" is a mathematical function of "F" which can be calculated for each value of "F" as:

$$B_t = -0.4977 \ln(1 - F) \tag{6}$$

A plot of "B_t" vs. "t" was a straight line with a correlation coefficient of 0.961, which confirmed that sorption took place through intra-particle diffusion mechanism.

There is a correlation between "q", the number of molecules adsorbed and "t" the time required to complete the sorption process is shown by the Lagergren relationship [20], represented as:

$$\log(q_e - q_t) = \log q_e - \frac{kt}{2.303} \tag{7}$$

where "q_t" is the amount adsorbed at time "t", "q_e" is the amount adsorbed at equilibrium and "k" is the rate constant. The graph of "$\log(q_e - q_t)$ vs. t" was a straight line indicating that the process of sorption is first order with respect to adsorbed concentration. Such trend is also reported for sorption of acid red-37 and acid blue-25 onto chitosan [21]. The straight line of Reichenburg and Lagergren plots was not passed through the origin indicating that the sorption is film diffusion controlled at initial stages and controlled by particle diffusion when sorbent becomes loaded with sorbate.

3.4. Effect of sorbate concentration

Sorption of acid yellow-34 onto sawdust was studied in the concentrations range of 10–50 ppm using the optimal conditions of 0.75 g of sorbent, buffer of pH 3 and shaking time of 12 h. It was found that sorption increased with the increase in concentration of the sorbate. The results are shown in Fig. 4. This might be due to the availability of maximum sorption sites. Similar results are reported for the sorption of acid green-25 and acid red-16 onto sawdust of Narra wood [15].

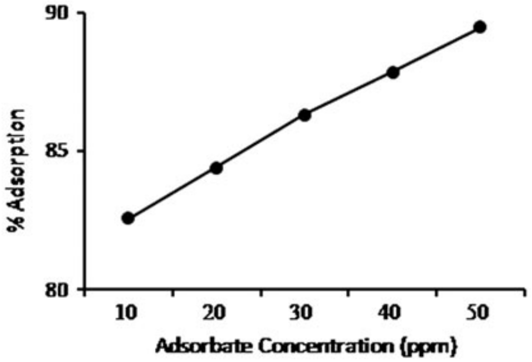

Fig. 4. Effect of acid yellow-34 concentration on its sorption onto sawdust of *C. deodara*.

The sorption data were subjected to Freundlich, Dubinin–Radushkevich and Langmuir isotherms. All the isotherms were obeyed over the entire range of concentration studied. The experimental results were fitted to the Freundlich isotherm which gave an empirical expression encompassing the surface heterogeneity, exponential distribution of active sites and infinite surface coverage. The Freundlich isotherm [22] was followed over the entire range of concentration applied in its linearized form.

$$\log C_{\mathrm{ads}} = \log C_{\mathrm{m}} + \frac{1}{n} \log C_{\mathrm{e}} \tag{8}$$

where C_e is the amount of sorbent in solution and C_{ads} is the amount of dye on sorbent while $1/n$ is Freundlich constant. When log C_{ads} plotted against log C_e, a straight line with a correlation coefficient of 0.999 was obtained (Fig. 5). The values of $1/n = 0.890 \pm 0.039$ the intensity of sorption, and $C_e = 51.70 \pm 2.25$ mmol g^{-1} the maximum sorption capacity were computed from the slope and intercept of the figure. The fractional value ($0 < 1/n < 1$) of the constant $1/n$ shows the

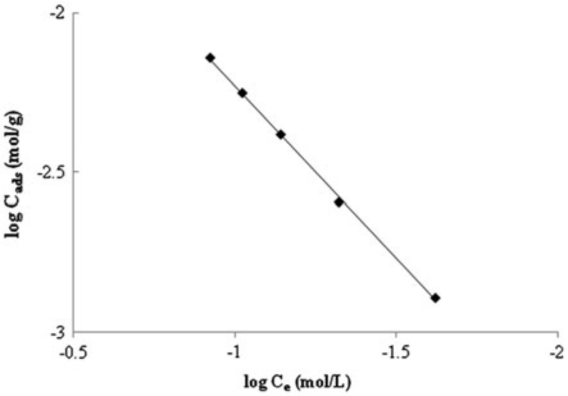

Fig. 5. Freundlich sorption isotherm of acid yellow-34 onto sawdust of *C. deodara*.

heterogeneous nature of the surface. The higher the fractional value, the higher the heterogeneity of the surface and vice versa. It is deduced from the higher value of constant $1/n$ that the surface of sawdust is heterogeneous in nature. It confirms the earlier results of sorption of Zn(II) ions onto sawdust ($1/n = 0.493$) [10] and sorption of Cu(II) ions onto biomass [23].

