

Removal of Basic Dyes from Aqueous Solution by Adsorption Using Rice Husk Ash-A Fixed Bed Column Study

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Abstract— The present article deals with the removal of two basic dyes MB and MG [i.e. methylene blue and malachite green] from a solution mixture using Rice Husk Ash, an agricultural waste. MB and MG are most commonly used dyes in textile industries and removal of two dyes with higher concentrations from the solution mixture is a typical attempt to solve the real life problem on dye removal. The effects of bed depth, influent concentration, flow rate, pH of the influent solution on breakthrough curve were investigated. Thomas, Yoon-Nelson, Adams-Bohart and Bed Depth Service Time [BDST] model were used to validate the adsorption performance and breakthrough curves. The removal of dyes at different flow rate [5, 7.5, 10, 12.5 ml/min], bed depths (6, 8, 10 cm), influent concentrations [170, 190, 200, 240 mg/L] and pH value [5.1, 7, 9.2] using Rice Husk Ash as an adsorbent were studied. The experimental results indicated that percentage adsorption of dyes increased with decreasing flow rate, increasing bed depth and lowering of influent concentration. The results further revealed that the percentage removal of dyes in the mixture was 99 %. Adams-Bohart and Thomas model were performed well than the other two models in use.

Keywords— adsorption, breakthrough curve, methylene blue, malachite green, models, rice husk ash.

I. INTRODUCTION

Dye manufacturing industries and many other industries using dyes and pigments generate liquid wastes, are characteristically high in color and organic content. Presently, it was estimated about 10000 of different dyes, pigments exist commercially, and over 7×10^5 tones are produced annually worldwide [1].

Dyes are widely used in industries such as textile, rubber, paper, plastic, cosmetic etc. Among these various industries, textile rank first in usage of dyes for coloration of fiber. The conventional wastewater treatments that rely on aerobic biodegradation have low removal efficiency for reactive and other anionic soluble dyes [2]. Hence,

conventional biological treatment processes are not found to be much effective in treating dye wastewater. Adsorption process is one of the most effective methods for removal of dyes from the effluent, which has a distinct edge over other methods due to its sludge free clean operation and complete removal of dyes, even from the diluted solution. Activated carbon is most widely used adsorbent because of its excellent adsorption efficiency for the organic compounds. However, commercially available activated carbon is very expensive. Furthermore, regeneration using solution produced small additional effluent while regeneration by refractory technique results in a 10-15% loss of adsorbents and its uptake capacity [3]. This had led to further studies for cheaper substitutes for the adsorbents. Nowadays, there are numerous numbers of low cost commercially available adsorbents, which are being used for the dye removal. The most widely used adsorbents for this purpose were orange and banana peels [4], neem leafs and other agricultural wastes [5]. Rice husk ash [RHA] is one of such low cost adsorbents, which is available as waste material in the agricultural fields in rural India. Rice husk ash contains abundant floristic fiber, protein and some functional groups such as carboxyl, hydroxyl etc., which make adsorption process possible [6].

The aim of the study is to develop a cost-effective technology for adsorption of dye solution, which is a mixture of two most commonly used basic dyes viz. Methylene Blue [MB] and Malachite Green [MG] in textile industries. The real life problem of removing color from textile effluent containing mixture of basic dyes has been addressed in this study. The objectives of the present work can be stated as to investigate the effects of the initial pH, flow rate, influent concentration and bed depth on adsorptive removal of mixture of two basic dyes viz. MB and MG in locally available rice husk ash [RHA] packed column. Thomas, BDST, Yoon-Nelson and Adams-Bohart models are used to predict the performance.

II. MATERIAL AND METHODS

2.1 Adsorbent

The Rice Husk was collected locally and was burnt in the muffle furnace near 550°C, washed a few times with distilled water, dried for 8 hours at 60°C in the oven. The ash was sieved through 600 micron and retained by 300 micron and finally was used for column studies.

2.2 Adsorbate

Malachite green [A.R. Grade, purity>96%] and Methylene blue (A.R. Grade, purity >95%) were procured from local market and used as received. The two dyes are mixed in equal proportion (1:1) to make the dye solution.

