Optimization of Dye Removal from Textile Wastewater using Activated Carbon from Sawdust

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ABSTRACT: This study is aimed at developing an adsorbent from sawdust for optimum removal of dye from textile wastewater. The adsorbent was developed, characterised and, the adsorptive capability for the removal of dye was determined by optimizing the process parameters (adsorbent dosage, contact time and agitation speed) using Response Surface Methodology. The physical and chemical characterization of the effluent was carried out before and after the adsorption studies. From the results, a maximum adsorption capacity of 98.5 % was obtained at the optimized conditions of 1.5 g, 90 min and 275 rpm for adsorbent dose, contact time and agitation speed respectively. The ANOVA of the regression model showed that the model is highly significant with R^2 of 0.98. Further analysis carried out revealed that, in addition to dye removal, trace metals were also adsorbed in the process. This fact was established when the concentration of copper in the wastewater was found to decrease from 0.09 ppm to 0.03 ppm corresponding to 66.7 % removal at the end of the process.

KEYWORDS: Pollution, Wastewater, Textile, Adsorption, ICP-MS, Optimization, Trace metal

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I. INTRODUCTION

Water is one of the most important resources of man; however, this often gets polluted through the activities of man either directly or indirectly. With advancement comes growth in industrial activities and one very prominent industry is the textile industry where different types of fabrics are produced. This industry has been implicated in the pollution of both surface and groundwater depending on the depth of the groundwater (Hettige and Mowjood, 2015; Wang *et al.*, 2007). The textile industry has always been known to use large quantities of water and chemicals in its operations some of which eventually get discharged with the wastewater into the environment and mainly water bodies. Dyes which constitute a major component of these discharges are readily visible, even when present in small quantities because of their brilliance (Pang and Abdullah, 2013; Robinson *et al.*, 2001).

Industrial effluents containing synthetic dyes have been implicated in the reduction of light penetration in rivers and thus affect photosynthetic activities of the aquatic flora leading to a decrease in food for the aquatic organisms (Jin *et al.*, 2007; Pereira and Alves, 2007).

Many of these dyes are toxic and pose a serious hazard to aquatic animals and eventually man, who ends up consuming these. Wastewater containing dyes contains recalcitrant organic molecules, which are resistant to aerobic digestion, very stable to light, oxidizing agents and, very difficult to treat (Sun and Yang, 2003). Reactive dyes are used to dye cotton materials which make up about 50 % of the world's fibre consumption and, they are problematic as, they tend to pass through conventional treatment systems unaffected (Allègre *et al.*, 2006).

Different methods have been employed in the removal of colour from textile wastewater; whole bacterial cells have been employed in the removal of colour from textile wastewater (Pearce *et al.*, 2003). Other methods used include ozonation, chemical precipitation, flocculation, photolysis and ion-pair extraction and biological processes (Grant and Buchanan, 2000). Many of these are very expensive and require considerable start-up costs. Biodegradation process requires strict conditions to be maintained and sometimes can be very difficult to operate on a large scale (Bhole *et al.*, 2004).

Extensive research has been carried out on the removal of dyes from textile wastewaters using adsorption due to its simplicity and efficiency in the removal of pollutants that are too stable for biological methods (Kyzas *et al.*, 2013). Adsorbents used are varied such as charcoal, bentonite, silica, peat, natural zeolites, waste from agriculture; sawdust, fly ash, lignocellulosic materials, etc. The replacement of synthetic compounds with unconventional, natural or biological materials (green or environmentally friendly) has become the subject of considerable interest (Suteu *et al.*, 2008).

The modification of natural materials or agricultural by-products has shown promising results in improving their capacity and selectivity towards the removal of specific pollutants from various wastewater. There are several studies on the use of sawdust confirming the effectiveness of sawdust for the removal of dyes from wastewater; neem sawdust has been used to remove malachite green from dye via adsorption. The authors after varying different process parameters concluded that the neem sawdust shows significant adsorption capacity for malachite green under suitable experimental

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conditions (Khattri and Singh, 2009). The removal efficiency of activated carbon prepared from coir pith for three highly reactive dyes has been investigated.

