Rice Husk Based Adsorbents for Textile Effluent Treatment

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Abstract: Adsorption of textile dyes onto rice husk based adsorbents from aqueous solutions was studied to enable comparison with alternative commonly available adsorbents. Batch experiments were conducted to determine the effect of method of treatment of rice husk on adsorption. Untreated and treated rice husk were tested for adsorption of Basic Green 4 and Acid Blue 80. The maximum adsorption was observed at solution pH values above 6 for Basic Green 4 and below 3 for Acid blue 80. Raw rice husk (RRH), base treated rice husk (BRH) and thermally treated rice husk (TRH) were superior to other adsorbents tested. Fixed bed column studies were conducted for RRH, BRH and TRH and breakthrough curves were obtained. Bed capacity, breakthrough time, length of the unused bed and time required for full bed exhaustion under ideal condition were calculated from breakthrough curves. Bed capacities for Basic Green 4 and Acid Blue 80 for thermally treated rice husk were 45 and 30 mg/g respectively. Experimental data obtained from unsteady state fixed bed column studies were used for scale-up calculations and for determination of industrial scale fixed bed adsorber dimensions.

Tests on real textile waste water consisting of a mixture of dyes showed that Biological Oxygen Demand (BOD) and Chemical Oxygen Demand (COD) levels can be reduced to 210 and 30 respectively using thermally treated rice husk.

1. Introduction

Textile, paper and dyeing industries continuously release dyes into water sources. Colour in water streams causes aesthetic and real hazard to the environment. The aesthetic value considers the discolorisation of recreational streams and waterways. The real hazard caused by colour in water is dye toxicity and the ability of the colouring agents to interfere with the transmission of light through water, thus hindering photosynthesis in aquatic plants. Removal techniques for dyes include coagulation, ozonation, membrane processes and adsorption [1, 2]. Among these, the adsorption process gives the best results as it can be used to remove different types of colouring materials. Further, adsorption is an efficient method for the removal of tracer components from water. Activated carbon-produced by carbonizing organic materials-is the most widely used adsorbent. However, the high cost of the activation process limits its use in wastewater treatment. Over the last few years, number of investigations have been conducted to test the low cost adsorbents for wastewater treatment. Rice husk [3,4], coir pith [5], tea and coffee waste [6,7], saw dust [8], and many other waste bio mass [9-13] have been tested for their adsorption characteristics

for dyes, heavy metals and other organic matter. The utilization of agricultural waste materials is increasingly becoming a vital concern because these wastes represent as unused resources and in many cases present serious disposal problems.

Rice husk (RH) is the waste material produced during rice processing. Approximately 20 kg of rice husk is produced when processing 100 kg of paddy. Sri Lanka has a potential of rice husk production of 830,000 MT/yr. Plant residuals such as rice husk are cellulose materials and have the potential to be converted into adsorbent. This conversion could contribute to reduce the volume of waste, while producing an adsorbent with a lower cost. Several researchers have investigated on dye removal using rice husk based adsorbents [3]. However, the work is limited to batch experiments and synthetic dye solutions. This work investigates the potential of rice husk based adsorbents for removal of colour in textile effluents in fixed bed columns.

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2. Materials and methods

Synthetic dye solutions and real textile wastewater samples were tested. The data obtained were used to determine an industrial scale adsorber plant.

2.1 Preparation of the adsorbent

Rice husk (RH) was obtained from local rice mills and Batalagoda rice research Institute. Soluble and coloured components were removed from husk by washing with water. This was repeated until the water was virtually

colourless. The rice husk was then washed with distilled water and oven dried for 12 hrs at 85 °C. The dried husk was sieved and stored in sealed polythene bags until use. Our preliminary tests has showed that small particles provide better adsorption capacities. However, handling very fine particles (<500 µm) in practice causes other problems and therefore, the intermediate size fraction between 500 -710 µm was used for all the experiments. The adsorbents were prepared by following methods as shown in Table 1;

