

SEQUESTRATION OF BASIC DYE FROM TEXTILE INDUSTRY WASTE WATER USING AGRO-WASTES AND MODELING WITH ANOVA

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ABSTRACT

In the present study agro-waste products were used for sequestration of basic dye from waste effluent of textile industry. Agricultural wastes products are quite commonly distributed as the result of agricultural practices. They are inexpensive and subject to biodegradable. Agricultural wastes are a good source for the adsorption of the dyes generated during the textile processing. For the process of adsorption, agricultural waste products are used in the modified form through activation with conc.H₂SO₄. This article focuses on the Areca Husk Carbon (AHC) and its adsorption capacity of Bismark Brown Y (BBY) dye. The mathematical tool ANOVA, adsorption isotherm and kinetics parameters are attempted effectively to optimize the potential of the use of agricultural waste products for removal of toxic pollutant from the effluent discharging into the water bodies.

Keywords: Sequestration, Basic Dye, Textile Industry, Waste water, Agro waste, Modeling, ANOVA

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INTRODUCTION

Synthetic dyes are extensively used in industries such as textiles, printing, paper, pharmaceutical, Kraft bleaching, food technology, hair coloring, plastics and cosmetics, etc. Textile industry causes considerable higher impacts to water pollution by discharging their effluents into various receiving bodies includes ponds, rivers and other public sewer. Major pollutant load from the textile industries are from the several of their wet-processing operations like scouring, bleaching, mercerizing and dyeing¹. Among these various processes, dyeing process normally uses large amounts of water for dyeing, fixing and washing processes². Thus, textile waste water contains large amounts of suspended solids, strong colour, pH, high temperature and low biodegradability caused by varying contaminants within water environment³.

The textile industry is currently facing severe pressure to reduce colour in wastewater from dyeing and finishing operations. Since dyes are used in the textile manufacturing it is imperative that the effluent generated by the textile industry need to be treated and discharged if not recycled within the industry. In view of this it is very essential that existing water bodies need to be conserved from the effect of pollution and the concept of reuse, recycle and reduce have been very well conceived by the industries, yet effort in that direction needs to be accelerated.

There are several methods used for textile effluent treatment of dye containing wastewater. Some of them involve reverse osmosis⁴, chemical oxidation⁵, photodegradation⁶, electrocoagulation⁷ and adsorption⁸. Some works of low cost non-conventional adsorbents have been carried out. Adsorbents used include agricultural solid wastes such as coconut husk⁹, coir pith¹⁰. Non-conventional material like red mud¹¹, silica fumes¹², fly ash¹³ and saw dust¹⁴.

EXPERIMENTAL

Preparation of activated carbon adsorbent

One part by weight of each powdered raw material was chemically activated by treating with two parts by weight of concentrated sulphuric acid with constant stirring and was kept for 24 hours in a hot air oven at 75°C, the carbonized material was washed well with plenty of water several times to remove excess acid, surface adhered particles, water soluble materials dried at 200°C in hot air oven for 24 hours. The adsorbent thus obtained were grounded well and kept in air tight containers for further use.

Analysis of Bismark Brown Y

The concentration of BBY in the supernatant solution after and before adsorption was determined using a double beam UV spectrophotometer (Shimadzu, Japan) at a wavelength of 516 nm. It was found that the supernatant from the activated carbon did not exhibit any absorbance at this wavelength and also that the calibration curve was very reproducible and linear over the concentration range used in this work. The physical properties and chemical structure of BBY were shown in fig.-1 and table-1.

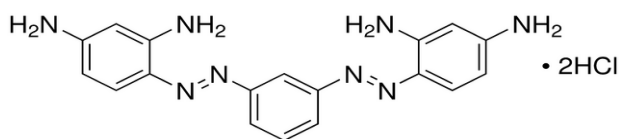


Fig.-1: Chemical structure of Bismark Brown Y.

Table-1: Physical properties of Bismark Brown

Dye name	Bismark Brown Y
C.I number	21000
Chemical formula	C ₁₈ H ₁₈ N ₈ · 2HCl
Molecular Weight	419.31 g mol ⁻¹
Wave length	516 nm
Solubility in water	1 g / 25 ml

Batch equilibrium studies

Batch experiments were carried out by shaking 100 ml of dye solution with 200 mg of adsorbent in a glass stoppered conical flask at a temperature at 30°C at the rate of 120 rpm. After agitation the solution centrifuged. Then the dye concentration in the supernatant solution was analyzed using a spectrophotometer by monitoring the absorbance changes at a wavelength of maximum absorbance (516nm) in these sorption experiments, the solution pH was used without adjusting.