The kinetics of the removal was studied and, it was concluded that the activated carbon prepared from coir pith was a suitable adsorbent for the removal of reactive dyes from both synthetic and textile effluents (Santhy and Selvapathy, 2006). Activated carbon prepared from mahogany sawdust has been confirmed effective for the removal of direct dyes (Malik *et al.*, 2007); Oil palm and fibre husk activated carbon has been used in the adsorption of basic dyes. The kinetics, equilibrium and thermodynamic studies have shown the effectiveness of these otherwise waste materials in the removal of the dye (Tan *et al.*, 2008; Tan *et al.*, 2007). The optimization of the adsorption of reactive dyes from textile wastewater using readily available, sawdust modified with Al^{3+} to remove dyes is not available in the literature. This study examines the treatment of effluent from a textile industry in Kano State, Nigeria.

This research aims to prepare a modified adsorbent from sawdust for the optimal removal of dyes from the textile wastewater, and the specific objectives are to:

- Develop and characterize the adsorbent prepared;
- Optimize the process variables (adsorbent dosage agitation speed and time) for the optimal removal of dyes from the textile wastewater; and
- Determine the adsorptive capacity of sawdust for the removal of dyes from textile wastewater.

II. MATERIALS AND METHODS

A. Materials

The textile wastewater used in this research was collected from a textile industry in Kano, Nigeria while the sawdust samples was collected from a sawmill in Ilorin, Kwara State, Nigeria. Aluminium sulphate, $Al_2(SO_4)_3$, was obtained from a chemical store in Ilorin, Kwara State. The equipment and apparatus used include Atomic Absorption Spectrophotometer (AAS), a jar test apparatus, an Inductively Coupled Plasma – Mass Spectrophotometer (ICP-MS), a muffle furnace, a turbidimeter, a mechanical shaker, a weighing balance, a set of sieves, and glassware.

B. Sample Collection and Pretreatment

The collected sawdust sample was sieved to obtain particles that will pass through a 1 mm sieve size. The particles were washed with distilled water to remove the surface adhering particles and were sun-dried for 48 h to reduce the moisture content.

C. Preparation of Adsorbent

The prepared sawdust was calcined in a muffle furnace (Carbolite CWF 1200) at 500^{o}C for one hour (Malik *et al.*, 2007). An activation solution was prepared by dissolving 2.0 g of $Al_2(SO_4)_3$ in 100 ml of distilled water. This solution was added to 10.0 g of the calcined sawdust and, shaken on a mechanical shaker (HY-4 Reciprocated shaker, Zenith Lab).

Thereafter, the mixture was left for 12 h, filtered, and washed several times with distilled water until the pH was 7.0. The activated sawdust was then dried in an oven at 100^{o}C untill a constant weight was attained; this was then cooled in a desiccator.

D. Characterization of Adsorbent

The adsorbent (activated sawdust) was characterized using Fourier Transform Infrared (FTIR) Spectroscopy Technique to determine surface functional groups present. The FTIR analyses before and after dye adsorption were carried out on the samples using Shimadzu FT-IR-8400S Spectrophotometer with a resolution of 4 cm^{-1} in the range of 4000 - 500 cm^{-1}.

E. Effluent Characterization

The collected textile wastewater was used to evaluate the effectiveness of the prepared activated carbon moved dye from textile wastewater. The pH, the turbidity, the colour and the presence of trace metals in the textile effluent was determined before and after the adsorption process. The physical properties of the textile effluent such as the pH, and the turbidity were analyzed using a digital pH meter (Model KL-03l) and a turbidimeter (HACH 2100NTU Turbidimeter) respectively.

The presence of trace metals was established using an inductively coupled plasma mass spectrometry (ICP-MS) and an atomic absorption spectroscopy (AAS). The Inductively Coupled Plasma Mass Spectrometer (ICP-OES iCAP6500 DUO, Thermo Scientific, UK) equipped with a charge injection device (CID) was used in the determination of metal ions present in the wastewater sample. The wastewater analysis using ICP-MS was carried out in the Analytical/Environmental Laboratory at the University of Johannesburg, South Africa. The specifications at which the ICP-MS operated are as shown in Table 1.