2.3 Adsorption experiments

Continuous adsorption of dyes in mixed solution of methylene blue and malachite green dyes was studied using a fixed bed acrylic column with internal diameter of 50 cm and single sampling point at the bottom. The adsorbent was packed over the glass beads and glass wool to ensure uniform inflow through the column. The mixed dye solution was introduced from the top to the bottom of the column using a peristaltic pump at desired flow rate. The experiment was carried out by passing the dyes solution through the column packed with RHA with controlled flow rate. The experimental observations were noted operating the column at steady state under different conditions.

III. COLUMN ADSORPTION MODEL

The performance of a packed bed is obtained through the concept of breakthrough curve. The time for breakthrough appearance and the shape of the breakthrough curve are important characteristics for determining the operation and dynamic response of an adsorption column. Moreover, successful design of an adsorption column requires prediction of the concentration-time profile from breakthrough curve for the effluent discharged from the column [(7)]. In many cases, kinetics of adsorption in column has been tested for Adams-Bohart model. However, it has also been shown that BDST, Thomas and Yoon-Nelson models can sometimes provide a better description of the adsorption kinetics. So in this study, an attempt has been made to find out the best model describing the adsorption kinetics in the column studied.

3.1 Thomas Model

The Thomas model is one of the most general and widely used methods in studying the performance of a continuous column operation. The expression by Thomas for an adsorption column (8) is given as follows:

$$\frac{C_t}{C_0} = \frac{1}{1 + \exp\{K_{TH}(q_0x - C_0V_{eff})/v\}} \quad (1)$$

where, C_t is the effluent dye concentration [mg/L], C_0 is the initial dye concentration [mg/L], x is the mass of the used adsorbent [g], V_{eff} is the effluent volume [mL], v is the flow rate (mLmin⁻¹), K_{TH} is the Thomas rate constant, and q_0 is the maximum dye adsorption capacity of the adsorbate (mg/g), which is calculated from the plot of $\ln[(C_t/C_0)-1]$ versus t .

3.2 Yoon-Nelson Model

The linear form of Yoon-Nelson model [9] is:

$$\ln[C_t/(C_0 - C_t)] = K_{YN}t - TK_{YN} \quad (2)$$

where, K_{YN} is Yoon-Nelson constant, T is the time required for 50 % adsorbate breakthrough and t is the sampling time. A plot of $\ln [C_t/(C_0-C_t)]$ versus t gives straight line curve with a slope of K_{YN} and intercept of TK_{YN} . Based upon the obtained value of T , the adsorption capacity, q_{0YN} was determined using:

$$Q_{0YN} = q(total)/X \quad (3)$$

Adsorption capacity (q_{0YN}) related to Yoon-Nelson varies as inlet dye concentration [C_0], flow rate [Q], 50% breakthrough time derived from Yoon-Nelson equation and weight of adsorbent [X].

3.3 Bed Depth Service Time Model

The BDST model relates the service time of a fixed bed with the height of adsorbent in the bed, hence with its quantity, as its quantity is directly proportional to the bed height. The measurement of sorbent quantity is more precise than the determination of the respective volume, especially for the case of granules. Therefore, sorbent quantity is being preferably used, instead of the bed height. The linear form of BDST model [10] is:

$$t = \frac{N_0}{C_0 F Z} - 1/K_a C_0 \ln[C_0/(C_t - 1)] \quad (4)$$

where, t is the service time [min], N_0 is the adsorption capacity [mg/g], F is the superficial liquid velocity [cm/min], Z is the height of column [cm], and K_a is the rate constant of adsorption (L/min/mg) at time t . A plot of t versus bed depth Z , should yield a straight line where N_0 and K_a , the adsorption capacity and rate constant respectively, can be evaluated. Application of the BDST model requires specification of the breakthrough time, which was selected arbitrarily in this work as the time corresponding to C_t/C_0 ratio is varying from 0.1 to 0.4.

3.4 Adams-Bohart Model

Adams-Bohart established the fundamental equations that describe the relationship between C_t/C_0 and time in an open system for the adsorption of dye solution of mixture of MB and MG onto RHA. The model proposed assumes that the adsorption rate is proportional to both the residual capacity of the RHA and the concentration of the sorbing species. Assuming certain conditions, the linear form of the model equation [11] is:

$$T = \left(\frac{N_0 x}{C_0 v} \right) - \ln(C_0/C_t - 1)/C_0 K_{AB} \text{ -----(5)}$$

where, K_{AB} is the rate constant and N_0 the adsorption capacity, which are calculated from plot of $\ln\{C_t/C_0\}$ versus time t .