Each experiment was carried out and average results are presented. Calibration curves were obtained with standard BBY solution using distilled water as a blank. Mass capacity of adsorption q_e , is calculated from the difference between the initial and final concentration of BBY.

$$q_e = \frac{(C_0 - C_e)}{W} V \quad (1)$$

where C_0 and C_e (mg L⁻¹) are the liquid-phase concentrations of dye at initial and equilibrium respectively. V is the volume of the solution (l), and W is the mass of dry adsorbent used (g).

RESULTS AND DISCUSSION

Influence of Adsorbent dosage and Particle size

Adsorbent dose is an important parameter influencing adsorption process since it determines the adsorption capacity of an adsorbent for a given initial dye concentration of the adsorbate at the operating conditions. Adsorbent dose on removal of BBY were studied in the range of 50–250 mg L⁻¹. Fig.-2 showed that the % removal of dye increased from 27% to 88%, 35% to 94% and 38% to 97% for the

particle size of 100, 150 & 250 BSS mesh as adsorbent dose increased from 50 to 250 mg L⁻¹. The study shows enhancement of BBY removal with the decrease of particle size of the adsorbent attributed to the increase in the surface area. Such a trend is attributed to an increase in the adsorptive surface area and the availability of more binding sites. Further increase in adsorbent dose, did not show significant increase in % removal of dye, therefore 200 mg L⁻¹ adsorbent dose was chosen for the successive experiments.

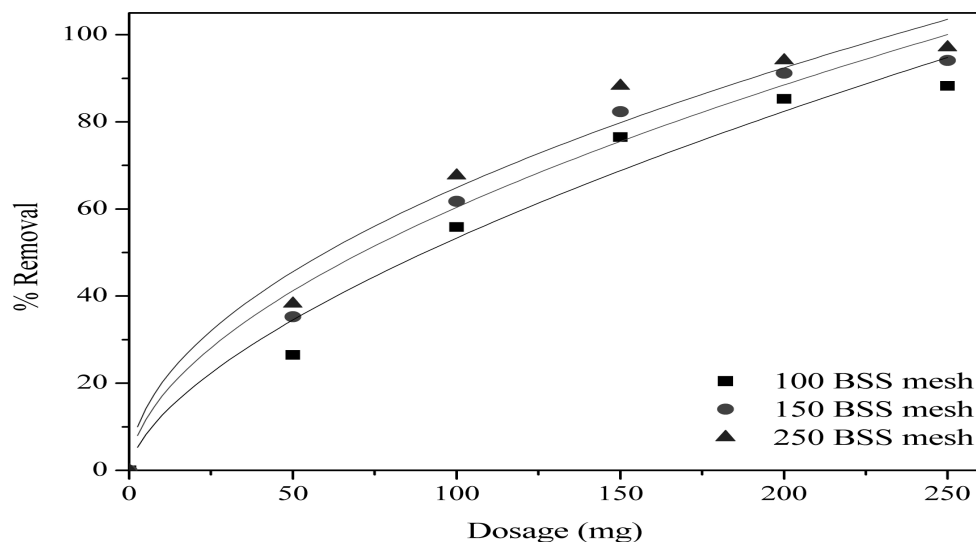


Fig.-2: Influence of adsorbent dosage on the removal of BBY dye onto AHC [dye concentration = 10 mg L⁻¹; contact time = 3 hrs; agitation speed = 120 rpm; T = 30°C; pH 7; Particle size = 100 – 250 BSS mesh].

Influence of Contact time

The influence of contact time on removal of dye was shown in Fig.-3. It is clear that the extent of adsorption is rapid in the initial stages and becomes slow in later stages till saturation is allowed. The final dye concentration did not vary significantly after 3 hours from the initial stage of adsorption process. This shows that equilibrium can be assumed to be achieved after 3 hours of contact time was found sufficient to acquire equilibrium. It is basically due to saturation of the active sites which does not allow further adsorption. The adsorption rate was found to decrease with increase in time.

Influence of Initial dye concentration

The adsorption of BBY on AHC was studied at different BBY initial dye concentrations (5-20 mg L⁻¹). Fig.-4 shows the result of influence of initial dye concentration on adsorption of BBY onto AHC. It is observed that dye uptake is rapid for the first 15 min and finally attains saturation. Fig. 5 shows that an increase in initial BBY concentration results in increase in the adsorption of BBY on AHC. Thus equilibrium removal of BBY gets decreased from 96% to 90% with an increase in the initial BBY concentration from 5 to 20 mg L⁻¹. However, the experimental data are measured at 180 min to make sure that a complete equilibrium is attained.

Influence of Initial pH

It is an important parameter for the adsorption of BBY from aqueous solutions that affects the form and quantity of dye, adsorbent surface sites in water, and the interaction between BBY and the functional groups on the adsorbent surface. Influence of pH on the adsorption of BBY to AHC was evaluated by using initial dye concentration of 10 mg L⁻¹ at the pH range of 2–12, and the results are illustrated in Fig.5. The amount of BBY removal was found to be increased from 83 to 94 with an increase in pH.