Table 1: Methods of adsorbent preparation

Adsorbent	Method of preparation
1 Raw rice husk (RRH)	Grinding and sieving of the washed rice husk.
2 Physically treated rice husk	Impregnation of rice husk in 0.1 M sodium hydroxide solutions (BRH), 0.1 M Hydrochloric acid (ARH) for 6 hrs followed by water washing and oven drying.
3 Chemically treated rice husk	Impregnation of rice husk in 60% phosphoric acid solutions (PRH)
4 Thermally treated rice husk (TRH700)	Carbonizing of washed rice husk in the muffle furnace at 700°C for 1 hour.
5 Chemical- thermal treatment	Chemical treatment (3 above) followed by thermal treatment (4 above)- (TPRH).

Commercial granular activated carbon (GAC) provided by Haycarb Ltd. was used for the experiments for comparison purposes.

2.2 Waste water preparation

Basic Green 4 (Malachite Green-MG) and Acid Blue 80 (Nylosan blue-NB), commonly used dyes in Sri Lanka were used for all the experiments. Synthetic dye solutions were prepared by dissolving dye powder in distilled water to obtain required solution concentrations. Real waste water containing a mixture of dyes collected from a leading textile mill in Sri Lanka was tested.

2.3 Batch Experiments

A known weight of adsorbent and the adsorbate solution of known dye concentration were mixed using the orbital shaker (Stuart® Orbital Shaker) at 200 rpm for desired time period. The mixture was filtered through Whatman No. 1 filter paper and residual dye

concentration in the filtrate was determined using the calibration curve. Effect of initial solution pH and effect of adsorbent preparation method on adsorption were determined from batch experiments.

2.4 Fixed bed experiments

Fixed bed column adsorption experiments were conducted in a small 3 cm diameter glass column. The column was filled with a known weight of adsorbent to obtain the required bed height. The dye solution containing 30 mg/L of dye was fed to the column at a constant flow rate of 20 ml/min through the bed using a peristaltic pump. The solution leaving the bottom of the column was collected at various time intervals and the samples were analyzed using UV visible spectrophotometer. Preliminary tests showed that if the solution was passed through dry adsorbent, channeling of the adsorbent bed occurred, and pockets of adsorbent remain unutilized. Therefore, for fixed-bed experiments rice husk particles were

pre-wetted with distilled water, and the dye solution was passed through the wet rice husk bed. The batch and column adsorption experiments were performed in duplicate to observe the reproducibility and the mean value was used for each set of values.

3. Results and Discussion

Batch experiments showed that adsorption of MG is favoured at solution pH values above 6 and acidic solution conditions are favoured for NB. Therefore, solution pH values for all the experiments were maintained at 3 for NB and 6 for MG. Table 2 compares the effect of method of rice husk treatment on adsorption of dyes. BRH, RRH, and TRH were selected for fixed bed experiments comparing the degree of removal, amount of chemical used in adsorbent preparation and the cost of preparation.

3.1 Fixed bed column tests

Batch adsorption tests provide information on adsorption equilibrium characteristics and adsorption kinetics. However, continuous fixed-bed column operations are common for industrial adsorber design. Column experiments were conducted to understand the adsorption behavior in fixed bed columns, and to determine deviation from batch operations. Results on fixed bed column experiments are presented and discussed below.

Breakthrough curves for NB and MG adsorption onto RH based adsorbents from single dye solutions at 30 mg/l are shown in Figures 1 and 2 respectively. Typical 'S' shaped breakthrough curves were obtained for all experiments. However, results show that, the breakthrough curve is very slow once the concentration of the solution leaving the column reaches 25-28 mg/l due to conversion of the faster initial kinetics to the slower adsorptive kinetics. Hence, full bed exhaustion can not be achieved in a reasonable time. The area above the breakthrough curve is a measure of the bed capacity (BC). Breakthrough concentration was taken as 5% of the initial concentration. Therefore,

breakthrough time is the time required for the effluent to reach 5% of the initial concentration. Bed capacity, breakthrough time and length of the unused bed was calculated from breakthrough curves and the results are shown in Table 3. Time required for full bed exhaustion under ideal condition (T_s) was obtained by drawing ideal breakthrough curves [14].

