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Optimization of column studies on the adsorption of congo red dye using phophoric acid-treated eichhornia crassipes

Kalai Selvi S^{1*}, Suganthi N²

¹Research scholar, ²Assistant Professor ¹Research and Development Centre, ²Department of Chemistry, ¹Bharathiar University, Coimbatore, India, ²LRG Govt Arts College, Tirupur, Tamilnadu, India

Abstract : The present paper examines the use of phosphoric acid-modified eichhornia crassipes for the continuous adsorption of congo red (CR) dye in columns. The adsorbent was characterized using Fourier transform infrared spectroscopy and X ray diffraction study. A fixed bed column analysis was carried out to evaluate the parameters that affecting the adsorption of CR dye onto phophoric acid-modified eichhornia crassipes, such as initial CR concentration (80–140 mg/L), column bed height (5–20 cm), and feed flow rate (5–15 mL/min). Maximum bed capacity of 15.21 mg/g was achieved at 100 mg/L inlet CR dye concentration with 10 cm bed height and 5 mL/min of feed flow rate. Thomas and model was in good agreement with the experimental results. Desorption and use of spent carbon as admixture in concrete datas has been obtained.

Key words : Adsorption, Eichhornia Crassipes, Congo red, Fixed bed column, Admixture.

I. Introduction

Rapid industrialization leads to destruction in natural environment. One of the most polluting industries is textiles and dyeing units. These sectors release enormous amount of colored dye effluent into near water streams. Dyes are synthetic aromatic compounds with more complex structure, which resist degrading or degrade to form toxic compounds. Most of the dyes and their degraded compounds are carcinogens and mutagens in nature. In particular, azo dyes are largely employed in food and textile industries. Azo dyes are synthetic organic dye with –N=N- (azo) group. This group is highly stable and resists degradation. Hence if not treated properly these dyes remain stable in the environment [1]. Consumption of colored water causes many health problems [2]. The textile waste water needs treatment before they are discharged into the environment [3,4]. Several techniques have applied to eliminate color from wastewater including advance oxidation, ion exchange, chemical coagulation or flocculation, electrocoagulation, nanofiltration, ultrafiltration. electrodialysis, ozonation and reverse osmosis [5-13]. All of the techniques have advantages and drawbacks. However, all these processes are costly and cannot be utilized by small industries to treat the wide range of wastewater [14]. In addition to already mentioned methods adsorption is a unique process for the removal of color from water and wastewater.

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Adsorption with activated carbon has been proved to be an efficient, cost effective successive process for the treatment of colored waste water. Countries like India; needs necessitate searching for a new class of cost-effective materials. Currently, attention has been focused on agricultural waste as a source to prepare adsorbents. Agricultural wastes have been accepted as an ecological burden, low cost and sustainable adsorbents for water treatment applications [15]. Attempts are made for the removal of azo dyes by using tea waste, Chitosan, orange peel, tamarind fruit shell, soya, rice husk, peanut hull, cattail root, baggase, sawdust, sugar molasses [16-25] were already reported. In this study, an aquatic weed Ecihhornia crassipes was used as adsorbent for removal of CR from aqueous solution by fixed bed column analysis.

II. Research Methodology

2.1 Adsorbate:

Congo red dye is benzidine-based anionic diazo dye. CR is difficult to biodegrade due to their complex aromatic structures, which provide them with physico-chemical, thermal, and optical stability [16]. It is the sodium salt of benzidine diazo-bis- 1-naphthylamine-4-sulfonic acid of molecular formula: $C_{32}H_{22}N_6Na_2O_6S_2$ and molecular weight of 696.66 g/mol). Figure1 shows the structure of CR



Figure: 1 Structure of congo red

2.2 Adsorbate preparation

A Stock solution of aqueous solutions of CR dye, for this study were prepared by the concentration of 1000 mg/L i.e., dissolving 1gm of the dye in a litre of distilled water. The working solutions of appropriate concentrations (80, 100, 120 and 140 mg/L) were prepared by diluting the stock solution with distilled water

2.2 Preparation of adsorbent:

Eichhornia crassipes was collected from nearby channel and ponds. They were washed repeatedly with distilled water to remove earthy impurities and dust present in it and then they were cut into small pieces and dried them at 110° C in air oven. The material was carbonized by the physical and chemical methods.