When pH was increased, electrostatic repulsion between BBY and adsorbent surface sites and competing effect of hydronium ions were decreased, So BBY removal was increased. The optimum pH was established as 7.0.

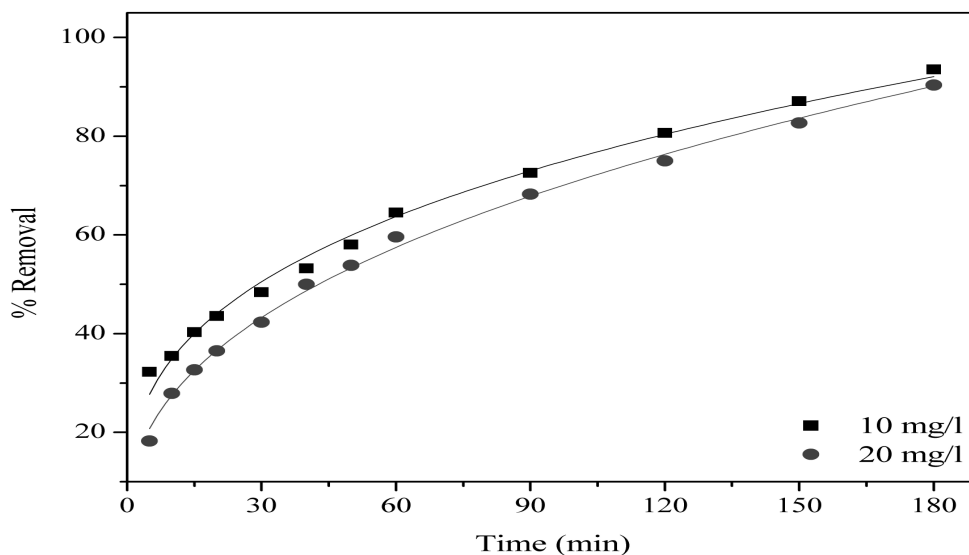


Fig.-3: Influence of contact time on the removal of BBY dye onto AHC [dye concentration =10mg L⁻¹; contact time=3 hrs; sorbent dosage=200 mg; agitation speed=120 rpm; T=30°C; pH 7; Particle size= 250 BSS mesh].

Adsorption kinetic study

In order to investigate the adsorption processes of BBY on AHC, three kinetic models were used, including pseudo-first order, pseudo-second order and intra-particle diffusion models.

Pseudo-first order model

The linear form of pseudo-first-order model was described by the following equation¹⁵ -

$$\frac{dq_t}{dt} = k_1 (q_e - q_t) \quad (2)$$

Where q_t is the amount of adsorbate adsorbed at time t (mg g⁻¹), q_e is the adsorption capacity in equilibrium (mg g⁻¹), k_1 is the rate constant of pseudo-first-order model (min⁻¹), and t is the time (min). After definite integration by applying the initial conditions $q_t = 0$ at $t=0$ and $q_t = q_t$ at $t=t$, the eq.(2) becomes-

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (3)$$

Values of adsorption rate constant (k_1) for BBY adsorption on AHC were determined from the plot of $\log(q_e - q_t)$ vs t . These values presented in Table 1 indicate that the adsorption rate was very fast at the beginning of adsorption and that rate of removal of BBY is faster on AHC. k_1 value of 0.01539 min⁻¹ and 0.273 min⁻¹ for 10 and 20 mgL⁻¹ respectively. The equilibrium adsorption capacities were 4.68 and 9.52 mg g⁻¹ respectively. The calculated equilibrium adsorption capacities were 5.89 and 7.30 mg g⁻¹. The calculated and experimental results reveal that, the pseudo-first order model provided a better approximation to the experimental kinetic data than the pseudo-second order model for adsorption of BBY from aqueous solution.