Table 1: Operating Conditions of the ICP-MS.

Parameters	Condition
RF generator power (W)	1150
Coolant gas flowrate ($Lmin^{-1}$)	12
Carrier gas flow rate ($Lmin^{-1}$)	0.7
Auxiliary gas flow rate ($Lmin^{-1}$)	1.0
Pump rate (rpm)	50
Plasma mode	Axial
Replicate	3

F. Design of Experiments

The central composite design (CCD) in response surface methodology was used to carry out the optimization of the studied parameters. The desirability (D) function was employed to obtain the optimum conditions to achieve better response in terms of the dye removal efficiency (Adewoye *et al.*, 2017). Three independent variables; the adsorbent dosage, the contact time and the agitation speed were used in designing the experimental matrix. Each numeric factor was minimum and maximum (factorial points) and the center-point. For a three-factor scenario where n is equal to three (3), the small CCD is characterized by 10, not center points and 5, center points resulting in a total of 15 experiments. The minimum and maximum values for each factor investigated are presented in Table 2.

Table 2: Experimental ranges for the Central Composite Design.

Factors	Variable	Unit	Minimum	Center	Maximum
A	Adsorbent dose	g	0.5	1.5	2.5
B	Contact time	min	30	75	120
C	Agitation Speed	rpm	100	200	300

Design expert version 6.0.8, (Stat – Ease, Inc., Minneapolis, MN 55413, USA) statistical software was used for the model fitting and for the evaluation of the statistical significance of the dye adsorption efficiencies. The dye removal efficiency was calculated using eqn (1).

$$\%\ Dye\ Removal = \frac{(TB_o - TB_i)}{TB_o} * 100\% \quad (1)$$

where, TB_o and TB_i (NTU) are the turbidity of the wastewater at initial stage and at equilibrium.

G. Adsorption Experiments

The removal of dye from the textile wastewater was carried out using the batch method at room temperature. The adsorption studies were carried out for different contact times, adsorbent dosages and agitation speeds. The different combinations resulting in 15 batch experimental runs as determined by the design of experiments using design expert software were followed. The batch experiments were carried out by adding each dosage of activated sawdust (0.5-2.5 g) to 100 ml of the wastewater sample and agitated using the jar test apparatus for the duration of contact time (30 -120 min) and agitation speed (100 - 300 rpm).

The mixture was filtered and the filtrate was analyzed for the extent of dye removal using a turbidimeter. The run with the highest dye removal was then utilized for the subsequent studies.

H. Effect of Contact Time and Adsorbent Dosage

The effect of contact time on dye removal was investigated by performing the experiment at an agitation varied over 5 levels: plus and minus alpha (axial points), speed of 200 rpm for an adsorbent dosage of 2.0 g and varying the experimental runs for periods of 0, 60, 90, 120 min respectively

The effect of adsorbent dosage on dye removal was investigated by performing the experiment at an agitation speed of 200 rpm for a period of 75 minutes and using absorbent dosages of 0.5, 1.0, 1.5, 2.0, 2.5 g respectively.

