

Study of Removal of Cationic Dyes using Palm Shell Powder as Adsorbent

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Abstract

Batch sorption experiments were carried out for the removal of Methylene Blue and Rhodamine 6G from aqueous solution using Palm Shell Powder (PSP) as adsorbent. The operating variables studied were adsorbent mass, solution pH, contact time and initial dye concentration. Adsorption experiments indicated that the adsorption capacity was dependent on operating variables and the process was pH-independent above pH 4.0. Kinetic measurements showed that the process was uniform and rapid. Sorption of dye reached equilibrium in 120 min. In order to investigate the mechanism of sorption, adsorption data were modeled using the pseudo-first-order and pseudo-second-order kinetic equations. It was found that the adsorption kinetics followed a pseudo-second-order model for the dye concentration range studied in the present work, suggesting that the rate-limiting step may be chemisorption. Equilibrium isotherm was analyzed using the Langmuir and the Freundlich isotherms. The characteristic parameters for each model have been determined. The results of Banghams equation states that the adsorption of the dye is pore diffusion controlled process. The exhaustive capacity was 121.5 and 105.0mg/g for MB and Rh6G mg/g at 25 °C with PSP .The negative value of free energy change indicated the spontaneous nature of adsorption.

Keywords: Rhodamine6G; Methylene Blue; Palm Shell Powder; Adsorbent; Kinetic modeling; Isotherms.

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1. Introduction

Synthetic dyes are used extensively in many industries including dye houses, paper printers and textile dyers. A significant proportion of synthetic dyes are lost annually to waste streams during textile processing, which eventually enters the environment. Textile dyes have synthetic origin and complex aromatic molecular structures that make them difficult to biodegrade when discharged in the ecosystem. Some of the dyes are carcinogenic and mutagenic. Colour/dye being one of the important recalcitrant, persist for long distances in flowing water, retards photosynthetic activity, inhibit the growth of aquatic biota by blocking out sunlight and utilizing dissolved oxygen and also damage the aesthetic nature of the environment [1]. As such dyeing wastes need to be treated before discharge into the environment [2]. Various treatment methods for removal of dyes from industrial effluents like coagulation using alum, lime, ferric chloride, ferric sulphate, chemical

oxidation methods using chlorine and ozone and membrane separation methods are in vogue [3].

Adsorption hold promise in the treatment of wastewater, as it is inexpensive, simply designed, easy to handle, and provides sludge-free cleaning operations. Activated carbon has long been used in industry as a standard adsorbent for removing color. However, although it is the most widely used adsorbent for the removal of color and the treatment of textile effluents, it is not used on a large scale on account of its high price. Therefore, the development of low-cost alternative adsorbents has been the focus of recent research [4-8]. Considerable attention has been focused in recent years upon the field of biosorption for the removal of heavy metal ions from aqueous solutions. The process of heavy metal removal by biological materials known as biosorption and the biological materials used are called biosorbents. Various biosorbents like bacteria, fungi, yeasts, agricultural by products, industrial wastes, etc have been used for biosorption [9]. The efforts made in the removal of dyes from wastewater and other aqueous solutions include the

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use of clays, metal hydroxides, baggase pith, sunflower stalks, hardwood, fertilizer, fly ash, coal, peat, sawdust, lignite, wood and steel wastes [10-11]. The objective of the study was to investigate the potential of palm shell powder (PSP)(*Borassus flabellifer*) for the removal of dyes from aqueous solutions. Palm shell is an agro waste available in plenty in the coastal areas of a tropical country like India. Column adsorption equilibrium, adsorption kinetics, effect of dose and effect of pH were studied and the efficacy of the adsorbent was compared with commercially activated carbon in granular form (CAC).

2. Materials and Methods

Palm shells, the agricultural wastes of palm fruits, which are available abundantly in coastal Andhra Pradesh, were used in the present study. The scientific name of this palm is *Borassus flabellifer*. Collected shells were washed to remove mud and sun dried for 24 hrs and then crushed by using jaw crusher. Powder of 710 microns size was separated and again washed thoroughly with distilled water and kept in water for 48 hours by changing water for every 10 hrs and then removed and washed finally and dried for 4 hrs at 110°C. The physical properties of PSP are presented in Table 1. The adsorbent made in this study are characterized by scanning electron microscopy (Fig. 1).