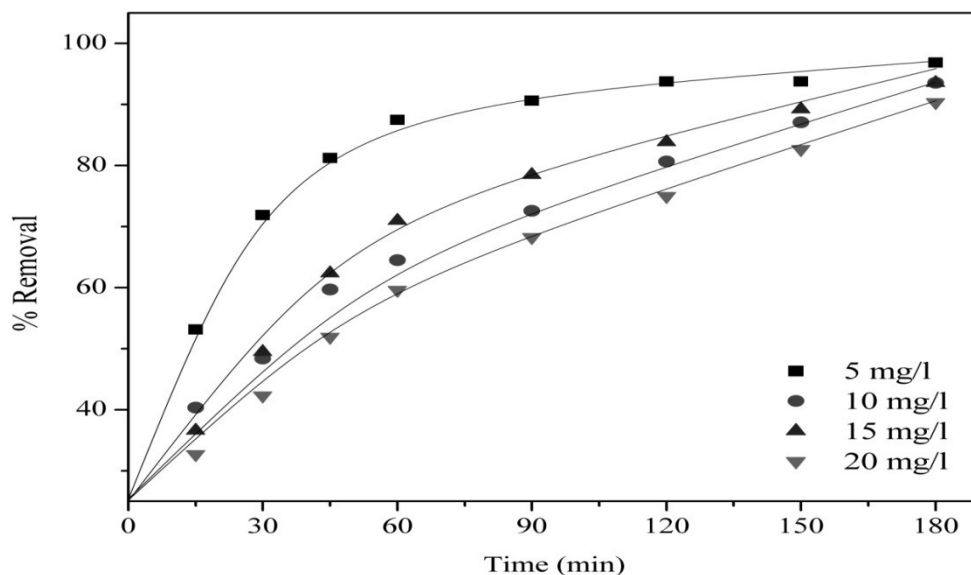


Fig.-4: Influence of Initial dye concentration on the removal of BBY dye onto AHC [contact time=3 hrs; sorbent dose=200 mg; agitation speed=120 rpm; T=30°C; pH 7; Particle size=250 BSS mesh].

Pseudo-second order model

The pseudo-second order adsorption kinetics¹⁶ can be written as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

Where k_2 is the rate constant of adsorption ($\text{g mg}^{-1} \text{min}^{-1}$), q_e and q_t are the amount of dye adsorbed at equilibrium and at time t . (mg g^{-1}). The values of k_2 and q_{cal} were calculated from the intercepts and slopes of the plots of t/q_t vs. t (Fig. 7) are presented in Table 1. The calculated q_e values computed from pseudo-second order equation don't show good agreement with experimental values, indicating that adsorption does not follow pseudo second order kinetic model.

Table-2: Kinetic Parameters for the adsorption of BBY dye onto AHC

Kinetic model	Model coefficients		
		$C_0 = 10 \text{ mg L}^{-1}$	$C_0 = 20 \text{ mg L}^{-1}$
Pseudo- first order	$q_e (\text{mg g}^{-1})$	4.68	9.52
	$q_{\text{cal}} (\text{mg g}^{-1})$	5.89	7.30
	$k_1 (\text{min}^{-1})$	0.0054	0.0061
	R^2	0.9872	0.9897
Pseudo-second order	$q_{\text{cal}} (\text{mg g}^{-1})$	0.43	1.039
	$k_2 (\text{g mg}^{-1} \text{min}^{-1})$	0.1967	0.103
	$h (\text{mg g}^{-1} \text{min}^{-1})$	5.70	0.08
	R^2	0.9762	0.9964
Intra-Particle diffusion	$k_{\text{int}} (\text{mg g}^{-1} \text{min}^{-0.5})$	0.2815	0.3098
	C	0.9285	5.4237
	R^2	0.9970	0.9906

Intra-particle diffusion model

To identify the importance of pore diffusion in the adsorption process, mathematical expression of intra-particle diffusion model¹⁷ was used-

$$qt = K_{int}t^{0.5} + C \quad (5)$$

Where k_{int} is the intraparticle diffusion constant ($\text{mg g}^{-1} \text{min}^{-0.5}$) and the intercept (C) reflects the boundary layer effect. The value of k_{int} was calculated from the slope of the plot of q_t vs. $t^{0.5}$ (Fig. 8) are presented in Table 1. The calculated value k_{int} of 0.28 to 0.31 $\text{mg g}^{-1} \text{min}^{-0.5}$ and C was found 0.9 to 5.4. The correlation coefficient (R^2) values of 0.9608 and 0.9869 for the AHC. The high R^2 value indicates that intra-particle diffusion might play a significant role in the initial stage of the adsorption. The value of intercept gives an idea about the thickness of the boundary layer.