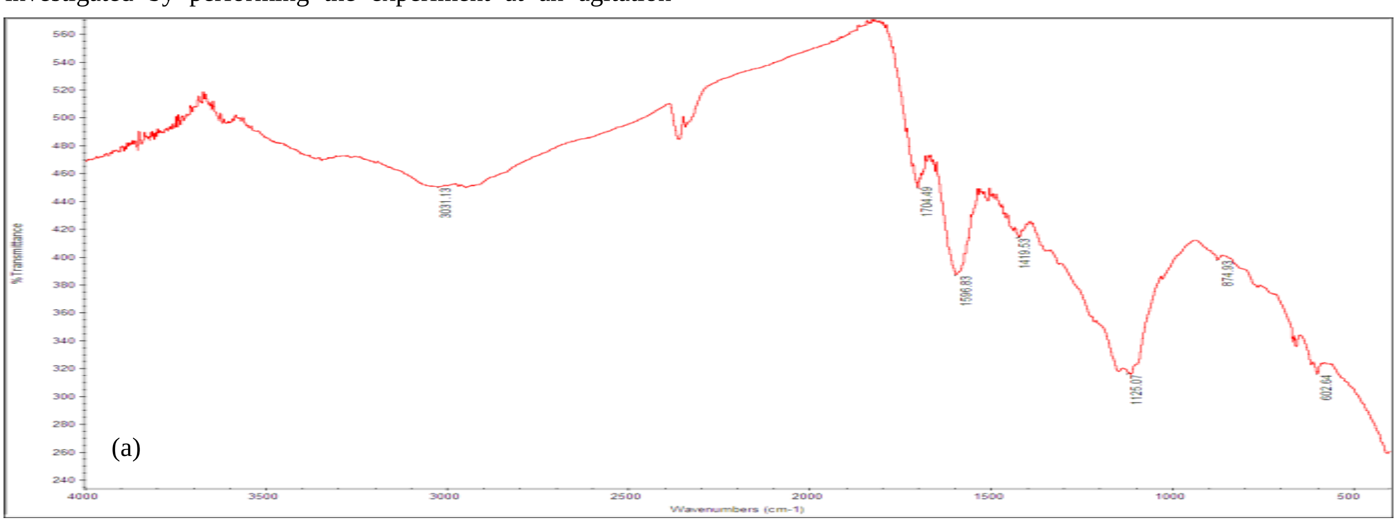
III. RESULTS AND DISCUSSION

A. Characterization of Adsorbent

The infrared spectra of the carbonized-activated sawdust sample before and after adsorption of dye molecules were determined on a Shimadzu FT-IR-8400S spectrophotometer, in the range 400-4000 cm^{-1}. Each sample was prepared using potassium bromide, (KBr). The infrared spectra of the two samples (Figure 1) did not show much difference in the region between 3500 and 3000 cm^{-1}assigned to O-H stretching vibration as they both show very weak peak at the region. The N-H stretching vibration was absent as no complementary N-H bending was observed as expected between 1650 and 1600 cm^{-1}.

The aromatic and aliphatic C-H stretching were equally observed at 3031-2850 cm^{-1} as weak peak in both samples. Moisture peak was present in both samples at 2363 cm^{-1} however; there was a sharp peak at 1736 cm^{-1} corresponding to carbonyl stretching of ketone, aldehyde, carboxylic acid or esters in the raw sample before the adsorption which was much reduced in the adsorbed sample. Both samples showed prominent peak of C=C unsaturated approximately 1600 cm^{-1}.

The peak at 1438 cm^{-1} corresponding to O-H bending vibration was shifted to a lower frequency 1419 cm^{-1} in the adsorbed sawdust. The out-of-plane bending vibration at 1365 cm^{-1} (sharp) of the C=C bond had also disappeared in the adsorbed sample probably due to some chemical reactions with the dye molecules. In the fingerprint region, the untreated sample also depicted twin sharp peaks at 1230 and 1216 cm^{-1} which were absent in the adsorbed sample. Generally, the sawdust that adsorbed the dye exhibited less vibrational peaks which may be due to the interactions of the dye molecules with the available functional groups.



