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Efficient use of agricultural wastes as Biosorbent in the removal of Fuchsin basic dye from aqueous solutions

S.D. Patil[1], S.T. Morbale[2] and N.T. Patel[3]

[1,2]Department of Chemistry, K.E.S.A.P. Science College, Nagothane 402 106, M.S., India
[3]Department of Chemistry, Yashwant Mahavidyalaya, Nanded 431 602, M.S., India

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ABSTRACT

Batch mode adsorption studies of Fuchsin basic (FB) on different agricultural waste materials were carried out using parameters including initial FB concentration, adsorbent dosage, agitation time, particle size of adsorbent and temperature. The best fitting isotherm model is elucidated using linear regression coefficient (R^2). Both Langmuir ($R^2 \geq 0.998$) and Freundlich ($R^2 = 0.968$ to 0.986) isotherms were found to be best fitting model for the adsorption studies. The monolayer (maximum) adsorption capacities were found to be between 142.857 to 250 mg/g of given adsorbents. Lagergren pseudo -second order model best fits the kinetics of adsorption as $q_{e(the)}$ values are in consistent with $q_{e(exp)}$ with whole range of agitation time. Adsorption process was found to be directly proportional to temperature but inversely proportional to particle size of adsorbent. Thermodynamic analysis showed that adsorption was favourable, spontaneous, increased disorder and randomness at the solid- solution interface of FB with adsorbents. Mangrove plant leaf powder was found to have greater adsorption capacity towards FB amongst the adsorbents under study.

Key words: *Fuchsin basic (FB), Agricultural wastes, Adsorption isotherms, Kinetic models*

Introduction

Dying process of textile industry belongs among important sources of contamination responsible for the continuous pollution of the environment. The production of textile industry as well as the volume of waste water containing processed textile dyes, steadily increases colour of the dye interfere with the transmission of sunlight into the stream and therefore reduce photosynthetic action. The colour in the effluent is mostly due to unfixed dye. The concentration of unused dyes in the effluent depends mainly upon the nature of dyes and dyeing processes (McMullan *et al.*, 2001). The biological, physical or chemical methods available for the removal of dyes from aqueous effluents include anaerobic/ aerobic treatment, coagulation, flocculation, oxidation, ozonation, membrane separation and adsorption. Among these methods, adsorption has been proven to be more efficient, offering many advantages over conventional processes. A number of biological adsorbents have also been investigated for the removal of reactive dyes, these include amongst others; apple pomace and wheat straw (Robinson *et al.*, 2002), corncob and barley husk (Robinson *et al.*, 2001), maize cob, wood and rice hull (Low *et al.*, 1997), wheat bran (Hamdaoui *et al.*, 2006), orange peel (Cui *et al.*, 2008), cashew nut shell (Subramaniam *et al.*, 2015), oil palm leaves (Setiabudi *et al.*, 2016), peach shell (Markovic *et al.*,

([1]Associate Prof., [2]Assistant Prof., [3]Prof.)

2016). These adsorbents were found tobe efficient in binding with basic dyes rather than acid dyes.

The present study was designed to investigate the possibility of using six different agricultural wastes as adsorbents for the removal of fuchsin basic dye from aqueous solution.

Materials and Methods

Adsorbents

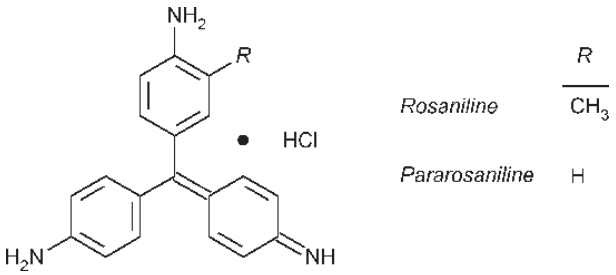
Following agricultural wastes are used as adsorbents in the present study-