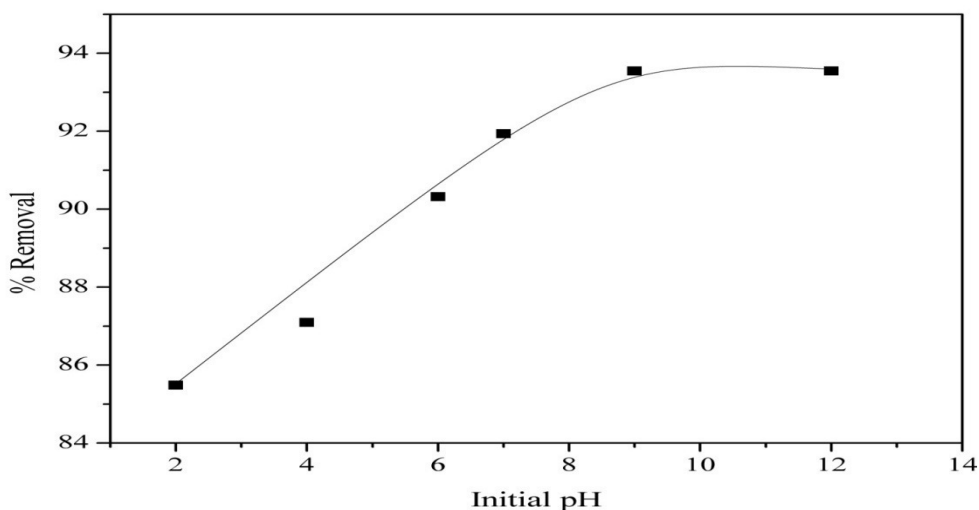


Fig.-5: Influence of pH on the removal of BBY dye onto AHC [dye concentration = 10 mg L^{-1} ; contact time = 3 hrs; sorbent dose = 200 mg; agitation speed = 120 rpm; $T = 30^\circ\text{C}$; Particle size = 250 BSS mesh].

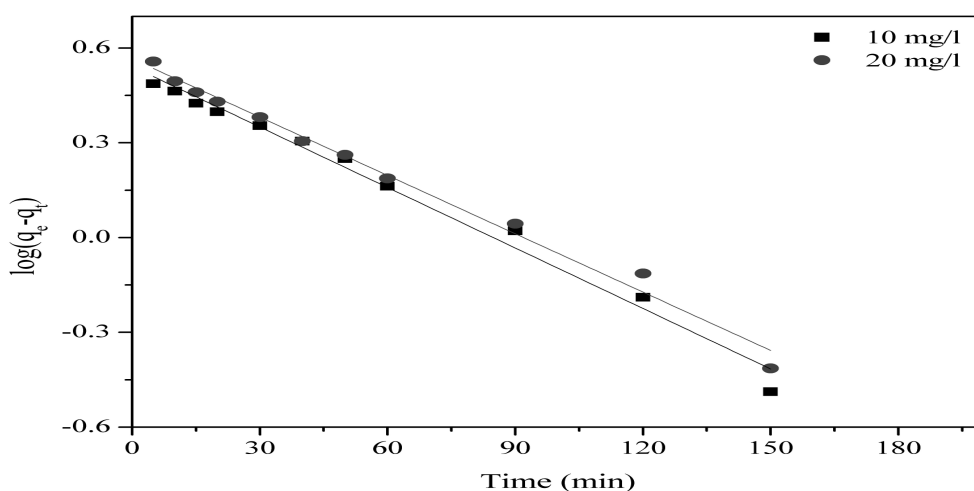


Fig.-6: Pseudo first order kinetics for the adsorption of BBY dye onto AHC.

Table-3: Adsorption Isotherm Parameters for the adsorption of BBY dye onto AHC

Langmuir		Freundlich		Tempkin	
$q_0(\text{mg g}^{-1})$	26.54	$k_f(\text{mg g}^{-1})$	5.9	A	1.3421
R_L	0.2318	N	2.56	b	6.06
$b(\text{L mg}^{-1})$	0.1625	R^2	0.9604	B	411.42
R^2	0.9905			R^2	0.995

Table-4: ANOVA for Various adsorption parameters for the adsorption of BBY onto AHC

Parameters		DF	SS	MS	F Value	Prob>F
Particle Size and Dosage	100 BSS mesh	6	24731.83	12275.11	270.36	8.01×10^{-5}
	150 BSS mesh	6	29013.84	14443.73	457.21	2.83×10^{-5}
	250 BSS mesh	6	32102.07	15962.25	359.59	4.56×10^{-5}
Time	10 mg L ⁻¹	12	46638.91	23300.25	6067.61	3.00×10^{-15}
	20 mg L ⁻¹	12	39551.59	19767.37	11744.27	1.11×10^{-16}
Initial Conc	5 mg L ⁻¹	8	57421.87	28625.00	999.33	6.66×10^{-8}
	10 mg L ⁻¹	8	39799.68	19895.31	13173.72	2.95×10^{-11}
	15 mg L ⁻¹	8	42621.11	21273.97	1744.80	1.26×10^{-8}
	20 mg L ⁻¹	8	34401.81	17196.65	12143.80	3.76×10^{-11}
pH	10 mg L ⁻¹	6	49006.244	24501.3	2670.00	8.42×10^{-9}

