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ADSORPTION OF REACTIVE BLUE-4 DYE FROM AQUEOUS SOLUTION ONTO ACID ACTIVATED MUSTARD STALK: EQUILIBRIUM AND KINETIC STUDIES

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Abstract

Present study deals with the adsorption of Reactive blue 4 (RB-4) dyes onto mustard stalk activated carbon (MSAC). Batch studies were performed to evaluate the influences of various experimental parameters like adsorbent dose, pH, contact time and initial concentration for removal of RB-4. The RB-4 removal were found to be 61.5% at optimum conditions pH 7, adsorbent dose 10g/l and equilibrium time 360 min. for 150mg/l of concentration. Adsorption of RB-4 followed pseudo-second order kinetics. Equilibrium isotherms for the adsorption of RB-4 on MSAC were analyzed by Freundlich, Langmuir, Temkin, D-R isotherm models. Out of these four models, a Langmuir isotherm was found to be best with experimental data for adsorption of RB-4 onto MSAC. The results indicate that MSAC is a good adsorbent for the removal of RB-4 from wastewater.

Keywords: *Adsorption, activated carbon, mustard stalk, Reactive Blue 4.*

1. Introduction

Many industries such as textile, paper, rubber, plastics, paints, printing and leather discharge colored effluent indiscriminately, which cause pollution in receiving water bodies. The problem is more severe for textile industries because they are major consumers of the dyes, most of which are toxic and non-biodegradable. Reactive Blue 4 dye (RB-4) an anthraquinone-based chlorotriazine dye which is very important in dyeing of cellulosic fabrics [4].The release of this dye into the environment is undesirable, not only because of their color, but because of dyes and their breakdown products are toxic and/or mutagenic to aquatic life. Numerous methods exist for the treatment of textile wastewater with varying degree of success [10]. Amongst these, the adsorption technique using low-cost adsorbents derived from various natural, agricultural and industrial wastes [2, 5] are most widely employed in wastewater treatment. Some agricultural wastes that have been converted to activated carbon for dye adsorption are olive kernels [22], *Euphorbia rigida* [7], bamboo shoot [8], jute fiber [16], coconut flower [15], bamboo dust, coconut shell, groundnut shell, rice husk, straw and silk cotton hull for removal of reactive dyes [17].

In this study, activated carbon of mustard stalk has been evaluated as a low cost adsorbent for the removal of RB-4 from aqueous solution. The kinetic and equilibrium studies were carried out to understand the adsorption process.

2. Material and Methods

The reactive blue 4 (abbreviation, RB-4, CI number: 61205, molecular formula ($C_{23}H_{12}C_{l2}N_6Na_2O_8S_2$) was purchased from Sigma Aldrich (Germany). An accurately weighed quantity of the dye was dissolved in double-distilled water to prepare a stock solution (1000 mgl^{-1}). The desired concentration ranges 50–200mg/l were obtained by successive dilutions with double-distilled water. Activated carbon prepared from chemically treated mustard stalk, the procedure mention by Madhava et al. 2006, having surface area 129 m^2/g was used as adsorbent.

3. Batch Study

To study the effect of important parameters like adsorbent dose, pH, contact time, initial concentration on the removal of RB-4, batch experiments were conducted at 32^0C. For the determination of optimum adsorbent dose, 100ml dye solution was treated with adsorbent dose range 2 to 12g/l, till equilibrium was attained. The effect of pH on dye removal was studied over a pH range 2–11. pH was adjusted by the addition of dilute aqueous solutions of HCI or NaOH (0.10M). For each experimental run, 100ml of RB-4 solution of known concentration, pH and a known amount of the adsorbent dose were taken in a 250ml conical flask. This mixture was agitated in a temperature controlled orbital shaker at a constant speed of 150 rpm at 32^0C. Samples withdrawn at appropriate time interval were filtered then analyzed spectrophotometrically. In order to investigate the kinetics of adsorption of dye onto adsorbent, various kinetic models, pseudo- first order, pseudo-second order, elovich and intra-particle diffusion were used. Langmuir, Freundlich, Temkin and D-R have been used to describe the equilibrium nature of adsorption of dye in the present study.