IV. RESULTS AND DISCUSSION

4.1 Effect of bed depth on breakthrough curve

Accumulation of two basic dyes in aqueous solution MB and MG in the packed bed column is largely dependent on the quantity of sorbent inside the column. In order to yield different bed heights, 4.165, 6.723 and 8.126 gm of RHA were added to produce the desired height of 6, 8 and 10 cm respectively.

Fig 4.1 shows the breakthrough curve obtained by varying the bed heights for different RHA between 6 cm, 8cm and 10 cm, at flow rate $[q_0]$ of 10 ml/min and at a fixed influent concentration $[C_0]$ of 170mg/L. Similar approach was made with other influent concentrations also. The time required for the effluent to reach breakthrough concentration increased with increase in the bed depth. The increase in adsorption with bed depth may be attributed to the increase in adsorptive binding sites of the adsorbent. Although an increasing bed depth increases breakthrough time, very high bed depth is not useful for single column. The shape and gradient of the breakthrough curves were slightly different with the variable bed depth. As the bed height increases in the order of 6 cm, 8 cm, and 10 cm the curves change their profile from steep concave to flatter concave [12].

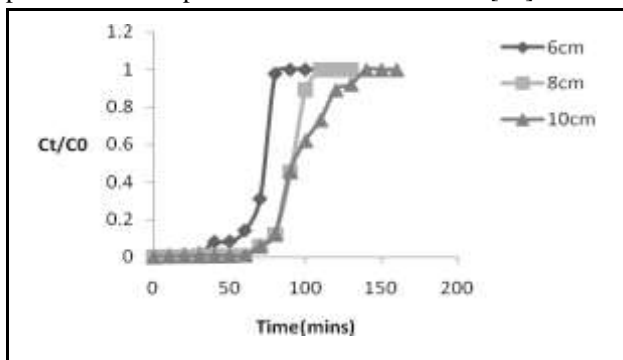


Fig.4.1 Effect of bed height (at 4, 6 and 8cm) on adsorptive removal of methylene blue and malachite green onto RHA (at $C_0 = 170\text{mg/L}$ $q_0 = 10\text{mL/min}$)

4.2 Effect of flow rate on breakthrough curve

Fig. 4.2 shows the breakthrough profile for the adsorption of mixed dye solution of methylene blue and malachite green in the continuous flow fixed bed with RHA at room temperature. The flow rate was varied from 7.0 mL/min to 14 mL/min while the inlet concentration and bed depth of the solution was held constant at 200 mg/L and 6 cm respectively. When the adsorption zone moves upwards and upper edge of this zone reaches the top of the column,

the effluent concentration has started to rise rapidly. This point is called breakthrough point. Using a smaller flow rate, the front of the adsorption zone reaches the top of the column later, thereby giving higher breakthrough time. An increase in the flow rate appears to increase the sharpness of the breakthrough curve. As the flow rate through the bed increases, the depth of the adsorption zone increases and the time of contact of the solution with adsorption zone decreases [13].

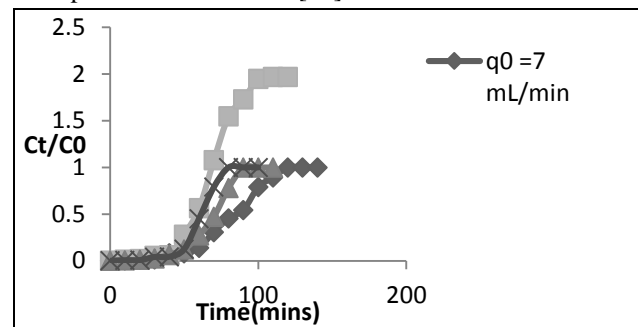


Fig.4.2 Effect of flow rate (at 7, 10, 12 and 14mL/min) on adsorptive removal of methylene blue and malachite green onto RHA [at $C_0 = 200\text{mg/L}$, $H = 6\text{cm}$]