Dubinin–Radushkevich (D–R) isotherm [24] was applied to determine the nature of sorption. According to this isotherm, sorption occurs within a sorption space nearest to the sorbent surface. It was tested in following linearized form and the results are presented in Fig. 6.

$$\ln C_{\mathrm{ads}} = \ln X_{\mathrm{m}} - \beta \varepsilon^2 \tag{9}$$

where X_{m} is the maximum sorption capacity, β is a constant related to energy and ε is Polanyi potential mathematically equal to:

$$\varepsilon = RT \ln\left(1 + \frac{1}{C_{\mathrm{e}}}\right) \tag{10}$$

where R is universal gas constant and T is absolute temperature. When ln C_{ads} was plotted against ε^2, the values of β and X_{m} computed from the slope and intercept of the plot are -1.395 ± 0.569 kJ mol^{-1} and 0.62 ± 0.35 mmol g^{-1}. The value of sorption energy E can be correlated to β using Eq. (11) [25]. The value of sorption energy calculated was 0.60 ± 0.03 kJ mol^{-1}.

$$E_{\mathrm{s}} = \frac{1}{\sqrt{-2\beta}} \tag{11}$$

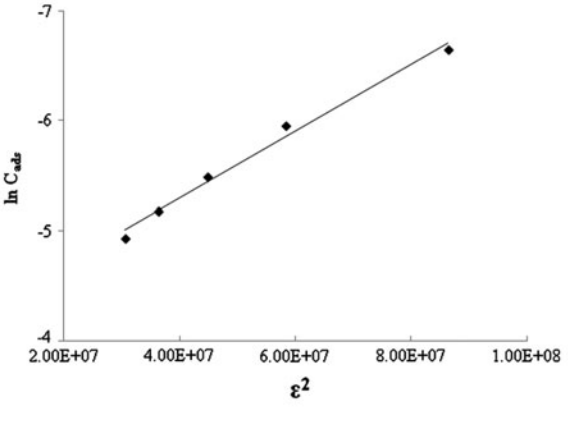

Fig. 6. D–R sorption isotherm of acid yellow-34 onto sawdust of *C. deodara*.

According to Langmuir isotherm, [26] sorption is monolayer and strength of intermolecular attractive forces decreases with increase in distance between sorbate and sorbent. The linearized form of Langmuir isotherm is presented as:

$$\frac{C_{\mathrm{e}}}{C_{\mathrm{ads}}} = \frac{C_{\mathrm{e}}}{Q} + \frac{1}{Qb} \tag{12}$$

where "C_{e}" is the amount of dye in solution, "C_{ads}" amount of dye on sorbate. "Q" and "b" are Langmuir constants. "Q" is the measure of amount of sorbate when monolayer is completed while "b" shows the sorption energy. When $C_{\mathrm{e}}/C_{\mathrm{ads}}$ was plotted against C_{e}, Langmuir model was followed over entire concentration range (Fig. 7). The Langmuir constants $Q = 0.0573 \pm 0.0051$ µmol g^{-1} and $b = 1.076 \pm 0.007$ dm^3 mol^{-1} were calculated from the slope and intercept of the plot. Sorption of Zn(II) [10] and Cu(II) ions [23] onto the sawdust and biomass, respectively, also follows Langmuir's isotherm and confirms monolayer sorption.

3.5. Effect of temperature

The effect of temperature on sorption of textile dye onto sawdust was studied over a temperature range of 10–50°C. The sorption decreased with increase in temperature. The increase in temperature provides greater amount of energy to the system and creates a situation of unrest and greater molecular movement, that is why sorption decreases with increasing temperature. Similarly, when temperature is decreased,

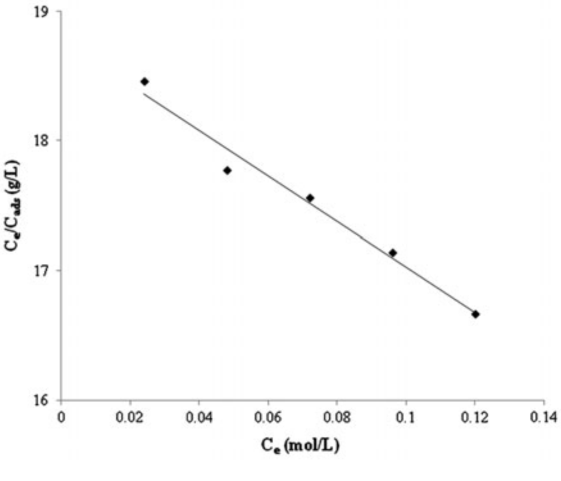

Fig. 7. Langmuir sorption isotherm of acid yellow-34 onto sawdust of *C. deodara*.

molecular movement starts to decrease and hence there is a greater chance for a molecule to deposit on the surface of sorbent and hence sorption increases.