- MPLP - Mangrove plant (*Sonneratia apetala*) leaf powder
- JPLP - Jackfruit plant (*Artocarpus heterophyllus*) leaf powder
- TTBP - Teak tree (*Tectona grandis*) bark powder
- ATBP - Almond tree (*Terminialia cattapa*) bark powder
- CLP - Chiku (*Manikara zopata*) leaf powder
- CPLP - Cinnamon Plant (*Cinnamonum zeylanicum*) leaf powder

Mature parts of all above agricultural wastes were collected from Raigad region of Maharashtra - India and washed many times with distilled water to remove dust and other adsorbed impurities. Washed parts were then dried for nearly 10 days in sunlight. Dried parts were cut in very small pieces and grounded in a domestic mixer- grinder. After grinding, the powders were again washed till complete removal of coloured pigments and dried again. Different sized sieves are used to get of different sized particles of each adsorbent, which are then stored in plastic bottle containers for further adsorption studies.

Adsorbate (Dye)

Fuchsin Basic (FB) is Benzenamine, 4-[(4-aminophenyl)(4-amino-2,5-cyclohexadien-1-ylidene)methyl-2-methyl] - monohydrochloride. It is a mixture of rosaniline and pararosaniline hydrochlorides. Molecular formula of rosaniline hydrochloride is $C_{20}H_{19}N_3$.HCl. It is classified as C.I. Basic Violet 14 monohydrochoride in dye classification.



A stock solution of concentration 1000 mg/l was prepared in double- distilled water and the solutions of the desired concentration were obtained from the stock solution.

Batch mode studies

The wavelength corresponding to the maximum absorbance (λmax = 550 nm) was determined using Equiptronic's Single Beam U.V.- Visible Spectrophotometer.

Batch mode adsorption studies were carried out to evaluate the efficiency of adsorbents under study. Specific amount of adsorbent was shaken in 25 ml Fuchsin Basic solution of desired concentration for pre-determined time interval at natural pH and temperature (about 303K). At the end of specified time interval, adsorbent was removed by centrifugation and supernatant was analyzed for the residual concentration of Fuchsin Basic spectrophotometrically at λmax.

Variation in agitation time, dosage of adsorbent, initial FB concentration, particle size and temperature were studied.

The values of percentage removal and amount of FB adsorbed were calculated as:

% removal of FB = $[(C_i - C_f) / C_i] \times 100$

Amount of FB adsorbed = $(C_i - C_f)/ m$

Here, C_i = Initial FB concentration, C_f = Final FB concentration, m = Mass of adsorbent.

Effect of agitation time

25 ml of FB solution of 150 mg/l concentration was shaken with 25 mg of adsorbents of ≥ 120 mesh size under study (MPLP, JPLP, TTBP, ATBP, CLP and CPLP) at nearly 303K on a oscillator at 230 rpm for 5, 10, 15, 20, 30, 40, 50 and 60 minutes. The optimum agitation time was identified, which is then used for further batch mode studies.

Effect of adsorbent dosage

Initial FB concentration of 500 mg/l were used in conjunction with adsorbent doses of 1, 2, 3, 4, 5, and 6 g/l. agitation time, agitation speed, temperature and particle size of 30 minutes, 230 rpm, 303K and ≥ 120 mesh respectively were kept constant.

Effect of Initial FB concentration

Initial FB concentration of 100, 125, 150, 175, 200, 225

and 250 mg/l were used in conjunction with adsorbent dose of 1 g/l. Agitation time, agitation speed, temperature and particle size of 30 minutes, 230 rpm, 303K and ≥ 120 mesh respectively were kept constant.

Effect of particle size

Three different particle sizes of adsorbents ≥ 120, 120 ≤ 85 and 85 ≤ 60 mesh were used in conjunction with 200 mg/l dye concentration. Agitation time, adsorbent dose, agitation speed and temperature of 60 minutes, 1 g/l, 230 rpm and 303K respectively were kept constant.