The percentage removal of dye and equilibrium adsorption uptake, q_e(mg/g), was calculated using the following relationship:

$$\text{Dye Removal (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \qquad (1)$$

$$\text{Amount adsorbed } (q_e) = \frac{(C_0 - C_e)v}{w} \qquad (2)$$

Where v the volume of the solution (L) and w is the mass of the adsorbent(g). The wavelength corresponding to maximum absorbance determined was 595nm using a double beam UV/VIS spectrophotometer (Perkin-Elmer 135).

4. Results and Discussion

4.1Effect of adsorbent dose

The effect of on the removal of RB-4 onto MSAC at concentration 150 mgl^{-1} is shown in fig.1. It can be seen that RB-4 removal increased up to a certain limit i.e.10g/l and after that it remained almost constant. An increase in the adsorption with the increase in adsorbent dose can be attributed to greater surface area and the availability of more adsorption sites. At adsorbent dose 12g/1, the adsorbent surface becomes saturated with RB-4. Hence 10g/l was selected as optimum dose for the RB-4 removal onto MSAC.

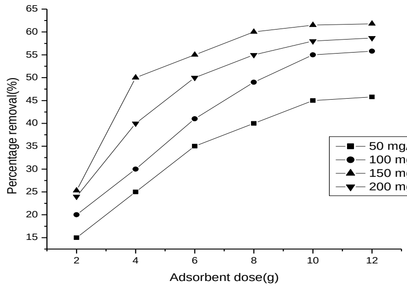

Figure 1. Effect of adsorbent dose for removal of RB-4 onto MSAC. At initial conc.150 mg/l, pH 7

4.2 Effect of pH

Figure 2 shows the effect of pH on the adsorption of dye onto mustard stalk. The maximum color removal 60% was obtained at optimal pH 7 for reactive blue 4. Either side of pH 7 color removal decreased as the pH increased to acidic or alkaline range. Solution pH is an important factor controlling the surface charge of the adsorbent and the degree of ionization of the materials in the solution. The adsorption capacity increased when the final pH was increased from 2 to 7 for the RB-4. After pH range, the adsorption capacity of the RB-4 slightly decreased, because of electrostatic interaction between MSAC and dye molecules [6].

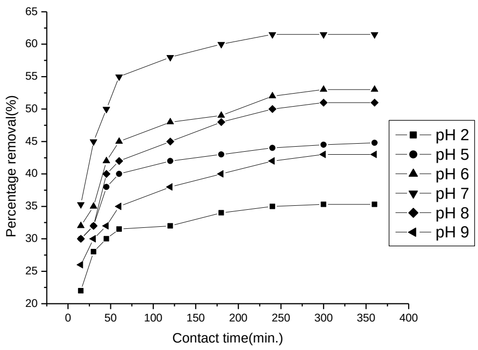

Figure 2. Effect of pH on removal of RB -4 onto MSAC. At initial conc.150 mg/l, adsorbent dose 10 g/l.

4.3 Effect of initial dye concentration

It is understood that the initial dye concentration possess significant influence on the amount of dye being adsorbed onto the sorbent [14]. Figure 3 showed the effect of initial dye concentration range (50–200 mgl^{-1}) on the adsorption capacity and removal efficiency of RB-4 onto MSAC. The adsorption capacity increased from 3.25 to 10mg g^{-1} with increasing concentration 50 to 200 mgl^{-1} of RB-4. For the RB-4, the adsorption capacity beyond dye concentration 150 mg l^{-1} did not increase, suggesting that MSAC the reached its maximum adsorption capacity. The sorption sites on the MSAC could not accommodate more dye molecule with the increase of RB-4 concentration and it is believed that the system had reached saturation point. At higher concentration, the available sites of sorption became fewer as compared to the molecules of dye present. In other words, the abundant dye molecules of RB-4 available in the solution were found to compete for the limited binding sites on the surface of MSAC at high initial concentration, suggesting that the available sites on the MSAC is one of the limiting factors for RB-4 sorption [15, 19, and 20].