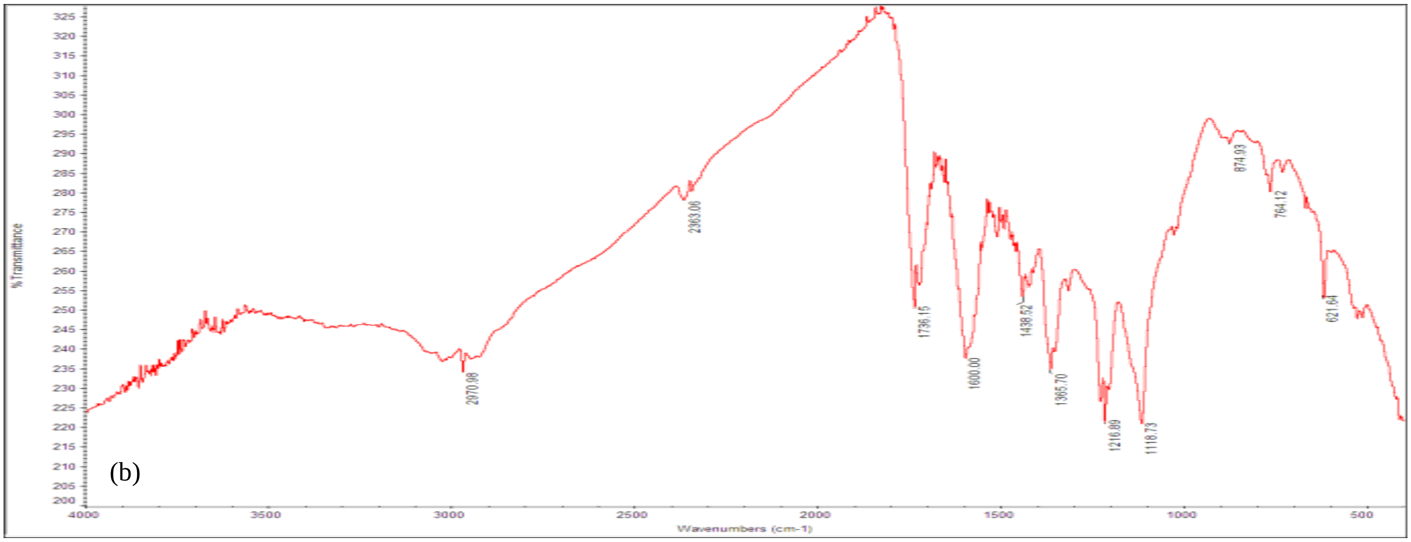


Figure 1: FT-IR Characterization (a) Before Adsorption (b) After Adsorption.

B. Effluent Characterization

Trace metal analysis was carried out on the raw wastewater sample using an ICP-MS (ICP-OES iCAP6500 DUO, Thermo Scientific, UK) equipped with a charge injection device (CID**).** The metals detected in appreciable concentrations were Zn (43.48 ppb), Cu (443.60 ppb), Fe (65.40 ppb), Mn (55.48 ppb) and Ni (153.92 ppb) as shown in Table 3. Further analyses were performed on the afore-mentioned elements using an AAS at the Central Research Laboratory at the University of Ilorin, Nigeria. Four (Zn, Fe, Mn and Ni) of the five metals selected based on the significance of their concentration in the raw wastewater (Table 3) were however, found to be below the detection limit of the AAS machine (detection limit: 0.01 ppm); except for Cu with concentration of 0.09 ppm.

Table 3: Result of raw wastewater analysis from ICP-MS.

Trace metal	Value (ppb)
Cd	0
Co	1.40
Cr	10.84
Cu	443.60
Fe	65.40
Pb	0
Mn	55.48
Ni	153.92
Ti	2.33
Zn	43.48

C. Design of Experiments

The design of experiments comprising of the studied factors, their ranges and the response (dye removal efficiency) is presented in Table 4. The response in terms of dye removal efficiency was in the range of 44.0 % to 79.9 %.

Table 4: Experimental design matrix for dye removal onto activated sawdust.

