Study on Reactive Blue Adsorption on Raw and Modified Wheat Straw Using Fixed-Bed Column

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Abstract

The intention of this study was to explore the efficiency and feasibility of adsorption of Reactive Blue dye (H3R) used in textile industries using Raw wheat straw (RWS) and Modified wheat straw (MWS) as a low-cost adsorbent. Wheat straw was modified using cationic surfactant (CTAB) to study the improvement of dye removal. The properties of Raw and Modified wheat straw are studied by means of Fourier transform infrared (FTIR) and scanning electron microscope (SEM) analyses to determine the functional groups and the nature of their surface. Continuous experiments were done by fixed-bed column to study the characteristics of the breakthrough curve using different bed heights and flow rates. Results showed that the breakthrough time increases with increasing bed height and decreasing flow rate, in turn results into higher removal capacity. Results also showed a higher flow rate lead a lower adsorption capacity due to insufficient residence time. Bed depth service time model (BDST), Adam-Bohart and Thomas models were used to predict the breakthrough curves and to determine the adsorption capacity of the column. The highest bed capacity of 12.95 and 32.2 mg/g for MWS was obtained using 10 mg/L, 10 cm bed height at 10 mL/min and 30 mL/min respectively.

Keywords: Adsorption, Fixed Bed, Reactive Blue, Wheat Straw

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1. Introduction

A huge amount of water is consuming in textile industry for all the stages of the manufacturing processes. The most polluted water produced from the industrial sectors is from textile plants, considering the quantity as well as the effluent composition [1]. There are a wide range of polluting substances in wastewater producing from textile industry including dyes. Color is the first contaminant and has to be treated before discharging the wastewater. Due to visibility of the small amounts of dyes it has highly effects on the aesthetic of water. The colored dye are highly toxic and it can damage the biota [2]. In general, dye is soluble in water at low concentration. There are three main categories to remove the dyes from polluted water, namely physical, chemical and biological methods [3]. Many techniques were used in the past for removal of dyes from wastewater. Most of the conventional treatment techniques are expensive. The adsorption process is effective method for treating dye-containing effluents. The advantages of this process are, easy operation, less investment, simple design, and free from or less generation of toxic substances [4]. At present, there are many researchers focus on using low cost, commercial and available materials for the adsorption of dyes. The agricultural wastes (wheat straw) are an available, renewable and less expensive as compared to other materials used as adsorbents [5]. (Annadurai et al. 2013) used banana and orange peels as a low-cost adsorbent. The results showed that the banana peel was more effective than orange peel [6]. (Robinson et al. 2002) studied the adsorption process using two low-cost adsorbents; apple pomace and wheat straw for textile dye removal [7]. (Santhy and Selvapathy 2006) studied the removal efficiency of activated carbon prepared from coir pith to remove the reactive dyes in textile industry using batch and column processes [8]. (Dizge et al. 2008) investigated the adsorption of three reactive dyes from aqueous solutions using fly ash [9]. (Hameed 2009) stated that the spent tea leaves can be used as adsorbent for the cationic dye (methylene blue) [10]. (Said et al. 2013) Removed reactive yellow 2 and reactive blue using bagasse [11]. The continuous adsorption in fixed-bed column is often desired from industrial point of view. It is simple to operate and can be scaled-up from a laboratory process. In this study we used an agricultural waste as an adsorbent with a chemical modification and compare the results.

The aim of this study is design a continuous adsorption system in order to removes of Reactive Blue dye (H3R) using Bed Depth Service Time model (BDST), Adam-Bohart model and Thomas model using locally available adsorbent which are Raw Wheat Straw (RWS), Modified Wheat Straw (MWS) and comparing the performance of (RWS) and (MWS) in decolorizing.

2. Column adsorption models

The breakthrough curve or concentration profile under the given operating conditions is the key to design a column adsorption process. The significant parameter for any sorption system is the maximum adsorption capacity. A number of mathematical models were developed to find the efficiency and applicability of the column models for large-scale operations. In this study BDST, Adams - Bohart and Thomas Models were used to analysis the behavior of adsorbent-adsorbate system. Equation 1 was used to calculate the amount of adsorbed dye $q_{ad}$ (mg) for a given flow rate, influent concentration and bed height:

$$q_{ad} = \frac{Q}{1000} \int_{0}^{t_{ad}} \frac{C_{eq}}{w} dt$$  \hspace{1cm} (1)

Where Q is the volumetric flow rate (ml/min), t is the total time taken for the flow of solution (min), and C eq is the concentration of adsorbed dye (mg/L). The equilibrium adsorption of the column $q_{eq}$ (mg/g) can be calculated as followed:

$$q_{eq} = \frac{q_{total}}{w}$$  \hspace{1cm} (2)

Where w is the amount of adsorbent in the column (g).