Effect of temperature

303K, 313K and 323K temperatures were used in conjunction with 250 mg/l FB concentration. Agitation time, adsorbent dose, agitation speed and particle size of 60 minutes, 1 g/l, 230 rpm and ≥ 120 mesh respectively were kept constant.

Results and Discussion

Effect of agitation time

Effect of agitation time on adsorption and % removal of FB from 150 mg/l initial FB concentration on various natural adsorbents is presented in Figures 1 and 2 respectively. Figure 1 showed that adsorption of FB increased with increase in the shaking time and attains constant value when equilibrium was established. The FB uptake process appears to be rapid in first 5 minutes and nearly 70 to 90% of total dye uptake (Figure 2) appears to have been adsorbed in this duration depending upon the adsorption ability of different adsorbents. Later on the process becomes relatively slower and attains equilibrium condition. The optimum shaking time was found to be 30 minutes, which was used for all further adsorption studies.

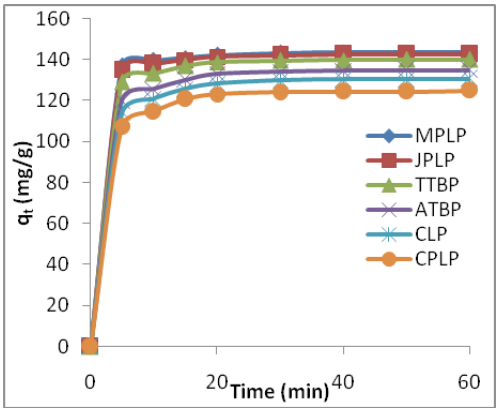


Fig. 1. Effect of agitation time on adsorption of FB.

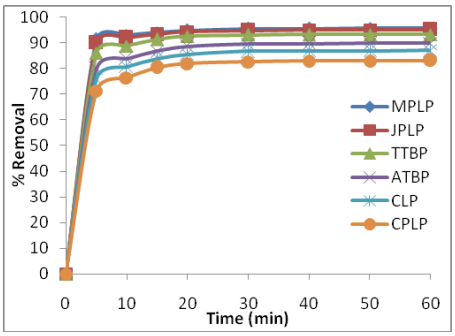


Fig. 2. Effect of agitation time on % removal of FB.

The mechanism of adsorption was investigated using pseudo - first order, pseudo- second order.

The Lagergren pseudo- first order rate equation (Singh *et al.*, 1998)is given as

$$\log (q_e - q_t) = \log q_e - (k_1 / 2.303)\, t \qquad .. (1)$$

Here, q_e and q_t are an amount of FB adsorbed in mg/g of adsorbent at equilibrium and at time t, respectively and k_1 is rate constant of pseudo first order adsorption in min^{-1}. The graph of log $(q_e - q_t)$ versus t is plotted (Figure 3). The slop of the plot used to determine pseudo first order rate constant (k_1) and y-intercept is used to determine theoretical amount of FB adsorbed per unit mass of adsorbent (Table 1). $q_{e(exp)}$ values varied from the corresponding $q_{e(the)}$ values indicates pseudo first order equation does not fit well for whole range of agitation time and is only applicable for initial stage of adsorption.

The Lagergren pseudo- second order rate equation (Ho *et al.*, 1999) is given as

$$t/q_t = 1/(k_2 q_e^2) + t/q_e \qquad .. (2)$$

Here, k_2 is rate constant of second order adsorption in g/mg/min. The slope and intercept values of plot t/q_t versus t (Figure 4) were used to determine $q_{e(the)}$ and k_2 respectively. The plot is highly linear plots showed that there may be a possibility of significant role of chemical adsorption in the rate determining step. The parameters, $q_{e(the)}$, h and k_2 obtained from pseudo second order plot are represented in Table 2.