Run	Independent Variables (Factors)			Dye removal, $Y_{dye\text{-}removal}$ (%)
	Adsorbent Dosage (g)	Contact time (min)	Agitation Speed (rpm)	
1	1.5	75	200	74.7
2	2.0	75	200	79.9
3	1.5	75	200	74.6
4	1.5	52.5	200	55.8
5	1.5	75	150	74.0
6	1.5	97.5	200	76.6
7	1.5	75	200	74.2
8	1.5	75	200	74.3
9	0.5	30	100	44.0
10	1.5	75	200	74.8
11	1.5	75	250	78.6
12	2.5	120	100	77.0
13	0.5	120	300	73.6
14	1.0	75.0	200	55.0
15	2.5	30	300	52.4

The experimental data were analyzed using CCD and the final empirical model equations showing the relationship between the adsorption process variables and the response was developed as shown in eqn (2).

$$Y_{dye_{removal}} = +72.63 + 24.90A + 20.80B + 4.60C - 13.83A^2 - 18.83B^2 + 21.57C^2 + 3.25AB + 7.25AC + 21.95BC \quad (2)$$

where A, B and C represents the adsorbent dosage, the contact time, and the agitation speed respectively.

The developed polynomial model equation shows that, the adsorbent dosage (A), contact time (B) and agitation speed (C) have a positive influence on the removal of dye from the wastewater onto the activated sawdust. It also reveals that the interactions of the adsorbent dosage and the contact time (AB), the adsorbent dosage and the agitation speed (AC) and the contact time and the agitation speed (BC) all have positive influence on the adsorbance. Of all the terms in the model, only (A^2) and (B^2) have negative influence on the adsorption of dye on the activated sawdust. The adequacy and significance of the developed model was evaluated using F-test and analysis of variance (ANOVA) as shown in Table 5. From Table 5, the terms A, B, B^2, C^2, and BC were found to be the signficant model terms while C, A^2, AB, and AC were insignificant for the dye removal model. A good correlation existed between the experimental and predicted data with an R^2 value of 0.98. This shows that the developed model is reliable.

Table 5: ANOVA of response surface quadratic model for dye removal.

Source	Sum of Squares	Degree of Freedom	Mean Square	F-Value	Prob>F	
Model	1764.85	9	196.09	27.15	0.0010	*Significant
A	310.01	1	310.01	42.93	0.0012*	
B	216.32	1	216.32	29.96	0.0028	
C	10.58	1	10.58	1.47	0.2802	
A^2	35.59	1	35.59	4.93	0.0771	
B^2	65.98	1	65.98	9.14	0.0293	
C^2	86.56	1	86.56	11.99	0.0180*	
AB	4.99	1	4.99	0.69	0.4438	
AC	23.36	1	23.36	3.24	0.1320	
BC	214.13	1	214.13	29.65	0.0028*	
Residual	36.11	5	7.22			

* Values of "Prob > F" less than 0.0500 indicate model terms are significant.

D. Optimization of Process Parameters

The optimization of process parameters for the removal dye onto activated sawdust was carried out using design expert software. The target goal of the optimization process was set to maximize dye removal within the experimental range of the studied independent variables. The solution of optimization is usually selected based on the highest desirability or its closeness to unity (Adewoye *et al.*, 2017).

The optimal conditions obtained for adsorbent dose, contact time, and agitation speed were 1.5 g, 90 min, and 275 rpm respectively. The dye removal efficiency at optimum conditions was found to be 98.5 %. The model validation was carried out and a good agreement existed between the predicted value and the experimental value with relatively small error of 2.3 % as shown in Table 6. Figure 2 (a – c) shows the combined interaction of the three studied parameters. The curvatures obtained for all the response surface plots clearly indicated that all the variables, either individually or by way of interaction contributed to the dye removal. However, the level of contributions may differ.

Table 6: Model Validation.

Adsorbent Dosage, A (g)	Contact Time, B (min)	Agitation Speed, C (rpm)	Dye Removal Efficiency, $Y_{dye\ removal}$ (%)		
			Predicted	Exptl	Error (%)
1.50	90.00	275.00	98.50	96.20	2.30