		Carbonisation	Drying	
Name of the process	Reagent Used	process	Process	
Pyrolysis Process	-	Carbonised at 300°C	-	
Carbonization with Chloride Salts Process Carbonization with Sulphate Salts Process	10 % of calcium chloride, zinc chloride, manganese chloride and ammonium chloride solution10 % solution of sodium sulphate and ammonium sulphate	for a period of 24 hours and thermally activated in muffle furnace at 800 ⁰ C for a period of 15	The material obtained was washed well with water and dried at 110° C	
Carbonization with carbonate salts process	10 % sodium carbonate and potassium carbonate solution	minutes.	in hot air oven.	

Table1: Preparation of carbon by various processes

Dolomite process	Eichhornia crassipes	were
	sandwiched between two	layers of
	calcium carbonate bed	
Phosphoric Acid	10 % Phosphoric acid	
Process		

2.3 Analysis of activated carbon

The carbons prepared by various methods were powered, sieved and particle in the range $75 - 300 \mu m$ mesh size was retained for the evaluation of different carbon characteristics. The activated carbons were analyzed for its moisture, ash, volatile matter and fixed carbon content, matter soluble in water and acid using the Method IS 1350, 1984. Ultimate analysis was done by using the Method IS 1350-4-2. Each carbon was separately evaluated and the results of the tests are summarized in Table – 1.

S. No	Control Tests	Pyrolysis process	Chloride process	Sulphate process	Carbonate process	Dolomite process	Acid process
1	Bulk density g/cc	0.57	0.55	0.16	0.6	0.67	0.29
2	Moisture %	4.98	3.56	4.36	6.3	7.81	4.1
3	Ash %	3.39	4.15	3.22	3.68	1.66	1.97
4	Fixed carbon content	96.61	95.85	96.78	96.32	98.34	94.07
5	Matter soluble in water (%)	0.51	0.37	0.57	0.65	0.96	1.11
6	Matter soluble in acid (%)	3.35	2.9	1.75	2.22	0.41	2.35
7	рН	5.72	4.71	4.51	6.44	5.87	5.2
8	Decolorizing power (mg /g)	20.5	23.5	20.41	23.7	22.38	31.50
9	Phenol number (mg)	15.7	16.6	18.3	19.0	16.1	20.0
10	Ion exchange capacity (meq/ g)	0.62	0.516	0.19	0.011	0.36	6.73
11	Iron content`	-	-	-	-	-	0.0699
12	Surface area m ² /g	263	156	123	217	120	320

Table 2: Carbon Characteristics

In the study, carbon prepared by the phosphoric acid method showed favorable physico-chemical characteristics. Hence it was selected for further dye adsorption studies. It was labeled as ECAC.

2.3 X-ray diffraction studies

X-ray diffraction (XRD) technique is a powerful technique to analyze the crystalline and amorphous nature of the material. In crystalline material, well defined peaks are observed whereas in non crystalline or amorphous material shows broad peaks instead of sharp peaks. The x-ray diffraction pattern of the synthesized carbon sample was shown in Figure 2. The prepared activated carbon (ECAC) has five peaks corresponding to reflections $2\theta = 10-80^\circ$. The three strong peaks [at $2\theta \approx 24.28^\circ$ (002), $2\theta \approx 64.85^\circ$ (200), $2\theta \approx 77.66^\circ$ (220)] [26] present in the figure indicated the amorphous and crystal nature of the material. The studies indicates that the carbon have highly disorder structure and with more interlayer distances which can accommodate more dye molecules [26].



Figure 3: XRD pattern of ECAC

2.4 Fourier transforms infra red spectra

The FTIR pattern was an important tool to identify some important functional groups, which are capable of adsorbing pollutant ions. FTIR spectroscopy was, therefore, done for preliminary quantitative analysis of major functional groups presented in ECAC. The FTIR spectrum of ECAC before adsorption (BAAC) and after adsorption of CR (AACR) was shown in Figure.4.

Assignment	Band Position in cm ⁻¹			
Assignment	BAAC	AACR		
O- H stretching of hydroxyl group	3431.87	3866.34 - 3434.28		
C ⁻ H stretching in alkanes or alkyl group	2924.59 - 2357.51	2924.11 - 2360.41		
$C \equiv O$ stretching of anhydride	1624.56	1693.51 - 1649.63		
$C \equiv C$ of aromatic ring	-	1550.30 - 1462.05		
C- N stretching of aliphatic primary amine	1382.49 - 1030.96	1031.44		
C- X stretching of carbon halogen group	666.41	669.30 - 643.27		

Table 3: FTIR spectra band assignments for BAAC and AACR



Figure 4: FTIR pattern of BAAC and AACR

Table 3 shows the tabulated data for FTIR spectra band assignments for BAAC and AACR samples obtained from Figure 4. These spectral lines describe the various changes that occurred in the BAAC and