Here,'h' is initial adsorption rate in mg/g.min and is obtained from equation, $h = k_2 q_e^2$

The regression coefficient for second order adsorption equation has values $R^2 = 1$ for both all the

adsorbents under study and $q_{e(the)}$ values are very consistent with $q_{e(exp)}$ showed that pseudo second order adsorption model fit well with whole range of agitation time and FB adsorption process is found to be controlled by chemical adsorption.

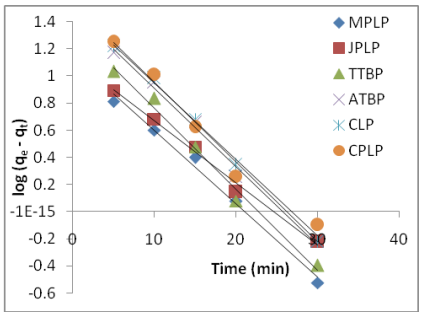


Fig. 3. Pseudo first order plot for effect of agitation time on adsorption of FB.

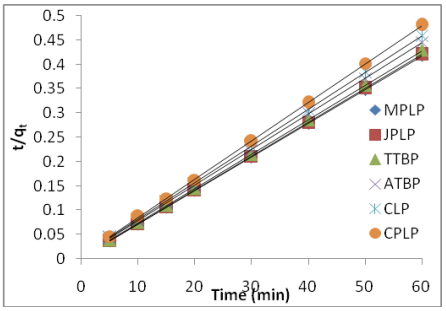


Fig. 4. Pseudo second order plot of effect of agitation time on adsorption of FB.

Effect of adsorbent dosage

The adsorption studies of FB from 500 mg/l initial FB concentration were carried out by varying the adsorbent dosage from 1 to 6 g/l. The percentage removal of FB was found to increase with increase in dosage of adsorbent (Figure 5). But amount of FB adsorbed at equilibrium per unit mass of adsorbent decreased with increase in adsorbent dosage (Figure 6). Rate of adsorption depends on number of active sites available for the adsorption.

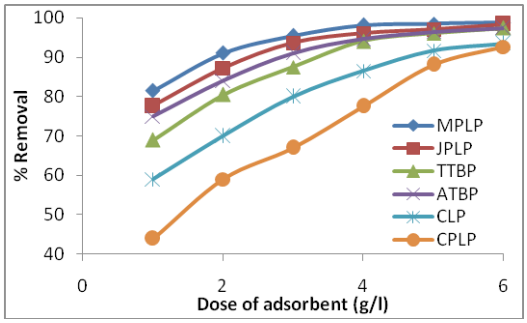


Fig. 5. Effect of adsorbent dosage on % removal of FB.

For low adsorbent dose, even though the numbers of active sites are less but most of active sites of adsorbents are available for adsorption. Therefore, percentage removal of FB may be less but amount adsorbed per unit mass of adsorbent is more.

As the adsorbent dose increases, the numbers of active sites are increases accordingly but due to

Table 1. Pseudo -first order model for effect of agitation time on adsorption of FB

Adsorbent	Initial Concentration of FB (mg/l)	$q_{e(exp)}$ (mg/g)	K_1 (min^{-1})	$q_{e(the)}$ (mg/g)	R^2
MPLP	150	143.5	0.122	13.677	0.99
JPLP	150	142.8	0.104	13.335	0.991
TTBP	150	140	0.136	22.909	0.989
ATBP	150	134.8	0.131	34.690	0.991
CLP	150	130.6	0.134	34.674	0.998
CPLP	150	124.8	0.129	32.211	0.975

Table 2. Pseudo -second order model for the effect of agitation time on adsorption of FB

Adsorbent	Initial Concentration of FB (mg/l)	$q_{e(exp)}$ (mg/g)	K_2(g/mg/min)	$q_{e(the)}$ (mg/g)	h(mg/g.min)	R^2
MPLP	150	143.5	0.018	166.666	500	1
JPLP	150	142.8	0.025	142.857	500	1
TTBP	150	140	0.016	142.857	333.333	1
ATBP	150	134.8	0.010	142.857	200	1
CLP	150	130.6	0.010	142.857	200	1
CPLP	150	124.8	0.008	142.857	166.666	0.999

crowding some of active sites of adsorbents may not be available for adsorption. Therefore, percentage removal of FB increases but amount adsorbed per unit mass of adsorbent decreases

For the given set of adsorbents, MPLP was found to have greater adsorption capacity and CPLP have least adsorption capacity.