Table-5: ANOVA for Kinetic parameters for the adsorption of BBY onto AHC

Model	Conc (mg L ⁻¹)	DF	SS	MS	F Value	Prob>F
Pseudo-first order	10	10	0.9364	0.92451	696.0458	7.80×10^{-10}
	20	10	0.86872	0.85976	863.2913	2.99×10^{-10}
Pseudo-second order	10	11	1479.728	1445.825	426.4562	1.57×10^{-9}
	20	11	402.8572	401.1177	2306.02	3.70×10^{-13}
Intra Particle diffusion	10	11	11.67187	11.64548	4412.412	1.45×10^{-14}
	20	11	14.21101	14.10697	1355.947	5.19×10^{-12}

Table-6: ANOVA for Isotherm parameters for the adsorption of BBY onto AHC

Model	DF	SS	MS	F Value	Prob>F
Langmuir	4	0.0040	0.0040	315.73	3.89×10^{-4}
Freundlich	4	0.7726	0.7652	309.76	4.00×10^{-4}
Temkin	4	186.21	185.29	605.09	1.47×10^{-4}

Adsorption isotherm

Langmuir isotherm

Langmuir proposed a theory to describe the adsorption of molecules onto adsorbent surfaces¹⁸. Langmuir's model of adsorption predicts the existence of monolayer coverage of the adsorbate at the outer surface of the adsorbent. The isotherm equation further assumes that adsorption takes place at specific homogeneous sites within the adsorbent, which implies that all adsorption sites are identical and

energetically equivalent. The saturated or monolayer capacity can be represented by the following expression:

$$\frac{1}{q_e} = \frac{1}{q_0 b C_e} + \frac{1}{q_0} \quad (6)$$

The data for linear transformation of the Langmuir isotherm are shown in Table 2. The Linear plot of $1/C_e$ Vs $1/q_e$ shows that the adsorption follows Langmuir isotherm model as shown in Fig.9. The values of monolayer capacity q_0 and Langmuir constant b have been evaluated from the intercept and slope of these plots by using graphical techniques. It is observed from this table that the monolayer capacity q_0 of the adsorbent for the dye is compared and the maximum adsorption capacity is evolved from adsorption isotherm. As b values reflect equilibrium constant for adsorption process, it shows the affinity of the adsorbent towards the dyes. From this b values, it is possible to understand which dye shown the best attraction towards this AHC particles. The Langmuir monolayer capacities q_0 for BBY onto AHC was found to be 26.54 mg g^{-1} . The R^2 value 0.9905 suggests that the Langmuir isotherm provides a good fit to the isotherm data. The value of R_L was 0.2318 which indicates the favour of adsorption.

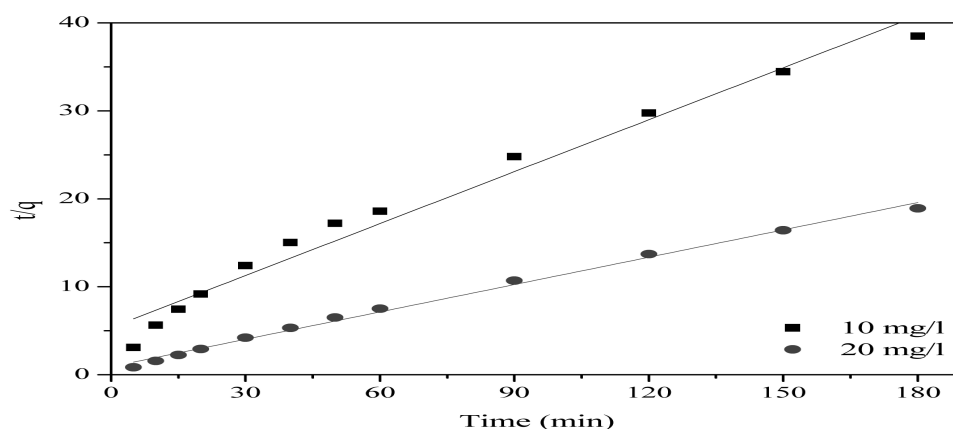


Fig.-7: Pseudo second order kinetics for the adsorption of BBY dye onto AHC.