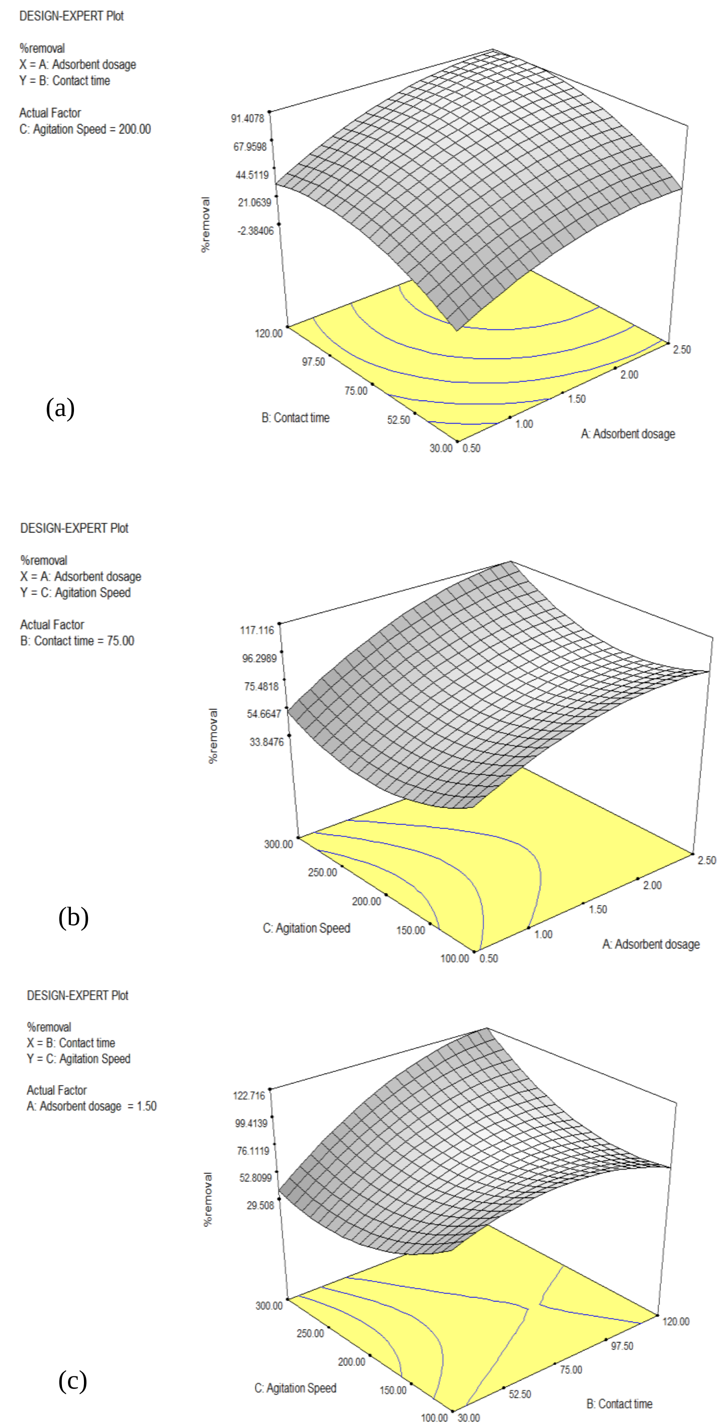


Figure 2: 3D response surface plots: (a) effect of adsorbent dosage and contact time on dye removal (b) effect of adsorbent dosage and agitation speed on dye removal (c) effect of contact time and agitation speed on dye removal.

At the optimum conditions of adsorbent dose, contact time and agitation speed established, the treated effluent was analyzed before and after the adsorption studies. The pH and turbidity were determined in triplicate and the average values were noted. After the adsorption studies with Al^{3+} activated sawdust, the pH of the wastewater was found to decrease from

8.88 to 7.24 while the turbidity was reduced from 7.90 to 0.3 NTU which translated to 96.2 % dye removal from the wastewater. Trace metal analysis was carried out on the raw wastewater sample and treated sample at optimum condition using the AAS. While all other metals was found to be below detection limit of the AAS available, there was a reduction in the concentration of Cu to 0.03 ppm showing 66.67 % removal of Cu which confirms that, besides dye removal, the adsorbent (activated sawdust) was also effective in the removal of trace metals from textile wastewater.