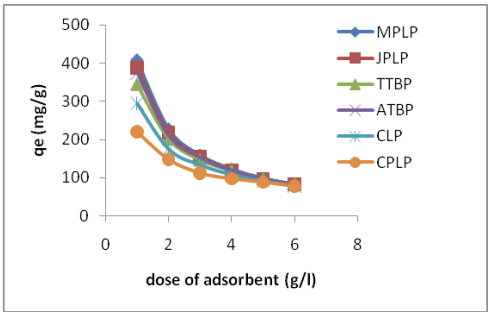


Fig. 6. Effect of adsorbent dosage on amount adsorbed (mg/g of adsorbent).

Effect of initial FB concentration

The adsorption studies of FB were carried out by varying the initial FB concentration from 100 to 250 mg/l. The percentage removal of FB was found to decrease with increase in initial FB concentration (Figure 7). But amount of FB adsorbed at equilibrium per unit mass of adsorbent increased with increase in initial FB concentration (Figure 8). Rate of adsorption depends on number of active sites available for the adsorption.

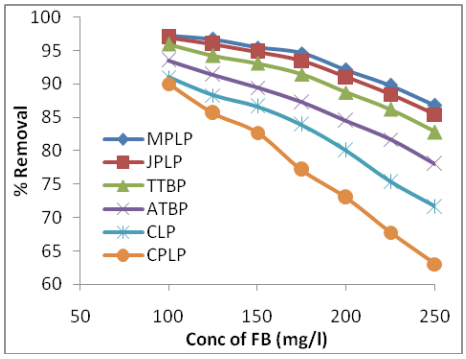


Fig. 7. Effect of initial FB concentration on % removal of FB.

It was found that the amount of FB adsorbed per unit mass of adsorbent are greater than their corresponding monolayer adsorption capacities (q_m) indicates multilayer physical adsorption for the adsorbents under study.

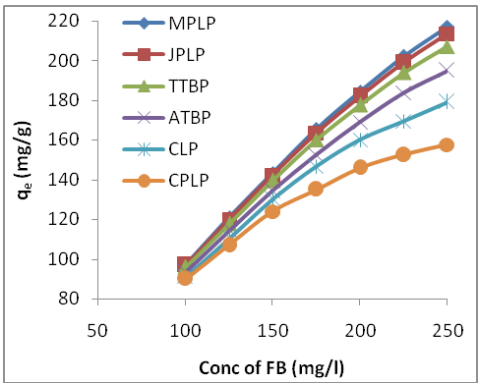


Fig. 8. Effect of initial FB concentration on adsorption of FB.

The linear form of Freundlich adsorption isotherm is represented by

$$\text{Log } q_e = \log K_f + 1/n \log C_e \quad .. (3)$$

Here 'q_e' is amount of FB adsorbed at equilibrium in mg/g of adsorbent and 'C_e' is the equilibrium concentration of FB in solution in mg/l. K_f and n are constants relates with the adsorption capacity and intensity of adsorption respectively. The plot log q_e versus log C_e showed good linearity with R^2 is between 0.977 and 0.99. Thus these adsorption studies of FB obey the Freundlich adsorption isotherm (Figure 9).

The values of constants K_f and n are given in the Table 3. The values of 'n' between 2 and 4 indicates an effective adsorption (Potgieter *et al*., 2006) while higher values of 'K_f' indicates an easy uptake of FB from the solution (Mahvi *et al*., 2004).