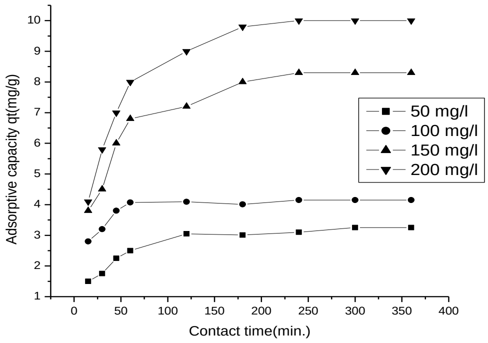

Figure 3.Effect of initial concentration for removal of RB-4 onto MSAC. At adsorbent dose 10 g/l, pH 7.

4.4 Effect of contact time

Figure 4 show the 61.5% removal of dye onto MSAC with increase in contact time from 5 to 360 min. The effect of contact time on the removal of RB-4 by the MSAC at concentration 50,100,150 200 mg /l with adsorbent dose 10g/l showed rapid adsorption of RB-4 in the first 240 min. and thereafter adsorption reached equilibrium in about 360 min. Aggregation of dye molecules with the increase in contact time makes it almost impossible for the dye molecules to diffuse deeper into the adsorbent structure at the highest energy sites. This aggregation negates the influence of contact time as the mesopores get filled up and start offering resistance to diffusion of aggregated dye molecules in the adsorbent [11,12,21].

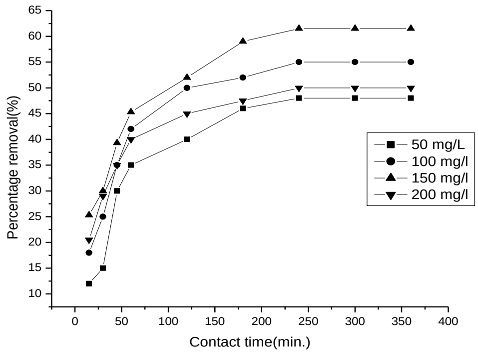

Figure 4.Effect of contact time on removal of RB-4 onto MSAC. At initial conc.150 mg/l, adsorbent dose 10 g/l.

5. Adsorption Kinetic Study

Four kinetic models, pseudo-first-order, pseudo-second-order, elovich and intra-particle diffusion models, were used to investigate the adsorption process of dye onto MSAC.

5.1 Pseudo-first order model

The pseudo-first-order model was described by Lagergren eq. (3)

$$\frac{dq_t}{dt} = K_1(q_e - q_t) \quad (3)$$

Where q_e and q_t refer to the amount of dye adsorbed (mg g^{-1}) at equilibrium and at any time, t (min), respectively and K_1 is the equilibrium rate constant of pseudo-first-order adsorption (min^{-1}). Integration of eq. (4) for the boundary conditions t=0 to t and q_t =0 gives.

$$\log(q_e - q) = \log(q_e) - \frac{K_1}{2.303}t \quad (4)$$

The values of log (q_e − q) were linearly correlated with t. The plot of log (q_e − q) vs. t should give a linear relationship from which the values of K_1 were determined from the slope of the plot. As shown in table1, lower correlation coefficient confirms that Lagergren expression cannot be applied for the entire process of adsorption of RB-4 onto MSAC.

5.2 Pseudo-second order model

The pseudo-second-order model is represented by the following differential eq. (5)

$$\frac{dq_t}{dt} = K_2(q_e - q_t)^2 \quad (5)$$

Where K_2 is the equilibrium rate constant of pseudo-second-order adsorption (g mg^{-1} min^{-1}). Integrating eq. (6) for the boundary condition t=0 to t and qt =0 to q, gives:

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \quad (6)$$

The slope and intercept of plot of t/q vs. t were used to calculate the second-order rate constant K_2 (Fig.5). The values of equilibrium rate constant (K_2) and correlation coefficient of all examined data were found very high (R^2 ≥0.998) for adsorption are presented in Table 1. This shows that the model can be applied for the entire adsorption process and confirms that the sorption of RB-4 onto MSAC follows the pseudo-second order kinetic model.