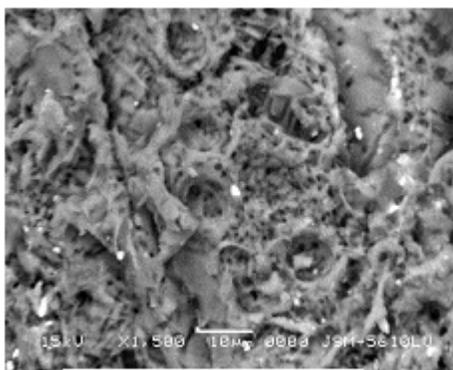


Fig 1: SEM PSP

Fig. 1 SEM analysis.

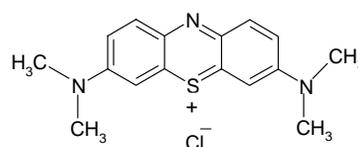
Table 1. Physical properties of PSP	
Bulk density g/cc	0.27
pH	6.4
Ion-exchange capacity meq/g	0.05
Matter soluble in water (%)	0.84
Carbon (%)	45.14
Hydrogen (%)	5.59
Nitrogen (%)	0.73

The figure shows that the adsorbent has an irregular rough and porous surface, with identifiable micropores (BET surface area 2.5174 m²/g).

This powder is named as PSP and was observed for physical properties to study its adsorption capacity. By using this powder as adsorbent, removal of cationic dyes was studied and the results were compared with the results of commercially available carbon in granular form.

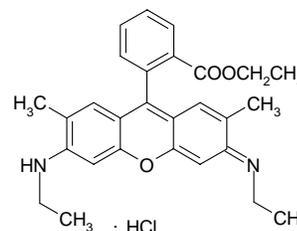
3. Experimental Procedure

All the chemicals used were of highly pure and commercially available AnalaR grade of Sulab Chemicals, Vadodara (India). Stock solutions of dyes (1g/L) were prepared by dissolving accurately weighed amount of individual dye in double distilled water and subsequently diluted to the required concentration. Adsorption studies were carried out with 100ml Durasil Stoppered flasks in a thermo regulated water bath at 25°C. A 25 mL of dye solution of known initial concentration was kept in contact with a required dose of PSP at constant stirring. After a specific stirring time period the reaction mixture was filtered. The dye concentration in the filtrate was determined by measuring absorbance at the wavelength of maximum absorption (609 nm for MB and 524 nm for Rh6G) using a SYSTRONICS Digital 166 model visible spectrophotometer. The pH of solutions was adjusted using HCl or NaOH solution. The percentage removal of the dye and the amount adsorbed (mg/g) were calculated by the following relationship: $100(C_i - C_f) / C_i$ where C_i is the initial concentration of the dye (mg/L) and C_f is the concentration of the dye present in solution after adsorption for a particular time interval (mg/L).



Methylene Blue

Basic Blue 9 M.W = 319.85 CI No. 52015



Rhodamine 6G

Basic Red 1 M.W = 479 CI No. 45160

4. Results and Discussion

4.1. Effect of initial pH

The effect of initial pH of the dye solution on the removal of MB and Rh-6G with PSP were determined at fixed concentration 80 mg/L of dyes over a pH range of 1.0 to 10.0 as shown in Fig. 2.

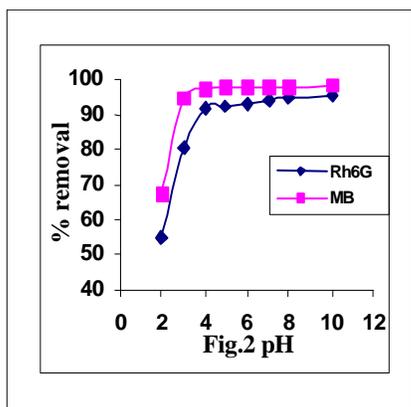


Fig. 2. Effect of pH variation

The maximum adsorption of the dyes was observed above pH 4.0. There was no significant variation in adsorption capacity with change in pH above 4.0. Hence the rest of the parameters were optimized at the original pH of the respective dye solutions itself which is 6.54 for methylene blue and 5.75 for Rhodamine6G. Lower adsorption of dyes at low pH is probably due to the presence of H^+ ions competing with the cationic groups on the dye for adsorption sites. As surface charge density decrease with an increase in the solution pH, the electrostatic repulsion between the positively charged methylene blue and Rhodamine 6G and the surface of PSP is lowered; this may result in an increase in the rate of adsorption.