2.1. Bed depth service time model (BDST)

BDST is a model describes the relationship between the service time (t) and the bed depth in terms of process concentrations and adsorption parameters. Bed depth service time model is used only for the description of the first part of the breakthrough curve, i.e. up to the breakpoint or 10–50% of the saturation points [12]. The surface reaction between adsorbate and the unused capacity of the adsorbent is the assumption that the rate of adsorption is controlled by the diffusion through the bed [12]. The fraction that remains in contact with the adsorbate system. Equation 1 was used to calculate $t$.

$$t = \frac{N_{u}D}{C_{u}K_{u}} - \frac{1}{K_{A}C_{i}} \ln \left( \frac{C_{u}}{C_{i}} - 1 \right)$$  \hspace{1cm} (3)

Where t is the service time of column (min), $N_{u}$ the adsorption capacity (mg/L), D is the bed depth of column (cm), $K_{u}$ the adsorption rate constant (L/mg.min), $C_{i}$ is the initial concentration of solute (mg/L), $C_{u}$ the desired concentration of solute at breakthrough (mg/L), $u$ is the superficial liquid velocity of feed to bed (cm/min). A plot of t versus bed depth D should yield a straight line where $N_{u}$ and $K_{u}$ the adsorption capacity and rate constant, respectively, can be evaluated [12].

2.2. Adam-Bohart model

The concentration of the sorbing species and residual capacity of the adsorbent are affected the adsorption rate when using this model. The characteristic parameters such as saturation concentration $N_{o}$ and the kinetic constant $K_{A}$ were estimated in this model [13]. The fraction that remains on the surface of the adsorbent is the driving force of the rate of adsorption is proportional. The Adams-Bohart model describes a relationship between the normalized concentration ($C_{t}/C_{o}$) and the bed height (H) equation 4 described this model:

$$\ln \left( \frac{C_{i}}{C_{o}} \right) = K_{A}C_{t} - \frac{K_{A}N_{o}}{u} H$$  \hspace{1cm} (4)

Where, $C_{i}$ and $C_{t}$ are the initial and breakthrough concentration (mg/L) respectively , $K_{A}$ is the Adam-
Bohart constant \((L/mg.min)\), \(t\) is the breakthrough time \((min)\), \(N_o\) is the adsorption capacity of the adsorbent in \((mg/L)\), \(n\) is the linear velocity in \((cm/min)\) and \(H\) is the bed depth in \((cm)\), \([14]\).

2.3. Thomas model

One of the most commonly used models in fixed bed adsorption processes is Thomas models. This model assumed that the process follows Langmuir theory of adsorption–desorption and there is no axial dispersion. Thomas model also assumes that the constant separation factor is applicable to either favorable or unfavorable isotherm \([15]\). The kinetic model presented by Thomas is of the form:

\[
\frac{C}{C_o} = \frac{1}{1 + \exp \left( \frac{k \cdot M \cdot (C_o - C)}{Q} \right)}
\]  \(\ldots \,(5)\)

Where \(C\) and \(C_o\) are the effluent and the influent concentrations respectively \((mg/L)\), \(Q\) is the volumetric flow rate, \(k\) is Thomas rate constant \((L/mg.min)\), \(M\) is mass of adsorbent \((g)\), \(q_o\) is the maximum capacity \((mg/g)\) and \(V\) is the throughput volume \((L)\).

3. Material and Work

3.1. Adsorbate

Reactive Blue dye (H3R) with wavelength (585 nm) and molecular weight (562) g/mol and is supplied from Al-Hilla textile factory south of Baghdad. Figure (1) shows the chemical structure of Reactive Blue.

![Chemical structure of Reactive Blue](image)

**Figure (1):** Chemical structure of Reactive Blue

3.2. Adsorbents

Wheat straw (RWS) and modified wheat straw (MWS) were used as adsorbents. The adsorbents are washed with distilled water and dried for 24 hours at 70 °C then, sieved to the desired particle size (1 mm). Glass container was used for stored the sample and. Cetyl-trimethylammonium bromide (CTAB) was used for modified by mixing 35 g of wheat straw with 4g/l of 500 mL of (CTAB) solution. Shaker was used to mixing the solution under room temperature for 5 hours. Distilled water was used for washing in order to remove any loosely adsorbed surfactant then dried to 70 °C and sieved. It’s worthy to stated that to increase the attraction between the dye molecules and the surface of the adsorbent the CTAB was used because its change the charge of the adsorbent \([16]\).