AACR samples. The spectrum for BAAC showed long bandwidth at 3431.87 cm⁻¹ which indicates the O -H stretching of hydroxyl group. The C - H stretching of alkanes or alkyl group was detected at bandwidth of 2924.59 - 2357.51cm⁻¹. The C–N stretching of aliphatic primary amine group was detected at bandwidths of 1382.49 - 1030.96 cm⁻¹. The C \equiv O stretching of carboxylic anhydrides functional groups was detected at bandwidths of 1624.56 cm⁻¹. The results indicate that some peaks were shifted or disappeared, and the new peaks were also detected in AACR sample. These changes observed in the spectrum indicate the possible involvement of those functional groups on the surface of the ECAC may used to adsorb CR from aqueous solution. Similar results were obtained in the study of the effects of acid leaching on porosity and surface functional groups of cocoa (Theobroma cacao)-shell based activated carbon [27].

2.5 Column adsorption studies

For the fixed bed column study of CR onto ECAC, the experiments were carried out in glass column of 20 cm length having inside diameter (2 cm). The activated carbon packed in the column with layers of glass bead and glass wool at bottom. The dye solution of specified concentration was allowed to flow from the top of the column in down flow method at fixed inflow rate using peristaltic pump. The amount of dye adsorbed was determined using UV-VIS spectrophotometer using UV-Vis spectrometer by fixing the wavelength of 500 nm for CR.

III. Result and Discussion

The ability of column adsorption process was evaluated using the breakthrough curves obtained at various initial concentration and bed height. The breakthrough time, volume and the shape of breakthrough curve are important characteristics for determining the operation and dynamic response of an adsorption column [28]. The effects of parameters such as flow rate, packed bed height and initial sorbate concentration were studied.

3.1 Effect of initial concentration

Fixed bed experiments were carried out for removal of CR dye using ECAC adsorbent. The effect of change in bed height, initial concentration and dye solution flow rate were investigated. The performance of fixed bed in the form of breakthrough curves was obtained. Adsorption of CR was presented in the form of breakthrough curves (i.e. Ct/Co Vs t).

The effect of influent dye concentration was evaluated by varying CR concentrations (80,100, 120) and 150 mg/L) at 10 cm bed height, 5 mL/ min flow rate and pH of 7 were depicted in the Figure 4.



Figure 4: Effect of initial concentration

Figure 5: Effect of flow rate

From the Figure 4 it was found that the breakthrough time decreased with increasing influent dye concentration. At lower influent CR concentrations, the breakthrough was distributed and reached slower; hence an extended breakthrough curve was obtained. As the dye concentration increased, sharper was the breakthrough. This result suggested that the change of concentration gradient effected the saturation rate and breakthrough time[28]. This can be described by the fact that more adsorption site on the sorbent's surface was being covered faster with an increase in influent concentration. Higher the dye concentration, steeper was the slope of breakthrough curves with smaller breakthrough time and volume. These results indicated that the change of concentration gradient affects the saturation rate and breakthrough time. As the influent concentration increased, CR loading rate on ECAC increased, causing the driving force for mass transfer to increase, which resulted in decrease of adsorption zone length. Han et al [29] reported similar results in the fixed bed adsorption of Congo red dye onto rice husk.

3.2 Effect of flow rate

Figure 5 shows the effect of flow rate for removal CR at fixed concentration of 100 mgL⁻¹, 10 cm bed height, pH of 7 and varying flow rate from 5 to 15 ml/min. Maximum removal was achieved at 5 ml/min flow rate for the carbon ECAC. Hence in all further studies 5 ml/min was maintained. It was found that the breakthrough time decreased with increasing flow rate from 5 to 15 ml/min. This is because of the fact that the contact time between the influent and ECAC is minimized at higher flow rate. Increasing the flow rate gave rise to a shorter time for saturation and the breakthrough curves become steeper and reached the breakthrough quickly [30].



3.3 Effect of bed depth

Adsorption of CR onto ECAC was conducted at varying bed height 5cm (4.502 g), 10cm (8.2 g), 15cm (11.811 g), 20cm (15.521 g) at influent CR concentration of 100 mg/L and flow rate of 5 mL/ min at pH of 7 and the results are shown in the Figure 6. The results indicated that the breakthrough curve and throughput volume increased with increase in bed height, this is due to the availability of more number of binding sites in the column [31]. As the bed height was reduced, the solute concentration in effluent increased more rapidly. This may be due to the fact that the adsorbate had no enough time to diffuse into the whole some of adsorbent mass; hence there is a decrease in sorption rate.