The Langmuir adsorption isotherm is represented by the equation,

$$C_e / q_e = 1/ (q_m b) + C_e / q_m \quad .. (4)$$

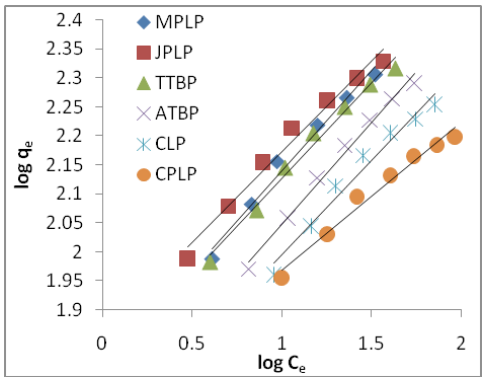


Fig. 9. Freundlich isotherm plot for effect of initial dye concentration on adsorption of FB.

Here 'q_m' is monolayer (maximum) adsorption capacity (mg/g) and 'b' is Langmuir constant related to energy of adsorption (1/mg). A linear plot of C_e / q_e versus C_e suggest the applicability of the Langmuir isotherms (Figure 10) (R^2= 0.998 to 0.999). The values of q_m and b were determined slope and intercepts of the plots and reported in Table 3. The essential features of the Langmuir isotherm can be expressed in terms of dimensionless constant separation factor (R_L) which is defined by the following relation,

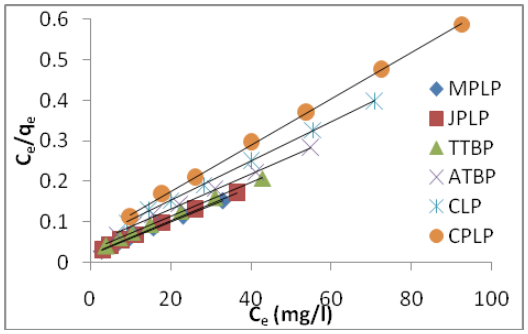


Fig. 10. Langmuir isotherm plot for effect of initial FB concentration on adsorption of FB.

$$R_L = 1/ (1+bC_o) \quad .. (5)$$

Here C_o is initial FB concentration in mg/l. R_L values lies between 0 to 1 (Table 4) indicates favourable adsorption.

Effect of particle size

Adsorption studies of FB were carried out on three different sized adsorbent particles ≥ 120, 120 ≤ 85 and 85 ≤ 60 mesh for 200 mg/l initial concentration of FB. Figure 11 shows the adsorption kinetics of the FB at three different particle sizes. As canbe seen, the removal of FB is increased as the particle size of adsorbent decreased. This is because the smaller sized particles have larger surface area and access to the particle pores is facilitated when the size of the particle is small. It is also believed that the breaking the large particle to smaller ones opens some tiny sealed channels, which might then be available for