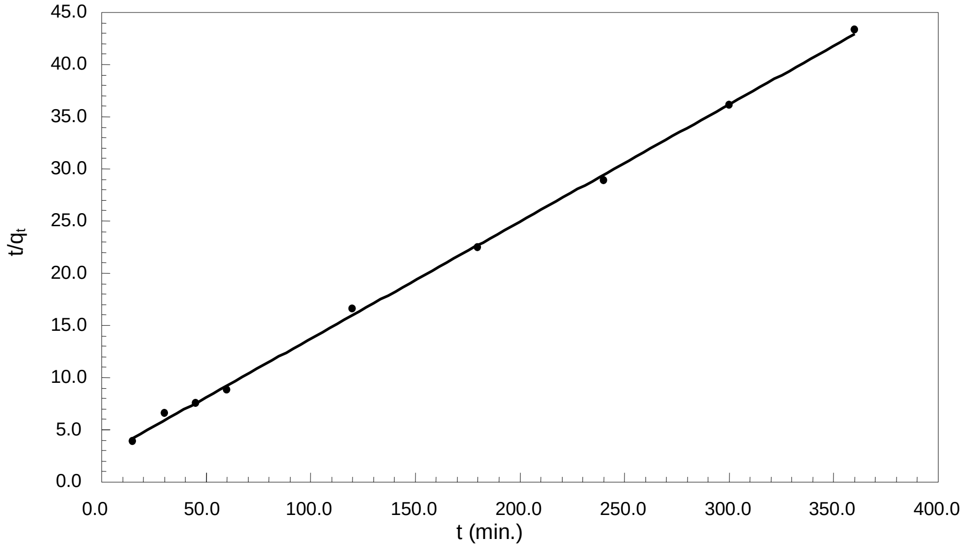

Figure 5. Pseudo-second order for RB-4 removal onto MSAC. At initial conc.150 mg/l, adsorbent dose 10 g/l, pH 7.

5.3 Elovich equation

The Elovich equation is generally expressed as eq.7

$$q_t = \beta \ln(\alpha\beta) + \beta \ln(t) \quad (7)$$

Where q_t is the sorption capacity at time t (mg g^{-1}), α is the initial sorption rate (mg g^{-1} min^{-1}) and β is the desorption constant (g mg^{-1}) during any experiment. Thus, the constants can be obtained from the slope and the intercept of a straight line plot of q_t against ln (t) as seen in fig.6 having lower correlation coefficient value confirms that, elovich eq. cannot be applied with experimental data.

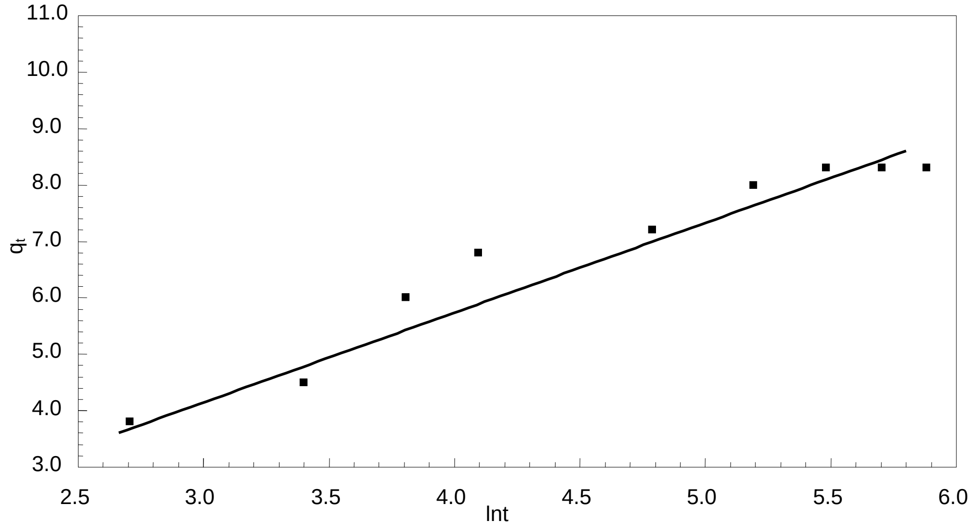

Figure 6: Elovich plot for RB -4 onto MSAC.At initial conc.150 mg/l, adsorbent dose 10 g/l, pH 7.