4.2. Effect of adsorbent dose and time

The effect of varying the PSP mass on aqueous dye solution is shown in Table 2. The data show that as the adsorbent mass is increased the rate of adsorption increases. The percentage removal with 4 g/L of adsorbent

was compared with that of CAC. The removal percent was 93.38% for MB, 75.00 % for Rh6G using PSP and 38.43% for MB, 13.28 % for Rh6G using CAC in 30 minutes at an adsorbate concentration of 100 ppm. Initially the rate of increase in the percent dye removal has been found to be rapid which slowed down as the dose increased. This phenomenon can be explained, based on the fact that at lower adsorbent dose the adsorbate (dye) is more easily accessible and because of this, removal per unit weight of adsorbent is higher. With rise in adsorbent dose, there is less commensurate increase in adsorption, resulting from many sites remaining unsaturated during the adsorption[12]. The results obtained indicate that PSP has a large potential as an adsorbent for dye removal than CAC.

4.3. Effect of initial adsorbate concentration

The effect of initial concentration of MB or Rh6G on the amount of dye adsorbed on PSP were studied. The results obtained are shown in Fig. 3.

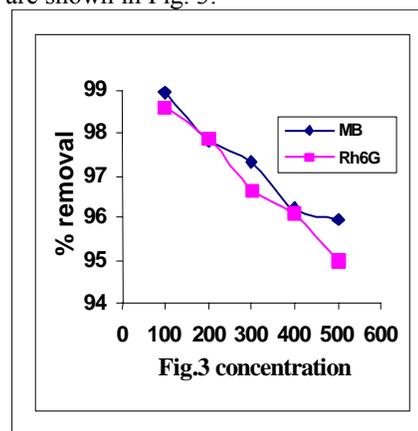


Fig. 3. Effect of initial concentration of Methylene Blue and Rhodamine 6G

From Fig. 3, it is seen that the percent adsorption decreases with the increase in initial dye concentration but the actual amount of dye adsorbed per unit mass of adsorbent increased with increase in dye concentration.

Table 2. Effect of adsorbent dose and time on 100ppm of MB & Rh6G by raw PSP

Time (min)	% removal (0.05g)		% removal (0.1g)		% removal (0.2g)		% removal (0.3g)		% removal (0.4g)	
	M.B	Rh 6G	M.B	Rh 6G	M.B	Rh 6G	M.B	Rh 6G	M.B	Rh 6G
10	73.88	59.84	85.35	72.11	96.90	82.50	98.85	94.24	99.02	98.49
20	80.69	61.41	89.92	74.30	98.00	89.25	98.96	97.16	99.10	98.70
30	92.16	62.26	93.38	75.00	98.60	91.00	99.01	97.64	99.34	98.73
60	92.63	73.52	95.52	85.55	98.90	95.25	99.05	98.27	99.38	98.82
120	96.55	80.47	96.83	90.94	99.19	95.75	99.36	98.88	99.48	99.08
150	96.50	80.50	96.80	90.50	99.20	95.60	99.35	98.90	99.50	99.00
180	96.60	80.50	96.85	90.10	99.20	95.70	99.38	98.92	99.50	99.10

Thus the adsorption is highly dependent on the initial concentration of dyes. The capacity of the adsorbent material gets exhausted sharply with increase in initial dye concentration. This may be probably as a result of the fact that for a fixed adsorbent dose, the total available adsorption sites remain invariable for all the concentrations checked. With increasing concentration the available adsorption sites become fewer and hence the percent removal of dye is dependent upon the initial concentration.

4.4. Effect of temperature

Tables 3 and 4 show the effect of temperature on the adsorption kinetics of PSP at a pH of 6.54 for MB, 5.75 for Rh6G and 100 mg L⁻¹ initial dye concentration. Results show that temperature plays an important role on the dye adsorption capacity of PSP.