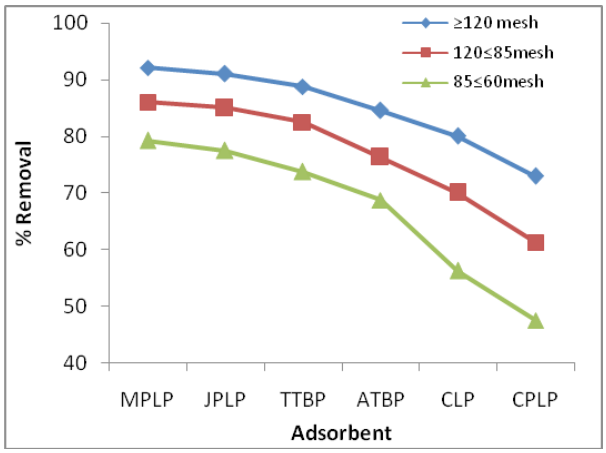


Fig. 11. Effect of particle size on % removal of FB

Table 3. Effect of initial FB concentration on adsorption of FB

Adsorbent	Freundlich isotherm parameters			Langmuir isotherm parameters		
	K_f	n	R^2	q_m	b	R^2
MPLP	62.087	2.88184	0.982	250	0.22222	0.998
JPLP	72.111	3.19489	0.981	250	0.19048	0.998
TTBP	62.517	3.03951	0.986	250	0.13793	0.998
ATBP	49.888	2.85714	0.99	250	0.08511	0.998
CLP	46.559	3.06748	0.968	250	0.06897	0.999
CPLP	51.642	3.93701	0.977	200	0.08197	0.999

Table 4. Dimensionless separation factor (R_L) calculated from Langmuir constant (b)

Initial FB Conc.	MPLP (mg/l)	JPLP	TTBP	ATBP	CLP	CPLP
100	0.043063	0.04988	0.0676	0.105141	0.12663	0.108731
125	0.034749	0.040306	0.054821	0.08592	0.103937	0.088919
150	0.029126	0.033816	0.046105	0.07264	0.088141	0.075213
175	0.02507	0.029126	0.039781	0.062916	0.076513	0.065169
200	0.022005	0.025578	0.034982	0.055488	0.067595	0.057491
225	0.019608	0.022801	0.031217	0.049628	0.060539	0.051432
250	0.017682	0.020568	0.028183	0.044888	0.054817	0.046528

adsorption, and so the adsorption of FB by the smaller particles is higher than that by larger particles. Also, the decrease in size of the particle leads to a decrease of equilibrium time. This is in agreement with the kinetic principle that smaller particles have a larger contact area by unit of mass of the adsorbent than larger ones, resulting in a faster adsorption. These observations indicate that FB adsorption occurs by a surface mechanism.

Effect of temperature

Temperature is also one of the important parameter in adsorption process. Adsorption of FB on adsorbents was studied at three different temperatures i.e. 303K, 313K and 323K from 200 mg/l initial FB concentration. Figure 12 shows the results of variation in temperatures on adsorption of FB. With increase in temperature, rate of diffusion of FB molecules across external boundary layer and internal pores of particle of adsorbent also increases. Changing the temperature also change the equilibrium capacity of the adsorbent for FB.

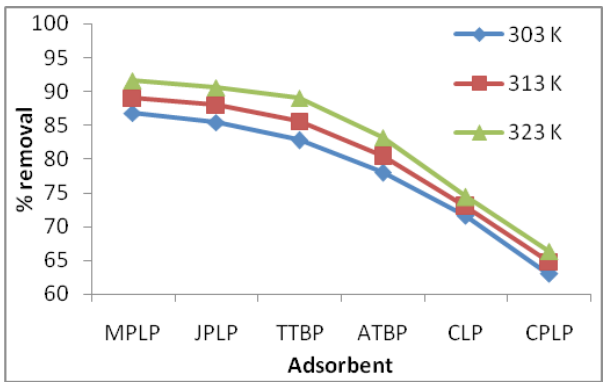


Fig. 12. Effect of temperature on adsorption of FB

The fact that the adsorption of FB is in favour of temperature indicates that the mobility of the FB dye molecule increases with a rise in the temperature, and it can be suggested that the FB molecule should interact more effectively with the surface of adsorbent. The increase in adsorption capacity with increasing temperature indicates that the process of removal of the FB by adsorbents under study is endothermicin nature and activated process.