E. Adsorption Experiments

Effect of Adsorbent Dosage

Figure 3 shows that dye removal occurs with an increase in adsorbent dosage until at adsorbent dosage of 2.0 g at which the dye removal was maximum (80% removal). This can be attributed to the increase in surface area and number of active sites and with further increase, there was an observed fall in the percentage removal of dye due to an increase in adsorbent dosage beyond maximum adsorption capacity which might be as a result of overlapping of the adsorption sites due to overcrowding of adsorbent particles beyond the optimum dose (Garg*et al.*, 2004).

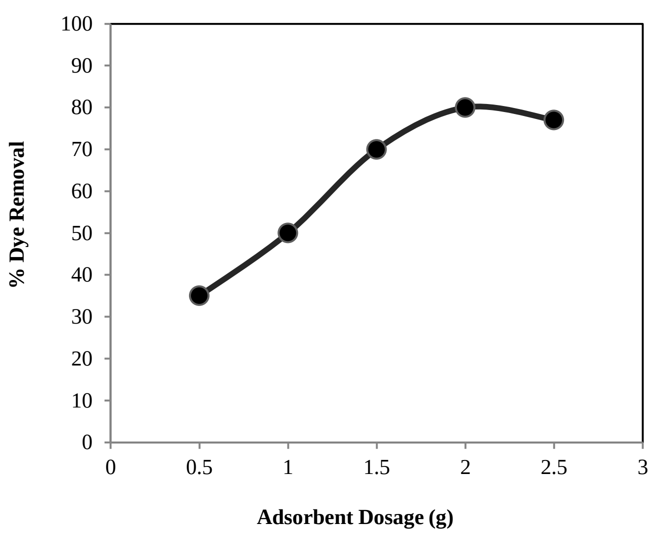


Figure 3: Effect of Adsorbent Dosage on Dye Adsorption Using Sawdust Activated Carbon [Ambient Temperature = 30 ^oC; Agitation speed = 200 rpm; Contact time = 75 mins].

Effect of Contact Time

The percentage of dye removal as a function of time shown in Figure 4 indicated that, the percentage was found to increase from 32.7 - 75.3% as the contact time increases from 30 - 120 min at 15 min interval. The maximum removal was 75.3 % corresponding to contact time of 85 min. In the batch type adsorption processes, the monolayer of adsorbate is normally formed on the surface of adsorbent and the rate of removal is controlled primarily by the rate of transport of the adsorbate species from the exterior/outer site to the interior sites of the adsorbent particles (Kannan *et al.*, 2001).

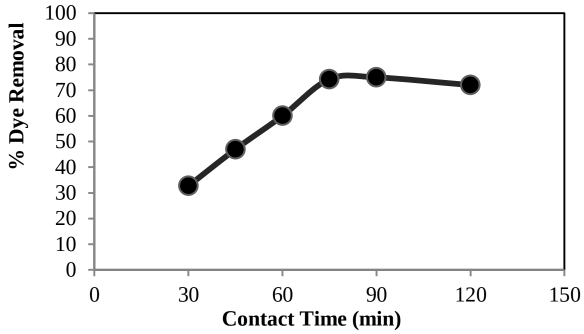


Figure 4: Effect of contact time on dye adsorption using sawdust activated carbon [Ambient Temperature = 30 ^oC; Agitation speed = 200 rpm; Adsorbent dosage = 2 g].

IV. CONCLUSION

The activated carbon prepared from sawdust shows significant adsorption capacity for the removal of dyes from textile effluent treatment processes under suitable experimental conditions. The process was optimized, and the maximum dye removal of 98.5% was achieved at optimum conditions of 1.5 g, 90 min, and 275 rpm for adsorbent dose, contact time, and agitation speed respectively. The ANOVA response shows the reliability of the data with a good correlation coefficient R^2 of 0.98. It was also found that aside dye removal, the activated sawdust was also effective in the removal of trace metals. Sawdust is readily available at negligible cost and hence will serve as a useful low cost and environmentally benign adsorbent. The result will be useful for designing and fabricating an economically cheap treatment process plant for the removal of dyes and trace metals from industrial effluents.

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