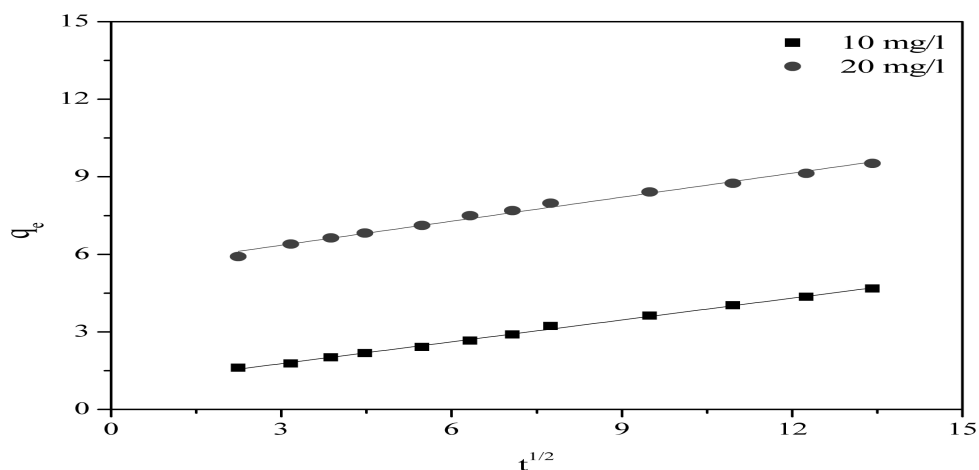


Fig.-8: Intra particle diffusion plots for the adsorption of BBY dye onto AHC.

Freundlich isotherm

The Freundlich adsorption isotherm is expressed as:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (7)$$

K_F and n are the constants incorporating all the factors affecting the adsorption process. K_F represents the quantity of dye adsorbed in mg g^{-1} adsorbent for a unit equilibrium concentration of the dye under test and $1/n$ is a measure of adsorption intensity. It is learnt that, If $1/n$ value equal to 1 then the partition between the two phases is independent of the concentration. If the $1/n$ value is below one it indicates a normal adsorption. On the other hand $1/n$ is above one indicates cooperative adsorption¹⁹. It is generally stated that the value of 'n' which is in the range of 2 to 10, represents good adsorption isotherm. The Freundlich adsorption capacity by this plot is 5.90 mg g^{-1} . From the results it was clearly observed that both models were well suited for adsorption of BBY on AHC.

Temkin isotherm

The Linear form of Temkin equation is expressed as²⁰:

$$q_e = B \ln A + B \ln C_e \quad (8)$$

where $B = RT/b$, b is the Temkin constant related to heat of absorption, A is the Temkin isotherm constant, R is the gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$) and T the absolute temperature (K). Therefore, by plotting q_e vs $\ln C_e$ enables one to determine the constants A and b as shown in Fig. 9. The constants A and B are listed in Table 2. AHC has maximum binding energy 1.34 J g^{-1} which is uniformly distributed. The value of constant B is 6.06 J mg^{-1} corresponds to the heat of adsorption. The correlation coefficient of 0.9951 obtained showed that adsorption of BBY also followed the Temkin model.

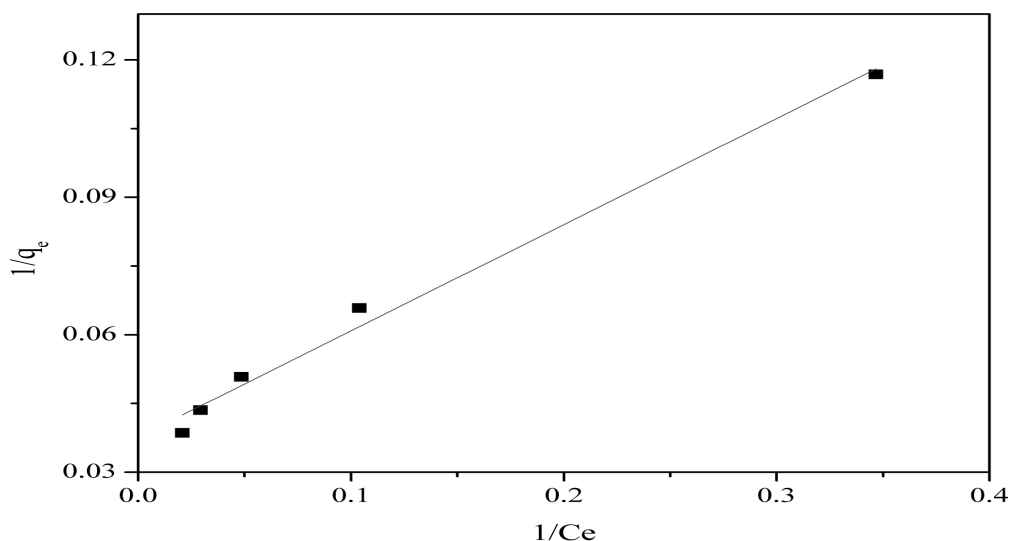


Fig.-9: Langmuir adsorption isotherm for the adsorption of BBY dye onto AHC

Analysis of variance

The results of analysis of variance (ANOVA) are given in Table 4, 5 and 6. Statistical analysis of variance was performed to check whether the process parameters are statistically significant or not. The F-value for each process indicates which parameter has a significant effect on the BBY removal²¹. Since the Probability value is bigger than 0.05. At the level, the variances are not significantly different. Suppose

the Probability value is smaller than 0.05. At the level, the variances are significantly different²². Usually, the larger the F-value has the greater the effect on the BBY removal. Optimal conditions for the process parameters can be predicted using ANOVA analysis and performance characteristics.