Parameters such as free energy change (ΔG) in J/mole, enthalpy change (ΔH) in J/mole and entropychange (ΔS) (J/K/mole) were determined using following thermodynamic equations

$$\text{Equilibrium constant } (K_o) = C_{solid} / C_{liquid} \quad .. (06)$$

Here, C_{solid} is solid phase concentration in mg/l and C_{solid} is liquid phase concentration in mg/l at equilibrium

Change in free energy is given by,

$$\Delta G = -RT\ln K_o \quad .. (07)$$

$$\ln K_o = -\Delta G/RT \quad .. (08)$$

By putting the value of $\Delta G = \Delta H - T\Delta S$ in equation (08) and rearranging,

$$\ln K_o = \Delta S/R - \Delta H/RT \quad .. (09)$$

Here, T is absolute temperature in Kelvin and R is Universal gas constant. ΔG values are obtained by using equation (07). Graph of plot $\ln K_o$ versus 1/T is plotted (Figure 13). ΔH and ΔS values are obtained

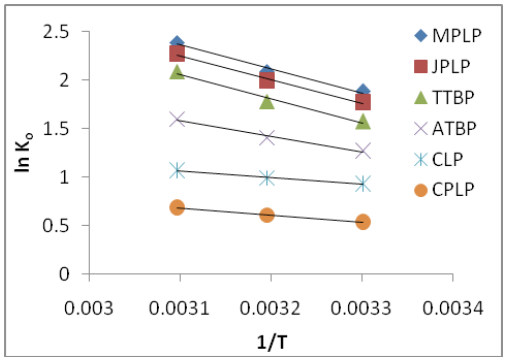


Fig. 13. Von't Hoff plot foreffect of temperature on adsorption of FB.

Table 5. Thermodynamic parameters for the adsorption of FB

Adsorbent	Ko			ΔG (J/mole)			ΔH (J/mole)	ΔS (J/K/mole)
	303K	313K	323K	303K	313K	323K		
MPLP	6.57576	8.09091	10.9048	-4744.5	-5440.7	-6416	20527.3	83.2231
JPLP	5.84932	7.33333	9.6383	-4449.6	-5184.9	-6084.5	20286.2	81.5603
TTBP	4.81395	5.94444	8.09091	-3958.9	-4638.5	-5614.5	21067.7	82.4666
ATBP	3.54545	4.10204	4.95238	-3188.4	-3673.1	-4296.3	13568.4	55.2465
CLP	2.52113	2.7037	2.90625	-2329.5	-2588.3	-2865	5779.89	26.7545
CPLP	1.7027	1.84091	1.97619	-1340.7	-1588.1	-1829.2	6060.91	24.4265

from the slope and intercept of the plot and presented in Table 5. As the values of ΔG are negative indicatingthe adsorption of FB on the adsorbents is spontaneous and favourable. It is also found that ΔG values increases with increase in temperature. The low positive values of ΔH indicating adsorption isendothermic and also there is role of physical adsorption. The positive values of ΔS show the increased disorder and randomness at the solid solution interface of FB dye with the adsorbent. The adsorbed water molecules displaced by FB molecules gain more translational energy than is lost by the FB molecules, thus allowing prevalence of randomness in the system. Adsorption capacities of the adsorbents at higher temperatures were found to increase due to enlargement of pore size and activation of adsorbent surface.

Conclusion

Agricultural wastes under study were found to have excellent adsorption capacities towards removal of Fuchsin Basic from aqueous solutions. Therefore, these wastes can be attractive alternative as low cost adsorbents for costly activated carbon.

Linearity of Langmuir and Freundlich isotherms and the parameters of these isotherms showed that the adsorption of FB on these adsorbents was favourable. The monolayer (maximum) adsorption capacities (q_m) were also found high.

Lagergren pseudo -second order model was best fitting kinetic model indicates adsorption depends on nature of Fuchsin Basic as well as adsorbents under study.

Thermodynamic analysis showed that adsorption of FB onthese agricultural wastes was favourable, spontaneous, endothermic physical adsorption, increased disorder and randomness at the solid- solution interface.

Adsorption capacities of the adsorbents under study were found to be in the following order,

MPLP > JPLP > TTBP > ATBP > CLP > CPLP

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