5.4 Intra-particle diffusion

The kinetic results were further analyzed by the intra-particle diffusion model to elucidate the diffusion mechanism by using the intra- particle diffusion model (Weber and Morris, 1963) .The amount of RB-4 adsorbed (qt) at time (t) was plotted against the square root of contact time ($t^{0.5}$) according to eq.8

$$q_t = K_{id}\, t^{\frac{1}{2}} + I \quad (8)$$

Where k_{id} is the intra-particle diffusion rate constant. In Fig. 7, a plot of qt versus $t^{1/2}$ is presented for adsorption of RB-4 onto MSAC. Values of I give an idea about the thickness of the boundary layer, i.e., the larger the intercept, the greater is the boundary layer effect [3] there are two separate zones: (1) first linear portion (phase I) representing surface adsorption and immediate utilization of the most readily available sorption sites on the surface of adsorbent (2) second linear part (phase II) illustrating the very slow diffusion of the adsorbate from the surface site into the inner pores [9].The present study indicates that the initial portion of RB-4 adsorption by MSAC may be governed by the initial intra-particle transport of RB-4, controlled by surface diffusion process and the later part controlled by pore diffusion. Figure 7 shows that the intercept of the lines fail to pass through the origin, which indicated the existence of some degree of boundary layer control, and the difference in the rate of mass transfer in the initial and final stages of adsorption. Such deviation of the straight lines from origin reveals that the pore diffusion is not the only rate-limiting step, but other kinetic models which may be operating simultaneously and thus control the overall rate of adsorption [1].

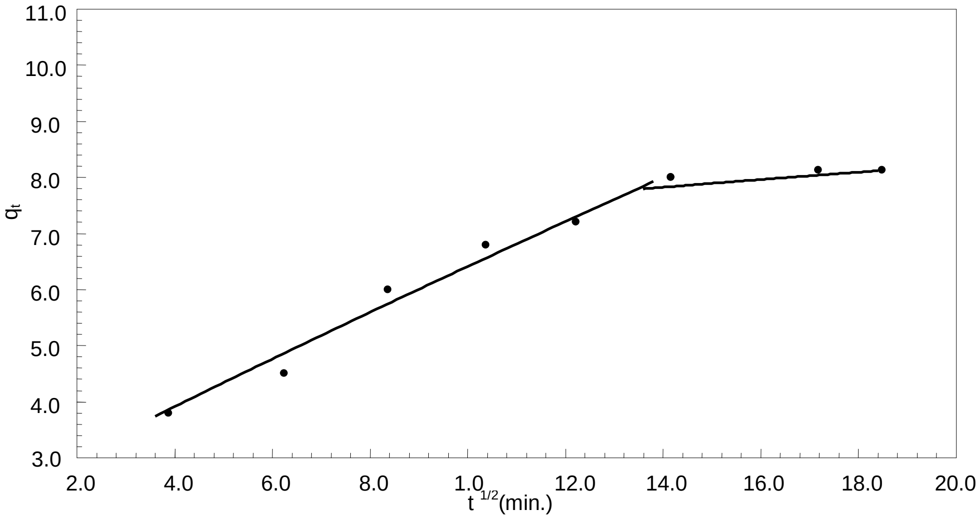

Figure7. Weber Morris plot for RB-4 removal onto MSAC. At initial conc.150 mg/l, adsorbent dose 10 g/l, pH 7.

6 Adsorption Isotherm Study

The adsorption isotherms are important to describe the sorbate-adsorbent interaction. The isotherm data were analyzed by fitting them into Langmuir, Freundlich, Dubinin-Radushkevich, and Temkin isotherm to find out the suitable model.

6.1 Langmuir Isotherm

The Langmuir equation (9) is represented in the linear form as follows:

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_m} + \frac{C_e}{Q_m} \quad (9)$$

Figure 8 shows the Langmuir (1/ C_e vs. 1/ q_e) plot of RB-4 onto MSAC, which is found to be linear over the whole concentration range. Q_m is the theoretical maximum adsorption capacity 25.8 mg g^{-1} and the correlation coefficient are extremely high, R^2 0.999 as shown in Table1. This confirms, Langmuir is a best fit model with the experimental data. The separation factor, which is a measure of adsorption favorability. The R_L values (Table1) are in between 0 and 1, thus validating a favorable adsorption process. The fitness of the Langmuir isotherm indicated the formation of monolayer coverage of the sorbate on the outer surface of the adsorbent.