3.3. Experimental set-up

The rates of adsorption depend on the concentration of solute in the solution being treated in Continuous-flow operations. For column operation, the adsorbent is continuously in contact with a fresh solution. Dynamic flow mode biosorption performance of the biosorbent was investigated by pumping the reactive blue dye solution using a peristaltic pump into a rotameter to adjust the flow rate at 10 and 30 mL/min where the solution is fed to the adsorber using small-scale cylindrical fixed-bed column LD 2.5 cm and with a height of 50 cm.

A sample was packed with three different bed heights 10, 15 and 25 cm between two layers of glass wool into the column. The effluent samples were taking every (30 min) and measured for the dye concentration by UV/Visible spectrophotometer. The flow to the column was continued until the effluent concentration \((C_e)\) approached the influent concentration \((C_o)\). Figure (2) shows a schematic design of continuous adsorption process.

![Schematic design of continuous adsorption set up](image)

**Figure (2):** Schematic design of continuous adsorption set up

4. Results and Discussion

4.1. FTIR analysis

FTIR was used for identification for the gropes responsible for the adsorption process. The region of test was 400 – 4000 cm\(^{-1}\) \([17]\). Figure (3) shows the FTIR analysis. From Figure it’s clear that the structure of the surface is complex. The peaks indicate the complex structure of wheat straw. The peaks of 1427 and 1373 cm\(^{-1}\) associated with the C-O in phenols and –CH\(_3\) respectively. The peaks of 1732 and 1655 cm\(^{-1}\) are referring to the carbonyl group –C=O, 1508 cm\(^{-1}\) represents the aromatic rings. While the strong peak around 3414 cm\(^{-1}\) represents the hydroxyl groups -OH, the bond at 2916 cm\(^{-1}\) is attributed to the presence of the C-H bond. \([18]\), \([19]\).

![FTIR analysis of Raw wheat straw (RWS)](image)

**Figure (3):** FTIR analysis of Raw wheat straw (RWS)
4.2. Scanning Electron Microscope (SEM)

SEM is used to show if the surface of RWS and MWS are heterogeneous or homogeneous and check their porosity. Figures 4 (a and b) show the SEM analysis of both RWS and MWS. From Figure (4) (b) it’s clear that the activation process is greatly improved the structure of the modified Wheat straw due to several porous presence. The adhering adsorbate molecules will be more due to a high surface area for [20]. The detailed of FTIR and SEM are shown in our previous works [24] and [25].

![SEM microscope (a): RWS, (b): MWS](image)

Figure (4): SEM microscope (a): RWS, (b): MWS

4.3. Continuous Experiments

Experimental breakthrough curves were conducted to study characteristics of a fixed-bed column. A normalized Figure have to be done to describes the loading behavior of the adsorbate on a fixed-bed column. The initial adsorbate concentration \( (C_t/C_0) \) as a function of time for a given bed height or adsorbate flow rate must draw [21]. Figures (5) and (6) show the breakthrough curves of Reactive blue using RWS and MWS adsorbents. The breakthrough generally occurred faster with higher flow rate. The breakthrough time reaching saturation was increased considerably with a decreased in the flow rate. As shown in Figures (4) and (5), due to insufficient residence time of the solution in the column and diffusion of the solute into the adsorbent pores the adsorption capacity was lower at a higher flow rate. It’s clear from Figures that that the adsorption capacity is low in the case of RWS because of the low surface area in comparing to MWS. The increasing in the bed height will increasing the time of breakthrough curve, this attributed to that the increase in bed height will increase the contact time between the dye and the RWS and MWS. The increasing of bed meaning increasing in mass transfer zone and this led to a higher removal capacity and lower solute concentration in the effluent. At the highest bed due to an increase in the surface area of adsorbent a high adsorption capacity was observed because more binding sites for the adsorption [22].