3.2 Real Textile wastewater treatment

The batch experiments were conducted for the real textile waste water mixing with prepared adsorbents RRH, ARH700 and GAC. As the adsorbent dose is increased, removal of organic matter can be increased. Table 4 shows that the quality of treated water is almost within the standard limits of industrial effluents.

Fig 1: Breakthrough curve for NB, Flow rate 20 ml/min, bed height 10 cm

Fig 2: Breakthrough curve for MG, Flow rate 20 ml/min, bed height 10 cm

Table 2: Comparison of Treatment Methods for MG and NB dye removal

Type of adsorbent	Percentage of removal (%)		Adsorption capacity (mg/g)	
	NB	MG	NB	MG
ARH	46.1	50.0	3.46	3.75
BRH	81.1	99.5	6.08	7.46
RRH	46.1	76.8	3.46	5.76
PRH	79.9	70.5	5.99	5.28
PTRH	86.7	84.1	6.50	6.31
TRH700	81.1	97.8	6.08	7.34
GAC	86.7	99.6	6.50	7.50

Table 3: Bed capacity and other parameters calculated from breakthrough curves at Flow rate 20 ml/min, bed height 10 cm

	Adsorbent	Breakthrough time (hrs)	Ts (hrs)	LUB (cm)	Bed capacity (mg/g)
MG	RRH	1.48	4.82	6.93	11.97
	BRH	5.21	9.20	4.33	22.85
	TRH700	4.94	6.44	2.33	45.54
NB	RRH	1.19	3.26	6.35	8.09
	BRH	1.53	3.21	5.25	7.98
	TRH700	1.98	4.21	5.31	29.77

Table 4: Results of batch experiments with real waste water

Parameter	Waste water	Adsorbent	Treated water		Standard limit
			Dose (0.8g/200ml)	Dose (2g/200ml)	
pH	7.75	RRH	7.72	7.70	6.5 to 8.5
		TRH700	6.73	6.73	
		GAC	7.4	7.35	
COD mg/l	760	RRH	575	325	250
		TRH700	480	210	
		GAC	225	180	
BOD mg/l	100	RRH	50	35	30
		TRH700	45	30	
		GAC	27	25

Table 5: Summarized pilot scale parameters for fixed bed operation at 1000 l/h, 30 mg dye/l and breakthrough time of 48 h.

Dye	Adsorbent	Column diameter (cm)	column height (cm)
MG	RRH	88	102.57
	BRH	88	56.50
	TRH700	88	76.85
NB	RRH	88	153.76
	BRH	88	154.69
	TRH700	88	119.33

3.3 Scale-up calculations

Laboratory experimental results obtained in fixed bed adsorption column can be used for scale up calculations in industrial adsorber design. Industrial adsorber dimensions were estimated for the removal of NB and MG using RRH, BRH and TARH 700 at 1000 l/h and for a breakthrough time of 48 hrs. Results thus obtained for RRH, BRH and TRH700 are shown in Table 5. The cost of production of rice husk based adsorbents is 50% less than that of the commercial activated carbon.

4. Conclusions

Rice husk based adsorbents can be effectively used for textile dye removal from waste water. Raw rice husk, base treated rice husk and thermally treated rice husk showed good adsorption capacities for Basic Green 4 and Acid Blue 80. Thermally treated rice husk were equally good as commercial activated carbon. Real textile waste water can be treated to reach allowable standards using the RH based adsorbents. Fixed bed column test results were used for industrial column design. However, trials runs on real textile waste water in pilot plant scale fixed bed columns are required before implementation.

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