It is seen that with increasing temperature from 40 to 70°C the adsorption capacity of PSP decreased. This indicates that adsorption of dyes on to PSP is controlled by exothermic process, and the maximum adsorption is nearly 98.25% and 95.51% at 120 min at 70°C for MB and Rh-6G respectively and the removal of dye is 24.5616 mg/g and 24.8663 mg/g for MB and Rh-6G respectively. In the case of CAC % removal is increased as the temperature increases with increase in time. It shows an endothermic physical adsorption and the maximum adsorption is nearly 75% and 60% at 120 min at 70°C for MB and Rh-6G respectively and the removal of dye is 18.76 mg/g and 15.09 mg/g for MB and Rh-6G respectively. Results show that temperature plays an important role on the dye adsorption capacity of PSP. At high temperature, the

thickness of the boundary layer decreases, due to the increased tendency of the dye to escape from the biomass surface to the solution phase, which results in a decrease in adsorption as temperature increases [13]. The decrease in adsorption with increasing temperature, suggest weak adsorption interaction between biomass surface and the dye, which supports physisorption whereas in the case of CAC it is chemisorption.

4.5. Thermodynamic studies

From Table 5, it is evident that the higher positive values of apparent enthalpy change show an exothermic physical adsorption favoured by increased temperature. The negative values of ΔG confirm that the basic dye adsorption on PSP is a spontaneous process. It has been reported that ΔG° up to -20 kJ/mol are due to electrostatic interaction between sorption sites and the metal ion (physical adsorption), while ΔG° values more negative than -40 kJ/mol involve charge sharing or charge transfer from the biomass surface to the metal ion to form a coordinate bond (chemical adsorption) [14]. The ΔG° values obtained in this study for the two dyes are <-10 KJ/mol, which indicates that physical adsorption was the predominant mechanism in the sorption process.

The apparent entropy change values are almost constant over the temperature range. The positive entropy characterize an increased disorder of the system due to the loss of water which surrounding the dye molecules at the sorption on the PSP. It can be suggested that the driving force for adsorption process is an entropy effect [15].

Table 3. Effect of Temperature on MB & Rh6G removal by raw PSP

Time(min)	% removal (40°C)		% removal (50°C)		% removal (60°C)		% removal (70°C)	
	M.B	Rh 6G						
10	98.40	94.06	97.64	92.50	96.08	88.09	94.64	83.98
20	98.51	94.38	97.85	93.67	97.54	89.77	96.68	88.59
30	98.60	96.36	98.49	95.08	98.40	91.99	96.76	88.87
60	98.84	97.59	98.71	96.02	98.60	94.14	97.76	92.11
120	98.99	99.03	98.82	97.62	98.75	97.42	98.25	95.51

Table 4. Effect of Temperature on MB & Rh6G removal by CAC

Time(min)	% removal (30°C)		% removal (40°C)		% removal (50°C)		% removal (60°C)		% removal (70°C)	
	M.B	Rh 6G	MB	Rh6G						
10	27.71		28.64		30.97	2.80	32.46	6.15	47.29	9.50
20	30.04	0.83	30.97	1.81	39.13	4.97	45.20	8.71	49.58	16.99
30	31.44	2.21	32.37	3.39	40.30	11.67	48.46	13.84	53.13	27.05
60	34.24	7.93	38.43	11.67	41.70	13.84	54.52	24.68	62.92	55.64
120	47.76	12.26	49.86	14.04	51.12	31.39	52.43	51.10	75.05	60.37

4.6. Column studies

Column studies were conducted using down flow technique, 1.0 gm of PSP was transferred into glass column of 1cm diameter. The column height is about 3 cm. Glass wool was kept at the bottom of the column to avoid the loss of adsorbent with the liquid flow. The dye solution was fed into the column at a flow rate of 1mL/min and definite volumes of the effluent were collected. The initial amount of the dye in the fraction passed and the amount found in the effluent fraction gave the amount of dye retained by the adsorbent.