3.4 Estimation of breakthrough curves using non-linear regression analysis

In order to illustrate the fixed-bed column performance and to extent its application to industrial treatment, two models, Thomas and Adams–Bohart were used to fit the experimental data in the column.

Adams-Bohart model:
$$ln \frac{c_t}{c_0} = K_{AB} C_0 t - K_{AB} N_0 \frac{z}{u}$$
(1)

where, C_0 and C_t are respectively the influent and effluent concentrations (mg L⁻¹), K_{AB} = adsorption rate coefficient (L/mg/min); N_0 = adsorption capacity coefficient (mg/L); z = bed depth (cm); u = linear velocity (cm/min); and t = time (min). K_{Th} is the Thomas rate constant (mL/mg/min), q_0 is the maximum capacity of adsorption (mg g⁻¹), m quantity of adsorbent in the column (g), V_{ef} is volume of solution (mL) and F is the feed flow rate (mL mn⁻¹).

The Bohart–Adams model plot for the adsorption of CR onto ECAC column was shown in Figure 7 and the results were presented in Table 4. The adsorption rate coefficient K_{AB} decreases on increasing the influent concentration from 80 to 140 mg/L as given in Table 4. On increasing the concentration more sorbate molecules form greater concentration gradient which eventually decreases the adsorption rate coefficient. When the flow rate increased from 5 to 15 mL/min the adsorption rate coefficient increases and it decreases while increasing the bed height from 5 to 20cm. More availability of sorbate molecules on the adsorbent surface results in higher uptake of dye molecules by unit mass of adsorbent. The derivative adsorption capacity of the adsorbent (q_{BA}) was calculated from N. The experimental and calculated adsorption capacity has more difference. The regression coefficient values are not good, hence the applicability of Bohart - Adam model fails to support the adsorption of CR onto ECAC column.

The column mode adsorption data for the adsorption of CR onto ECAC column at various initial dye concentrations, flow rate and bed depth applied to Thomas model was shown in the Figure. 4.22 and the results of the plot were given in Table 4. It can be observed from the Table 4 that the Thomas constant K_{TH} varies from 1.0 X 10⁻⁴ to 1.6 X 10⁻³ L/min/mg on increasing the initial dye concentration, flow rate and the bed height. The adsorption capacity calculated using Thomas model increases on increasing the initial dye concentration from 80 to 140 mg/L and the bed height from 5 to 20 cm, while it decreases on increasing the flow rate from 5 to 15 mL/min. The calculated adsorption capacity values were in good agreement with the experimental adsorption-capacity. The calculated correlation coefficients are higher and equal to unity for Thomas model than that for Bohart–Adams model. Therefore, the sorption of CR can be approximated more appropriately by Thomas kinetic model than the Bohart–Adams model kinetic models for the adsorption.

Carbon	Concen tration, mg/L	Flow Rate, mL/m in	Bed height, cm	Mass of adsorbent (g)	Break through time t0.5 (min)	Break through time t0.5 volume (ml)	Complet e exhausti on time	Complete exhaustion volume
	80	5	10	8.2	300	1500	440	2200
	100	5	10	8.2	260	1300	380	1900
	120	5	10	8.2	220	1100	340	1700
U	140	5	10	8.2	200	1000	320	1600
CA	100	5	5	4.502	120	600	240	1200
Ē	100	5	15	11.811	360	1800	540	2700
	100	5	20	15.521	500	2500	680	3400
	100	10	10	8.2	180	900	300	1500
	100	15	10	8.2	140	700	220	1100

Table 4 (a): Column results for adsorption of CR onto ECAC



Figure 7(a): Bohart – Adams plot-Variation in initial concentration



Figure 7(c): Bohart – Adams plot-Variation in depth



Figure 7(b): Bohart – Adams plot-Variation in flow rate



Figure 8(a): Thomas plot-Variation in initial bed concentration

at L	l, e,	ш	f nt			Adams Bo	hart Model		Т	homas Mod	el
Concentr ion, mg/	Flow Rat mL/mir	Bed height, cı	Mass of adsorber (g)	q_0 (exp), mg/g	K _{AB} (L/mg/ min)	N ₀ (mg/L)	q0 (AB), mg/g	\mathbb{R}^2	K _{TH} (ml/min /mg)	q0 (T), mg/g	R^2
80	5	10	8.2	13.67	0.00013	13.286	16.2	0.802	0.00032	13.702	0.949
100	5	10	8.2	15.21	0.00010	13.651	16.65	0.762	0.00045	15.857	0.971
120	5	10	8.2	16.61	0.00010	13.936	16.99	0.653	0.00034	16.926	0.944
140	5	10	8.2	17.60	0.00006	14.333	17.48	0.938	0.00021	18.424	0.957
100	5	5	4.502	13.9	0.00013	11.776	14.36	0.913	0.0005	12.004	0.995
100	5	15	11.811	15.6	0.00008	16.758	20.44	0.819	0.00025	16.679	0.989
100	5	20	15.521	16.2	0.00007	7.996	9.75	0.793	0.00027	16.657	0.982
100	10	10	8.2	11.93	0.00011	22.863	27.88	0.755	0.00022	10.687	0.868
100	15	10	8.2	7.85	0.00011	34.336	41.87	0.709	0.00033	8.683	0.907