4.3 Effect of initial dye concentration on breakthrough curve

Fig. 4.3 shows the breakthrough profile for the adsorption of methylene blue and malachite green in dye solution in the continuous flow fixed bed with RHA at different influent concentrations at room temperature. The influent concentration was varied from 170 mg/l to 240 mg/l, while the bed depth of the column and influent flow rate were held constant at 6 cm and 10 mL/min respectively. The similar approach was taken for bed height 8 cm and 10 cm respectively. It was illustrated that the breakthrough time decreased with increasing influent dye concentration. At lower concentrations, breakthrough curves were dispersed and breakthrough occurred slower. As influent concentration increased, sharper breakthrough curve were obtained. These results demonstrated that the concentration gradient affects the saturation rate and breakthrough time. This can be explained by the fact that more adsorption sites were being converted and utilized as the dye concentration increased. The larger the influent concentration the steeper is the slope of breakthrough curve and smaller is the breakthrough time. As the influent concentration increases, dye loading rate increases, so does the driving force increases for mass transfer, which results in a decrease in the adsorption zone length [14].

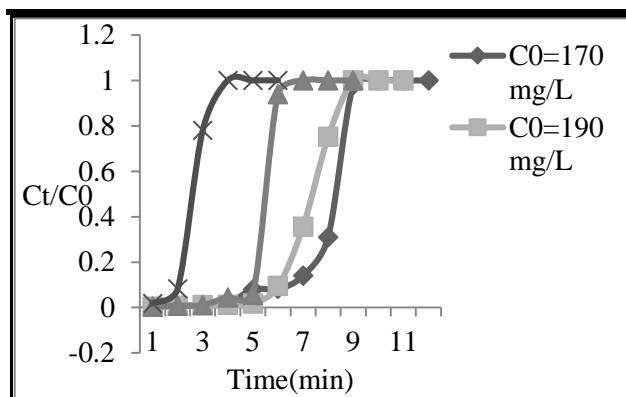


Fig.4.3 Effect of influent concentrations [at 170, 190, 200 and 240mg/L] on adsorptive removal of methylene blue and malachite green onto RHA [at $H=6\text{cm}$, $q_0=10\text{mL/min}$]

4.4 Effect of pH on breakthrough curve

The pH is regarded as a critical parameter in the adsorption process since it controls the uptake mechanism of the adsorbate as well as influences the adsorption sites on the solid surface. In the present study, the pH of the dye solution was taken as 5.1, 7.0 and 9.2 by using pH capsules into the solution while the bed depth and concentration were restricted at 8 cm and 200 mg/L respectively. The figure 4.4 shows that the adsorption increased as the pH value increased. This was due to the fact that in basic solution [$\text{pH}=9.2$] the adsorption of cationic dyes was favoured due to presence of negatively charged sites on the adsorbent surface, resulting in the electrostatic forces between groups and positively charged dye molecules. On the other hand, in acidic solution ($\text{pH}=5.1$) the adsorption of dyes was retarded due to sorption competition between hydrogen ions (H^+) and dye molecules for occupancy of the active sites (15).

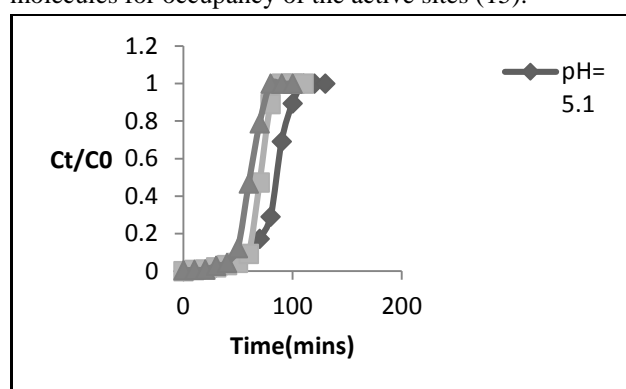


Fig.4.4: Effect of pH (at 5.1, 7.0 and 9.2) on the adsorptive removal of methylene blue and malachite green onto RHA (at $H=8\text{cm}$, $C_0=200\text{mg/L}$).