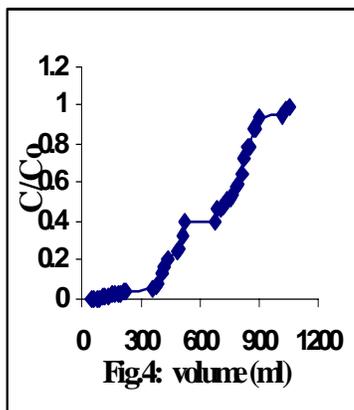


Fig. 4. Column studies for Rhodamine 6G

The break through capacity of MB and Rh6G when a column is used was studied by passing 100 ppm solution of both the dyes. The ratio C_e/C_0 where C_e is the initial concentration of dye in the effluent and C_0 is the dye in

the feed was calculated. A graph was plotted of C_e/C_0 versus volume of solution passes as shown in Figs. 4 and 5. From Figs. 4 and 5, it is evident that the volumes of aqueous solution containing 100 ppm of dye that can be treated are 1.215 L and 1.005 L for MB & Rh6G respectively. The exhaustive capacities determined are 121.5 and 105.0 mg/g for MB and Rh6G respectively.

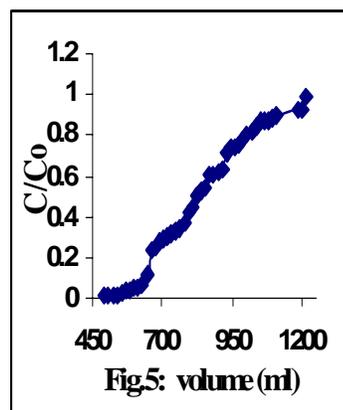


Fig. 5. Column studies for Methylene Blue

The adsorbent was used for the removal of Rh6G from an effluent coming from Rh6G manufacturing industry. 300 mL of effluent containing 150 ppm of Rh6G could be treated. The exhaustive capacity determined was 45 mg/g. In the packed column, the available active sites of adsorbent are minimum. So, the amount of dye adsorbed was found to be less when compared with batch adsorption experiments.

Table 5. Thermodynamic Parameters for Rhodamine 6G and Methylene Blue at different time and temperature intervals

Adsorbate	Temp. (K)	Rhodamine6G				Methylene Blue			
		ln K	-ΔG° KJ/mole	ΔS° KJ/mole	ΔH° KJ/mole	ln K	-ΔG° KJ/mole	ΔS° KJ/mole	ΔH° KJ/mole
Time 120 (min)	313	3.2314	8.4090			3.2013	8.6607		
	323	2.3168	6.2217	0.2573	76.8728	3.0455	8.1784	0.0659	13.0996
	333	2.2360	6.1906	0.1481	43.1270	2.9832	8.2590	0.0532	9.4526
	343	1.6607	4.7358	0.1501	46.7323	2.6394	7.5269	0.0707	16.7175
60	313	2.3035	5.9943			3.0617	7.9673		
	323	1.7860	4.7962	0.1493	43.4256	2.9534	7.9310	0.0527	9.1025
	333	1.3804	3.8218	0.1316	39.9931	2.8688	7.9426	0.0489	8.3540
	343	1.0608	3.0250	0.1166	36.9737	2.0024	5.7104	0.1085	31.5144
30	313	1.8799	4.8919			2.8688	7.4655		
	323	1.5648	4.2022	0.0971	26.4814	2.7907	7.4943	0.0435	6.5644
	333	1.0447	2.8924	0.1173	36.1838	2.7299	7.5579	0.0408	6.0201
	343	0.6804	1.9401	0.1097	35.6889	2.0024	5.7104	0.0918	25.7778
20	313	1.4238	3.7051			2.8034	7.2951		
	323	1.2984	3.4869	0.0434	10.5351	2.4338	6.5358	0.1164	31.0626
	333	0.7747	2.1447	0.0856	26.3715	2.2927	6.3474	0.0855	22.1264
	343	0.6530	1.8621	0.0730	23.1938	1.9849	5.6603	0.0875	24.3524
10	313	1.3664	3.5557			2.7358	7.1194		
	323	1.1158	2.9965	0.0745	21.0586	2.3365	6.2739	0.1234	33.5820
	333	0.6036	1.6711	0.1044	33.0489	1.8134	5.0204	0.1351	39.9691
	343	0.2595	0.7401	0.0982	32.9308	1.4841	4.2321	0.1209	37.2432