The results of ANOVA analysis for the removal of BBY onto AHC are listed in Table 4. Larger the F-value more is the effective parameter in the BBY removal. The sequential order of the process variables is given below for BBY removal onto AHC-

Dosage> Particle Size> C_o >pH> Time

From the table-5 and 6 kinetic and isotherm parameters shows at least two groups of the four have significant different means, since the p-value is smaller than 0.05.

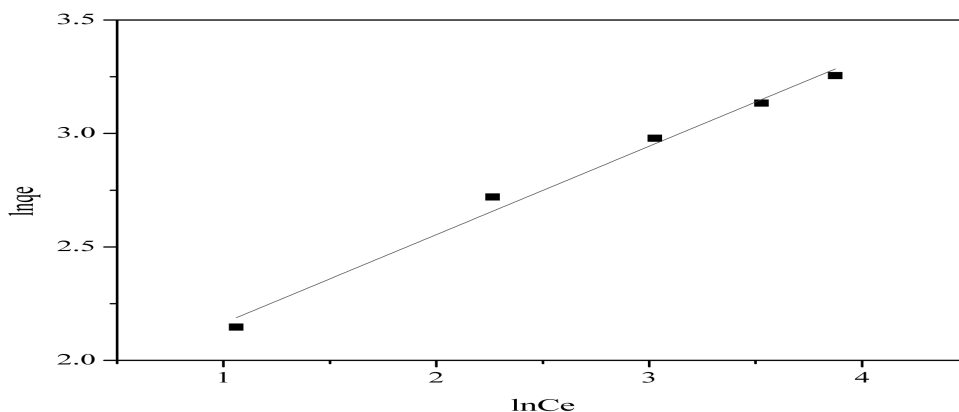


Fig.-10: Freundlich adsorption isotherm for the adsorption of BBY dye onto AHC.

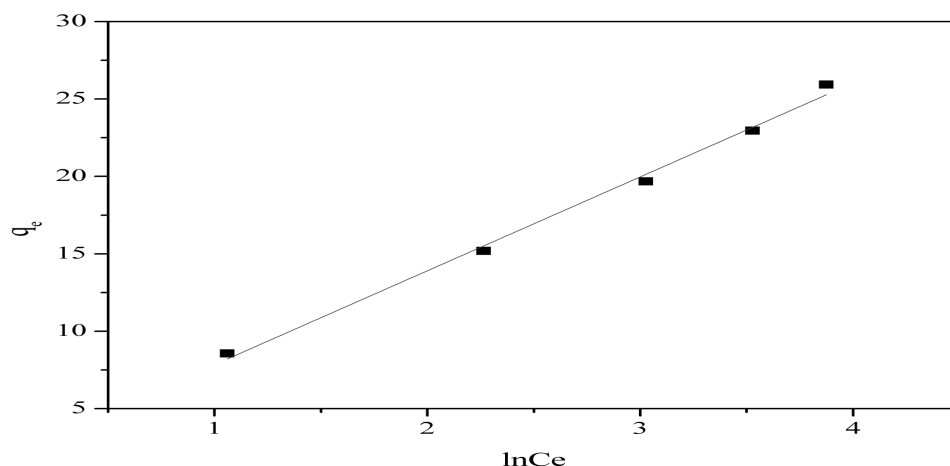


Fig.-11: Temkin adsorption isotherm for the adsorption of BBY dye onto AHC.

CONCLUSION

The agricultural waste products are predominant in the agricultural practicing country like India, where the effluent holding the toxic pollutant from textile industry is a threatening factor. Under this circumstance low cost effective adsorbents from the agricultural waste for the sequestration of dye can be considered. The adsorption capacity of the adsorbent was considerably affected by particle size, adsorbent dose, contact time, initial dye concentration and initial pH at ambient temperature. The maximum removal of BBY took place in a pH range of 7-14. The adsorption of BBY onto AHC was found to increase with increase in adsorbent dose. Based on correlation coefficient (R^2 value), the experimental data was fitted for Langmuir, Freundlich and Temkin isotherm models. The monolayer adsorption

capacity of AHC for BBY was found to be 26.54 mg g⁻¹. The suitability of pseudo-first order, pseudo-second order and intra-particle diffusion kinetic models for the sorption of BBY onto AHC was also discussed. Kinetic data follow the pseudo-first order kinetic model. Intra-particle diffusion model proves that pore diffusion plays major role in the dye adsorption. ANOVA indicated that the most considerable factor was Particle size and adsorbent dosage. The results showed the possibility of AHC for dye removal from aqueous solution as an alternative for most costly used adsorbent.

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