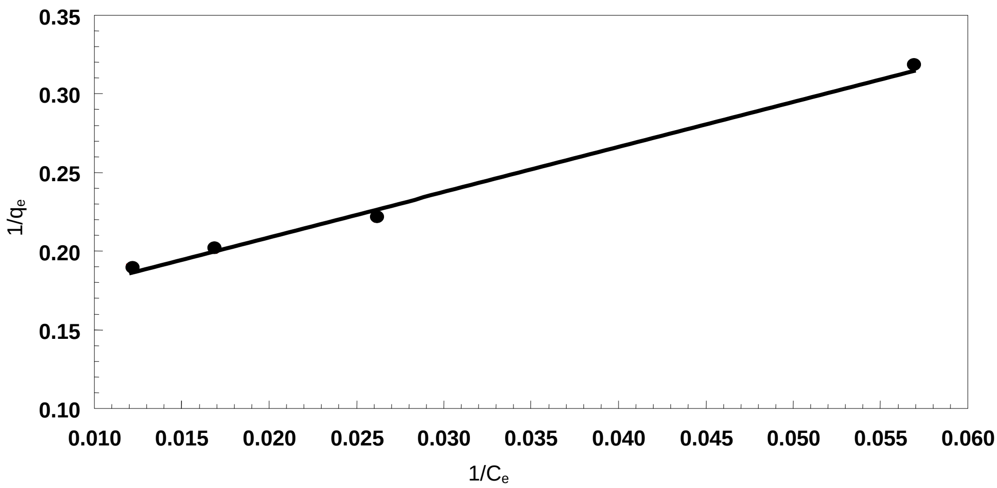

Figure 8. Langmuir Isotherm for RB- 4 removal onto MSAC. At adsorbent dose 10 g/l, pH 7

6.2 Freundlich Isotherm

The linear Freundlich isotherm is expressed as eq. (10):

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (10)$$

Figure 9 shows that linear plot of log q_e vs. log C_e of RB-4 onto MSAC also follows freundlich isotherm. The freundlich constant value, 1/n (0.19) and correlation coefficients, R^2 (0.974) were reported in Table 1, indicates less favorable than Langmuir isotherm.

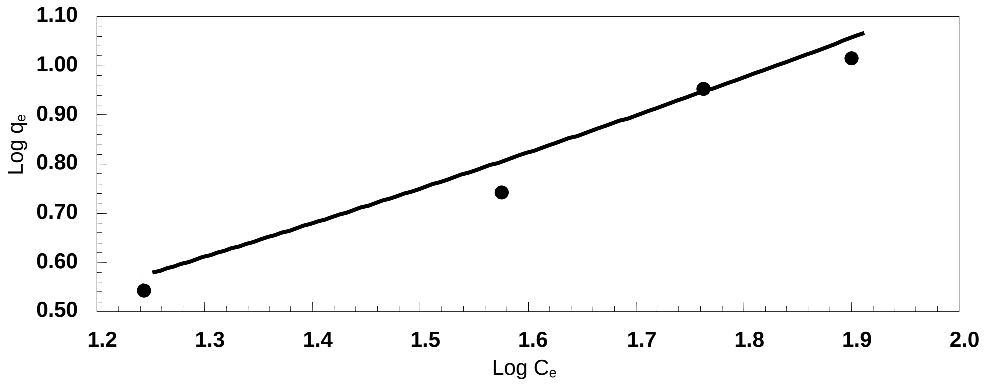

Figure 9. Freundlich Isotherm for RB -4 removal onto MSAC. At adsorbent dose 10 g/l, pH 7.

6.3 Temkin isotherm

The Temkin isotherm is given in eq. (11) as:

$q_e = B_1 \ln K_T + B_1 \ln C_e$ (11)

Where $B_1 = RT/b$, T (K) is the absolute temperature, R is the universal gas constant (8.314 J mol^{-1}), K_T is the equilibrium binding constant (l mg^{-1}), and B_1 is related to the heat of adsorption. The Temkin constants are obtained from the plot of q_e versus ln C_e as shown in fig.10.The lower correlation coefficient as shown in Table 1. Confirms that Temkin isotherm cannot be fitted with experimental data.