4.7. Adsorption isotherms

The experimental equilibrium adsorption data were analyzed using Freundlich and Langmuir adsorption isotherm models [16-17]:

The linearized Freundlich isotherm is shown in equation (1):

$$\text{Log}x/m = \log K_f + 1/n \log C_e \quad (1)$$

The linear form of Langmuir equation is represented in equation (2):

$$1/(x/m) = 1/Q_o + 1/bC_e \quad (2)$$

where X/M = amount of solute adsorbed per unit weight of adsorbent(mg/g), C_e =concentration of solute remaining in solution at equilibrium(mg/L), Q_o = amount of solute adsorbed per unit weight of adsorbent in forming a complete monolayer on the surface(mg/L), b = a constant related to the energy or net enthalpy, K_f and n = Freundlich constants

The data obtained from the isotherm studies were tested for applicability to the above two isotherm models. Table 6 shows the values of the parameters of the two isotherms and the related correlation coefficients. From Table 6, it can be observed that Rh6G conforms to the Freundlich isotherm model for sorption onto PSP. The values of $1/n$, less than unity is an indication that significant adsorption takes place at low concentration but the increase in the amount adsorbed with concentration becomes less significant at higher concentration and vice versa [18]. Negative values for the Langmuir isotherm constant indicates the inadequacy of this model to explain the adsorption process, since the constant represents the surface binding energy. For MB however, sorption onto PSP conforms to both the Langmuir and Freundlich models.

4.8. Adsorption dynamics

The Kinetics of basic dyes sorption onto palm shell powder was investigated using two different models: the pseudo-first order and pseudo-second order kinetics.

The pseudo-first order Lagergren model, traditionally used for describing sorption kinetics, is generally expressed by the equation:

$$\text{Log} (q_e - q_t) = \log q_e - K_1 \cdot t$$

where K_1 (mg/g.min) is the Lagergren rate constant of the first order sorption, evaluated from the slope of the plot $\log (q_e - q_t)$ versus t .

In order to obtain the rate constants, the straight-line plots of $\log (q_e - q_t)$ against t were made for PSP at different initial dye concentrations (Figs. 6 and 7). The intercept of the above plot should equal to $\log q_e$. However, if the intercept does not equal to the equilibrium dye uptake then the reaction is not likely to be first order even this plot has high correlation coefficient with the experimental data [19].

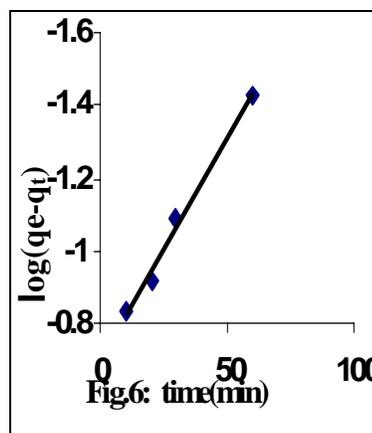


Fig. 6. First-order model of Methylene Blue

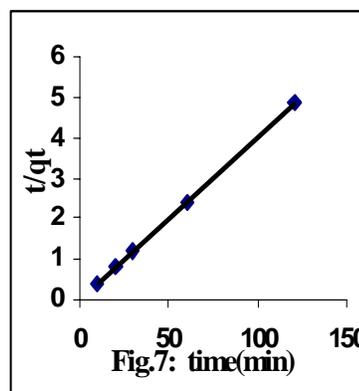


Fig. 7. Second-order model of Methylene Blue

Correlation coefficients were found to be between 0.9724 and 0.9925 of Rh6G and MB, but the calculated q_e was found to be not equal to experimental q_e , suggesting the

Table 6. Freundlich Isotherm Constants								
Name of adsorbent	Freundlich Constants				Langmuir Constants			
	K_f	$1/n$	N	R^2	Q^o (mg/g)	b (L/mg)	R_L	R^2
Rhodamine 6G	-2.4654	1.9786	0.5054	0.925	19.6531	-0.2048	-0.1035	0.9813
<i>Methylene Blue</i>	0.6605	0.7886	1.2681	0.9449	4.2512	2.4507	0.0399	0.9423

insufficiency of pseudo-first-order model to fit the kinetic data for the initial dye concentrations examined. The rate constants and the correlation coefficients for all concentrations tested have been summarized in Table 7.