Table 4 (b): Column results for adsorption of CR onto ECAC



Figure 8(b): Thomas plot-Variation in flow rate

(using eqn 3.32)

83.4



operation

95

3.5 Desorption Studies

A significant amount of dye was desorbed from the dye loaded carbon, since the adsorption process was mainly physical in nature. Maximum desorption was observed for the CR as 73.8% for ECAC under an optimum concentration of 0.40 N NaOH. A.Bennani Karim[32] et al indicated that the electrostatic forces between the adsorbent surface and adsorbate ions on an increase in ionic strength will decrease the adsorption capacity. The regeneration efficiency of desorbed ECAC and CAC was as good as to fresh carbon with regeneration efficiency was also more in iodine adsorption test and in MB adsorption test were listed in Table 5.

Tuble 5. Regeleration Efficiency Furameters of Se							
	Regeneration	Iodine	MB	Yield (%) after 5			
Carbon	efficiency (%)	adsorption	adsorption	cycles of			

test (%)

81

test (%)

85

Table 5: Regeneration Efficiency Parameters of SC

3.6 Reuse of spent carbon:

ECAC

(

In order to keep the desorbed carbon not another pollutant it was used for its constructive property mixed with cement was studied. Spent carbon (SC) was partially replaced in the ratio of 1%, 2%, 3% and 4%. Fresh concrete tests like physical, chemical characteristics, workability and hardened concrete tests (compressive strength) at the age of 7 days, 14 days, and 28 days were determined.

The raw materials used in the production of cement are finite and non-renewable and need to be conserved for future generations. To attain sustainable construction a strong trend favoring the increased use of admixture in concrete is emerging throughout the world. In this study, the spent carbon (SC) after 5 cycles of operation was used as an admixture. Replacement made of 0%, 1%, 2%, 3% and 4%. High % replacement of 4 % was considered.

Physical Property	Value
Specific gravity	2.12
Moisture content	18.84%

Table 6: Properties of Spent carbon:



Figure 9: EDAX observations of ECAC after adsorption

Chemical Constituent	Composition (%)
Carbon	53.10
Oxygen	40.71
Sodium	0.35
Magnesium	0.25
Silica	4.34
Calcium	1.27

From the Table, it was noted that SC is primarily characterized by the presence of carbon, oxygen, silica, calcium, etc. Presence of silica in SC makes it excellent pozzolanic material.

Table 8: Physical properties and ultimate strength of the Specimen.

	ing material, Dharath come		e
S.No.	Test	Results	IS code values as per IS12269:2013
1	Fineness Test	3.2 %	Not more than 10%
2	Soundness	0.5 mm	10 mm
3	Consistency	27 %	
4	Initial Setting Time	35 mins	30 mins
5	Final Setting Time	630 mins	600 mins
	Compression Strength	35.81 N/mm ² (7 days)	37 N/mm2
6	Test (Average of 5	44.22 N/mm ² (14 days)	
	Cubes)	46.31 N/mm^2 (28 days)	53 N/mm2

Testing material: Bharathi cement - OPC53 Grade + 4% SC

IV Conclusion

On the basis of present investigation the following experimental results were drawn:

- Eichhornia crassipes as an adsorbent can be used in wastewater treatment for removal of CR from solution
- The amount of dye adsorbed was found to vary with bed height, initial CR dye concentration and flow rate of dye solution

- In column kinetics studies, Bohart Adams model showed low regression coefficient values. Hence, the breakthrough capacity of adsorbates onto adsorbents at optimized conditions was found to be more favorable to Thomas model.
- Therefore, it is concluded that the adsorbent prepared from waste aquatic weed, Eichhornia Crassipes (EC) was found to be cost effective in removing color from industrial effluents and aqueous solutions of the dyes without leaving any secondary pollutant.
- From the desorption studies, it was found that the spent carbon can be repeatedly used for numerous cycle and further used as an alternative construction material. Therefore, it can be said that Eichhornia Crassipes would be an efficient and economical adsorbent.

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