![Breakthrough curve of Reactive Blue using RWS and MWS, pH=6.8, Co= 10 mg/L, flow rate =10 mL/min and bed height = 10, 15 and 25 cm](image)

Figure (5): Breakthrough curve of Reactive Blue using RWS and MWS, pH=6.8, Co= 10 mg/L, flow rate =10 mL/min and bed height = 10, 15 and 25 cm

![Breakthrough curve of Reactive Blue using RWS and MWS, pH=6.8, Co= 10 mg/L, flow rate =30 mL/min and bed height = 10, 15 and 25 cm](image)

Figure (6): Breakthrough curve of Reactive Blue using RWS and MWS, pH=6.8, Co= 10 mg/L, flow rate =30 mL/min and bed height = 10, 15 and 25 cm

4.4. Modeling and analysis of data column

4.4.1. Bed Depth Service Time model (BDST)

Figure (7) shows the BDST model by applying Eq. (3) for RWS and MWS using 10 mL/min flow rate. Table 1 shows the BDST model parameters using 10 and 30 mL/min flow rates. From Table (1), it is apparent that with increase in flow rate, the adsorption capacity No and kinetic constant KB increased, this indicated that in the initial part of biosorption, the overall system kinetics was dominated by external mass transfer for all the adsorbents [23].
4.4.2. Adam-Bohart model

In order to provide a simple and comprehensive approach for running and evaluating sorption-column tests Adams-Bohart model was used. The validity of this model is limited to the range of conditions used. Figure (8) shows the Adam-Bohart model fitting of two adsorbents. Table (2) shows the parameters of Adam-Bohart model using Eq. (4). The values of $K_A$ decreased with flow rate increase and it increased with increasing bed heights. This showed that the overall system kinetics was dominated by external mass transfer in the initial part of adsorption in the column [23]. The regression shows a high correlation fitting with RWS while shows a good fitting of MWS indicating that this model is appropriate for the initial part of adsorption process which is controlled by the mass transfer. [24].

| Table (1): Bed depth service time model parameters of Reactive Blue. |
|-----------------|----------------------|----------------------|
| Flow rate (mL/min) | Adsorbent | RWS | MWS |
| | Bed height (cm) | | |
| 10 | N_e (mg/L) | 78.68 | 87 |
| | $K_e10^4$ (L/mg.min) | 7.0 | 4.2 |
| | $R^2$ | 0.964 | 0.893 |
| 30 | N_e (mg/L) | 235.7 | 347.1 |
| | $K_e10^4$ (L/mg.min) | 9.0 | 6.3 |
| | $R^2$ | 0.965 | 0.862 |

4.4.3. Thomas model

The Thomas parameters, adsorption capacity $q_{cal}$, $q_{exp}$, rate constant $K_T$ and correlation coefficient values are illustrated in Table (3), as shown in table the rate constant $K_T$ is increased with increasing bed height and flow rate and the adsorption capacity $q_{cal}$ is decreased with increasing bed height and increased with increasing the flow rate. Figures (9)-(12) show the Thomas model for both RWS and MWS using different bed heights.
Figure (11): Thomas model of Reactive Blue using MWS, 10 mL/min, (10, 15, 25 cm)

Figure (12): Thomas model of Reactive Blue using MWS, 30 mL/min, (10, 15, 25 cm)

Table (3): Thomas model parameters of Reactive Blue

| Flow rate (mL/min) | Adsorbent | RWS | MWS |
|-------------------|-----------|-----|-----|
|                   | Bed height (cm) | 10  | 20  | 25  | 10  | 20  | 25  |
| 10                |                      | 3.8 | 2.7 | 2.62 | 16.9 | 5  | 11.2 | 7.8 |
|                   |                      | 8.94 | 6.77 | 4.65 | 12.9 | 5  | 9.07 | 6.11 |
|                   | K10 (L/mg min)      | 2.1 | 1.9 | 2.2  | 8.2  | 8.9 | 8.6 |
|                   | R²                  | 0.98 | 0.98 | 0.97 | 0.80 | 0.80 | 0.86 |
| 30                |                      | 9.6 | 7.12 | 5.7  | 31.4 | 7  | 24.8 | 17.0 |
|                   |                      | 24.7 | 17.3 | 12.8 | 32.2 | 23.9 | 16.1 |
|                   | K10 (L/mg min)      | 2.3 | 2.3 | 2.0  | 5.8  | 5.7 | 6.2 |
|                   | R²                  | 0.99 | 0.98 | 0.98 | 0.94 | 0.96 | 0.96 |

5. Conclusion

The investigation showed that the Modified wheat straw using CTAB cationic surfactant was a promising for Reactive Blue dye removal from aqueous solution using fixed-bed adsorption column. The highest breakthrough time was found at 10 cm bed height with flow rate of 10 mL/min for MWS. The highest maximum bed capacity was obtained at a lower bed height 10 cm and 10, 30 mL/min. Fixed-bed models showed a good fitting with the experimental data for MWS. RWS showed a good regression data fitting with BDST model for 10 mL/min (R²=0.964) and 30 mL/min (R²=0.965). RWS also showed a highly fitted regression with Adam-Bohart model and Thomas model. MWS regression is highly fitted with Thomas model.

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