4.5 Data fitting to Thomas Model

The data obtained from the experiment were fitted to the Thomas model using equation (1). From the analysis, it can be concluded that all of the factors viz. the inlet

concentration, bed depth, flow rate, pH influence the Thomas rate constant [K_{TH}] and the equilibrium of mixed dyes uptake [q_0]. The inlet concentration, bed depth, flow rate and pH of the influent dye solution of MB and MG influences the K_{TH} and q_0 as given in the Table -1. From the above table it is obvious that when concentration was increased from 190 mg/L to 200 mg/L for a bed depth of 6 cm, q_0 increased from 13.82 to 14.09 and K_{TH} decreased from 0.126 to 0.125. Similarly, for the same inlet concentration for a bed depth of 10cm the K_{TH} value decreased from 0.084 to 0.042 where as q_0 increased from 18.01 to 18.14.

When the bed height was maintained at 6 and 10 cm, the K_{TH} value was decreased as 0.126, 0.084 and q_0 increased as 13.82, to 18.01 respectively for inlet concentration of 190 mg/L. In another set of experimental result where bed depth was varied successively at 6 and 10 cm, the K_{TH} value was obtained as 0.0125 and 0.042 respectively, for a concentration of 200 mg/L. The corresponding values for q_0 increased with the increase in bed depth were 14.09, and 18.14 respectively.

When the flow rate increased from 7.0 mL/min, to 14 mL/min, the K_{TH} value increased from 0.105 to 0.265 and corresponding values for q_0 increased from 12.11 to 16.34.

When the pH of the inlet concentration increased from 5.1 to 9.2, the k_{TH} values were 0.165, 0.192 and 0.205 respectively whereas, uptake q_0 decreased as 24.26, 20.17 and 18.44 respectively. The R^2 value for over all reactions varied 0.915 to 0.986 indicated that Thomas model described the experiment well.

4.6 Data fitting to Bed Depth Service Time [BDST] Model

The constants, N_0 and K_0 of BDST model at different conditions were evaluated using linear regression analysis and the linear plot of this model is given in Table-3. The trend of K_0 and N_0 could be determined from the values presented in the table. As C_t/C_0 value increased, the value of K_0 and N_0 were found to be decreased. The R^2 value for overall reactions varied from 0.7 to 0.75 indicated that this model cannot describe the experimental run well.

4.7 Data fitting to Yoon-Nelson Model

Different parameters of the Yoon-Nelson model can be determined by using linear regression analysis. The equation of straight line was used to determine the rate velocity constant (K_{YN}) and time (T) required to achieve 50% breakthrough. From the Table-2, the effect of influent concentration, bed depth, flow variation and variation of initial pH of the mixed dyes solution over the model parameters were studied. When the concentration of influent dyes increased from 170 to 240 mg/L at a

constant bed depth of 6 cm, the values of K_{YN} increased from 0.016 to 0.027 and those of T decreased from 40.69 to 34.88. Similarly, for bed depth of 8 cm, when the concentration increased from 170 to 240 mg/L, the values of K_{YN} increased from 0.014 to 0.023 and those of T decreased from 69.92 to 44.83.

With the increase in bed depth for a fixed influent concentration there was no significant change for the values of K_{YN} and T .

With the increase in flow rate from 7.0 to 10, 12, and 14 mL/min, K_{YN} increased from 0.040 to 0.046, 0.056 and 0.064 respectively and the values of T increased from 53.11 to 57.10, 57.78 and 58.23 respectively.

With the increase of pH of the inlet concentration from 5.1 to 9.2, there was no significant effect on K_{YN} value. T value increased slightly with the increasing pH of the solution. The linear regression coefficient (R^2) in all cases was around 0.76 which indicated that this model cannot be used to explain the overall kinetics explicitly in the column adsorption.