Thermodynamic parameters such as enthalpy ΔH, entropy ΔS, and Gibbs free energy ΔG were calculated from a plot obtained by plotting log K_c against $1/K$ (Fig. 8). While, Kc = F/(1-F) where F is fraction adsorbed onto the sorbent surface at equilibrium. The thermodynamic parameters $\Delta H = -39.00 \pm 2.57$ kJ mol^{-1}, $\Delta S = -116.29 \pm 8.54$ J mol^{-1} K^{-1} and $\Delta G = -456.54 \pm 2.23$ kJ mol^{-1} were calculated from the slope and intercept of the plot using Eqs. (13) and (14).

$$\log K_c = \frac{-\Delta H}{2.303} RT + \frac{\Delta S}{2.303R} \quad (13)$$

$$\Delta G = -RT \ln K_c \quad (14)$$

Earlier results for sorption for acid blue-25 onto *S. dasyphylla* sawdust $\Delta H = -11.49$ kJ mol^{-1}, $\Delta S = 72.76$ J mol^{-1} K^{-1} and $\Delta G = -30.72$ kJ mol^{-1} are of the same magnitude [15].

3.6. Effect of foreign ions on sorption

Different anions, cations or complexing agents present in the solutions as contaminants can affect the sorption of a specific sorbate. To explore the effect of these ions on the removal efficiency of sawdust, different anions in the form of its sodium salt and cations in its chloride salts were added to the system. The salts were added in the amount of 10 mg. The anions studied were chloride, acetate, citrate, oxalate, bicarbonate and carbonate. The results are presented in Table 1. It is obvious from the results that chloride, acetate and citrate have a very small and negligible effect on sorption while oxalate, bicarbonate and carbonate have reduced the sorption up to some extent. This might be due to complexation with dye. To achieve maximum sorption, oxalate, bicarbonate and carbonate should be removed first. Like anions, effect of different cations was also studied. Different cations whose effect was studied were NH_4^+, Ni^{2+}, Cd^{2+}, Pb^{2+} Zn^{2+} and Ca^{2+}. The results are given in Table 2. It is obvious from the results that all the cations investigated have a small effect on sorption.

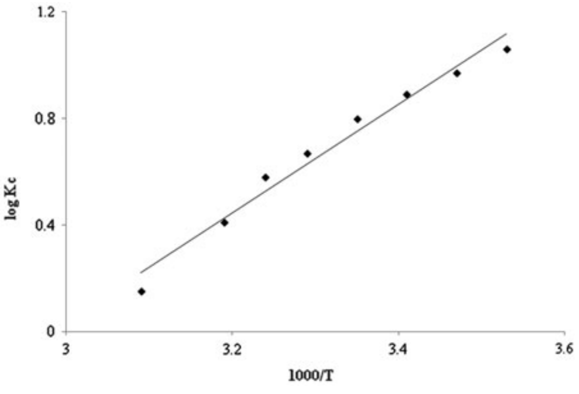

Fig. 8. Variation of sorption equilibrium of acid yellow-34 onto sawdust of *C. deodara* with temperature.

Table 1
Effect anions on the sorption of acid yellow-34 onto sawdust of *C. deodara*

Anion	% Sorption
Nil	89.49
Chloride	87.58
Acetate	87.09
Citrate	86.96
Oxalate	75.56
Bicarbonate	62.32
Carbonate	52.76

Table 2
Effect of cations on the sorption of acid yellow-34 onto sawdust of *C. deodara*

Cations	% Sorption
Nil	89.49
NH_4^+	87.78
Zn^{2+}	87.44
Ni^{2+}	86.96
Pb^{2+}	86.75
Cd^{2+}	84.98
Ca^{2+}	74.19

4. Conclusion

Our studies concluded that sawdust of *C. deodara* is a very effective and cheaper material for the removal of acidic textile dyes (acid yellow-34) from textile wastewater. The kinetic data showed that the sorption process follow first-order kinetics and intra-particle diffusion mechanism. The sorption process is pH dependent and follows Langmuir, Freundlich and Dubinin–Radushkevich (D–R) adsorption models. Sorption capacity of sorbent $C_e = 51.70 \pm 2.25$ m mol g^{-1} and sorption energy $E = 0.60 \pm 0.03$ kJ mol^{-1} are evaluated using these isotherms. The dye removal process is affected by temperature yielding thermodynamic parameters $\Delta H = -39.00 \pm 2.57$ kJ mol^{-1}, $\Delta S = -116.29 \pm 8.54$ J mol^{-1} K^{-1} and $\Delta G = -456.54 \pm 2.23$ kJ mol^{-1} at 298 K and reflects the exothermic and spontaneous nature of sorption. It is also concluded that sorption decreases in the presence of oxalate, bicarbonate, carbonate and Ca^{2+} ions, and these ions need to be removed prior to the use of sorbent. The sawdust columns can also be used for the continuous separation process.

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