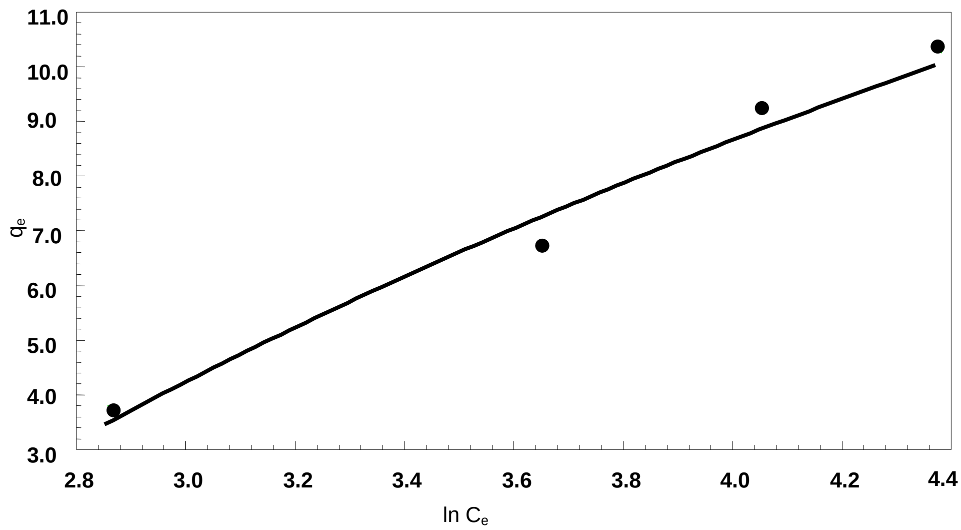

Figure 10: Temkin isotherm for RB -4 onto MSAC. At adsorbent dose 10 g/l, pH 7.

6.4 Dubinin and Radushkevich isotherm

The linear form of Dubinin and Radushkevich isotherm can be expressed as eq.(12)

$$\ln q_e = \ln Q_S - B\varepsilon^2 \quad (12)$$

ε, is the Polanyi potential and is equal to as given in eq.(13)

$$\varepsilon = RT \ln \left[1 + \frac{1}{C_e}\right] \quad (13)$$

The mean energy of sorption, E (k J mol^{-1}), is related to B as eq. (14)

$$E = \frac{1}{\sqrt{2B}} \quad (14)$$

The mean adsorption energy (E) gives information about chemical and physical nature of adsorption. The D-R isotherm plot with experimental data have been shown in figure 11. Constant were calculated from the slope and intercept of the plot between ln q_e and ε^2.Table 1 show that lower correlation coefficient, R^2 0.681cannot be fitted with the experimental data.

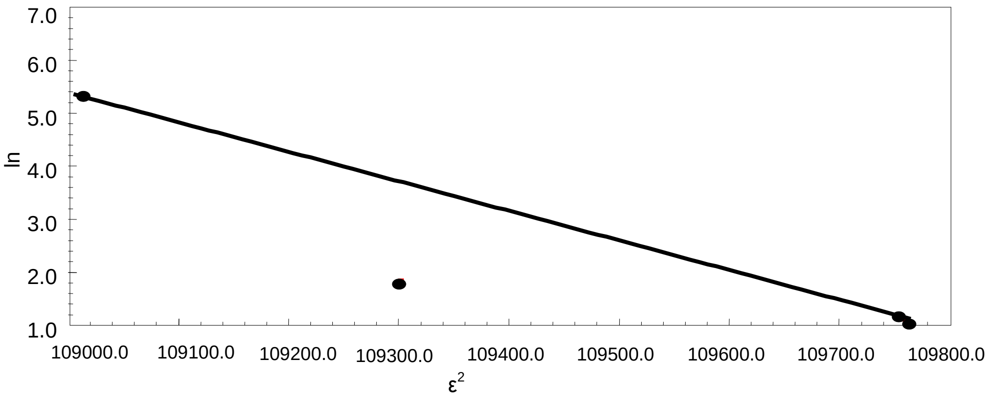

Figure 11: Dubinin–Radushkevich (D–R) isotherm for RB- 4 onto MSAC. At adsorbent dose 10 g/l, pH 7.