4.8 Data fitting to Adams-Bohart Model

A plot of $\ln(C_0/C_t)$ against time (t) was used to calculate the values of N_0 and K_{AB} . The Table-4 the different model parameters like bed depth, concentration of initial adsorbate concentrations, flow rate of solution dyes and the pH of the solution were studied. From the table it indicated that with the increase in influent concentration from 200 to 240 mg/L at bed depth of 8 cm, K_{AB} increased from 0.040 to 0.050 whereas the values of uptake (N_0) decreased from 0.09 to 0.006. Similar trend on parametric values was also observed for a bed depth of 10cm

The effects of bed depth over the reaction constant K_{AB} and uptake N_0 was investigated and was shown in the Table. With the increase in bed depth, the reaction constant (K_{AB}) increased while the uptake (N_0) decreased. The effect of flow rate over the reaction constant (K_{AB}) and the uptake value (N_0) was depicted in the Table-3. As the flow rate increased from 7.0 to 14 mL/min, the values of K_{AB} and N_0 increased.

The effect of pH of the inlet solution was studied. The reaction constant (K_{AB}) decreased and uptake (N_0) increased when pH increased from 5.1 to 9.2. The R^2 value for over all reactions more than 0.9 indicated that Adams-Bohart model described the experiment well.

Table.1: Thomas model parameter

H cm	C_0 mg/L	q mL/min	pH	K_{Th} mL/mg/min	q_0 mg/g
6	190	7.5	7.0	0.126	13.82
	200			0.125	14.09

10	190			0.084	18.01
	200			0.042	18.14
6	200	7.5	7.0	0.105	12.11
		10.0		0.170	13.43
		12.5		0.235	15.36
		14.0		0.265	16.34
8	200	7.5	5.1	0.165	24.26
			7.0	0.192	20.12
			9.2	0.205	18.44

Table.2: Yoon-Nelson Model Parameters

H cm	C_0 mg/L	q mL/min	pH	K_{YN} L/min	T min
6	170	7.5	7.0	0.016	40.69
	240			0.027	34.88
8	170	7.5	7.0	0.014	69.92
	240			0.023	44.83
6	200	7.5	7.0	0.040	53.11
		10.0		0.046	57.10
		12.5		0.056	57.78
		14.0		0.064	58.27
8	200	7.5	5.1	0.019	64.63
			7.0	0.014	69.93
			9.2	0.014	81.00

Table.3: BDST Model Parameters

C_0	C_0/C_t	K_0	N_0
170	0.1	0.0005	485.9
	0.2	0.0002	302.3
200	0.2	0.0003	1174
	0.4	0.0001	774.18

Table.4: Adams-Bohart Model Parameter

H cm	C_0 mg/L	q mL/min	pH	K_{AB} L/mg.min	N_0 mg/L
8	200	7.5	7.0	0.040	0.09
	240			0.050	0.006
10	200	7.5	7.0	0.050	0.007
	240			0.053	0.004
6	200	7.5	7.0	0.09	0.09
		10		0.10	0.10
		12.5		0.13	1.16
		14		0.15	1.21
8	200	7.5	5.1	0.047	0.009
			7	0.024	0.012
			9.2	0.029	0.013

V. CONCLUSION

The present study confirmed that the abundant agricultural waste product rice husk ash could be used as an alternative to commercial activated carbon for the removal of two basic dyes MB and MG from aqueous mixture. The breakthrough curve (C_t/C_0 vs. t) for various parameters viz. flow rate, initial concentrations of two dyes in solution, bed heights of the adsorbent, and pH of the dyes in initial solution was plotted. The breakthrough time and exhaust time decreased with increasing flow rate, increases with increasing bed height and pH value of the concentration. The breakthrough time and exhaust time decreased with increasing initial dye concentration. These variations can be accounted for by the fact that for lower flow rate and higher bed depth, the contact time is longer and hence the interaction between adsorbate and adsorbent is greater. The experimental data were fitted to Thomas, Adams-Bohart, Yoon-Nelson, and BDST models. The maximum adsorption capacity related to BDST model was found to be 774.18 mg/l at initial concentration (C_0) 200mg/L, flow rate (q_0) 10 mL/min, and bed height (H) 10 cm. The investigation showed that the Thomas model and Adams-Bohart model were more effectively applicable than other two models for their higher R^2 value.

ACKNOWLEDGEMENTS

The authors are thankful to Mr. Anirban Kundu Chowdhury, Research Scholar, School of Environmental Sciences, Jadavpur University for his sincere support during the course of experimental work. The authors are also grateful to Mr. Snehil Kumar M.E. student and all laboratory staff members of Department of Civil Engineering, Jadavpur University for their technical assistance.

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