Table 1: Constant values of different kinetic models and adsorption isotherms for Reactive blue-4 onto MSAC

Pseudo-first order		**k_i**		**R^2**
		0.004		0.533
Pseudo-second order	**k**	**h**		**R^2**
	1.51	85.9		0.998
Elovich equation	**α**	**β**		**R^2**
	0.27	1.47		0.765
Intra-particle diffusion	**$K_{id\,1}$**	**I**		**R^2**
	1.05	75.1		0.976
	$K_{id\,2}$	**I**		**R^2**
	0.012	101.8		0.965
Langmuir Isotherm	**Q_m**	**K_L**	**R_L**	**R^2**
	25.8	0.08	0.00135	0.999
Freundlich Isotherm	**K_f**	**1/n**		**R^2**
	3.19	0.19		0.974
Dubinin Radushkevich(D–R)				
	Q_s	**$B \times 10^{-6}$**	**E**	**R^2**
	16.1	4.5	3.3	0.521
Temkin	**K_T**	**B_1**		**R^2**
	0.21	7.93		0.755

Conclusion

The present study shows that the mustard stalk activated carbon (MSAC) is an effective adsorbent for the removal of RB-4 from aqueous solution. Although the surface area of MSAC not is too much, but removal percentage (61.5) of RB-4 was possible at optimum adsorbent dose 10 g/l, pH 7, concentration 150 mg/l of solution at contact time of 360 min. The kinetics of RB-4 confirms that adsorption followed second-order rate expression and demonstrated that intra particle diffusion plays a significant role in the adsorption of RB-4. However, from the comparison of the adsorption isotherms it can be seen that adsorption experimental data for RB-4 on MSAC were best represented by the Langmuir followed by Freundlich and Temkin isotherms. The fitness of Langmuir's model indicated the formation of monolayer coverage of the sorbate on the outer surface of the adsorbent. The results indicated that the MSAC as adsorbent is capable for the removal of RB-4 with high affinity and capacity and use as a low cost adsorbent in near future for other toxic compounds removal.

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Abbreviation

Symbol	Description	Unit
1/ n	Heterogeneity factor	dimensionless
B	Dubinin–Radushkevich model constant	$(mol^2\ k\ J^{-2})$
B_1	Heat of adsorption	
C_0	Initial concentration of adsorbate in solution	$(mg\ l^{-1})$
C_e	Equilibrium liquid phase concentration	$(mg\ l^{-1})$
C_t	Concentration at time t	$(mg\ l^{-1})$
E	Mean energy of sorption	$(k\ J^{-1}\ mol)$
h	Initial sorption rate	$(mg\ g^{-1}\ min^{-1})$
I	Boundary layer	
k	Rate constant of pseudo second- order sorption	$(g\ mg^{-1}\ min^{-1})$
Kf	Freundlich constant	$((mg\ g^{-1})\ (mg\ l^{-1})\ ^{-1/n})$
ki	Rate constant of pseudo first order sorption	(min^{-1})
k_{id1}	Intra-particle diffusion rate constant. at the first step	$(\ mg\ g^{-1}\ min\ ^{1/2}\ )$
k_{id2}	Intra-particle rate constant transport at second step	$(\ mg\ g^{-1}\ min\ ^{1/2}\ )$
K_L	Langmuir adsorption constant	$(l\ mg^{-1})$
K_T	Equilibrium binding constant	$(l\ mg^{-1})$
q_e	Sorption capacities at equilibrium	$(mg\ g^{-1})$
Q_m	Theoretical maximum adsorption capacity	$(mg\ g^{-1})$
Q_s	Theoretical monolayer saturation capacity	$(mg\ g^{-1})$
q_t	Sorption capacities at time t	$(mg\ g^{-1})$
R	Universal gas constant	$(8.314\ J\ K^{-1}\ mol)$
R^2	Correlation coefficient	
R_L	Separation factor, dimensionless	
T	Temperature	(°C)
t	Time	(min.)
α	Initial sorption rate	$(mg\ g^{-1}\ min^{-1})$
β	Desorption constant	$(g\ mg^{-1})$
ε	Polanyi potential	