Dye	pseudo-first order kinetic model		pseudo-second order kinetic model		
	K_1	R^2	K_2	H	R^2
MB	-0.0122	0.9874	0.4038	2.47315	0.9966
Rh6G	-0.0113	0.9537	0.4013	2.4593	0.9333

The negative values of the rate constant suggest that pseudo first order is not a good fit. According to the pseudo-second order model the dye adsorption kinetic is described by the equation,

$$T/q_t = 1/K_2 \cdot q_e^2 + t/q_e$$

where K_2 is the rate constant of second order adsorption (g/mg. min) and $K_2 \cdot q_e^2 = h$ is the initial adsorption rate (mg/g. min). By plotting T/q_t versus t , a straight line could be obtained and q_e , K_2 and h can be calculated [20]. Good fits were observed for all initial concentrations (Figs. 8 and 9) indicating that adsorption reaction can be approximated with the Pseudo-second-order model.

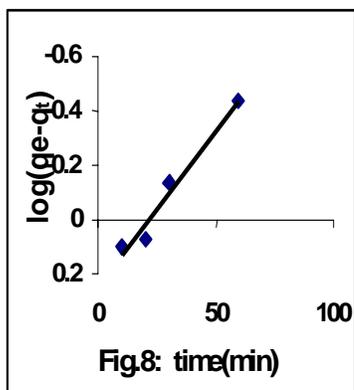


Fig. 8. First-order model of Rhodamine 6G

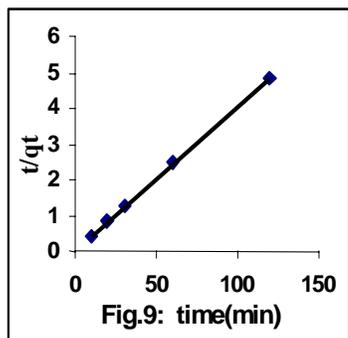


Fig. 9. Second-order model of Rhodamine 6G

Correlation coefficients are tabulated along with the rate constants in Table 8, which were found to be 0.9966 and 0.9333 for MB and Rh6G respectively, and the calculated q_e values obtained from the second-order kinetic model agree with the experimental q_e values for both the cases.

Name of adsorbate	q_e (mg/g)		
	Initial	Theoretical	Calculated
MB	25	24.748	25.2795
R6G	25	24.756	25.7445

These suggest that the adsorption of dyes onto PSP follows the second-order kinetic model and chemical adsorption might be the rate-limiting step.

Since PSP in our experiments has very high equilibrium adsorption capacity q_e , the adsorption rates are very fast and the equilibrium times short. The adsorption capacities q_t of the dyes at 10 min for almost all the initial concentrations reached over 98% of the calculated equilibrium adsorption capacities. Such short equilibrium times coupled with high adsorption capacity indicate a high degree of affinity between the dyes and PSP [21].

In order to know rate-controlling step in adsorption process, the applicability of the Bangham's equation [22] to present dye adsorption studies was tested:

$$\text{Loglog} [Co'/Co'-q' m'] = \log [k_0 m'/2.303V] + \alpha \log t$$

where Co' is the initial concentration of adsorbate in solution (mmoldm^{-3}), V the volume of solution (ml), m' the weight of adsorbent used per liter of solution (g dm^{-3}), q' the amount of adsorbate (m mol g^{-1}) retained at time t and α (<1) and k_0 are constants. As such $\text{Loglog} [Co'/Co'-q' m']$ was plotted against $\log t$ (Figs. 10 and 11).

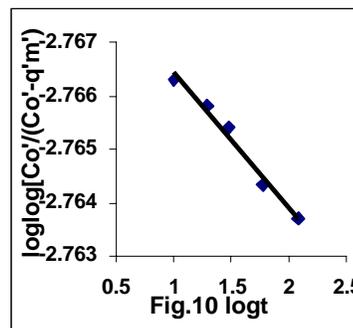


Fig. 10. Bangham plot for Methylene Blue

The plots obtained were linear with good correlation coefficients (MB: 0.9789, Rh6G: 0.9499). The results thus conform to Bangham's equation and indicate that the adsorption of the dye is pore diffusion controlled process [22-23].

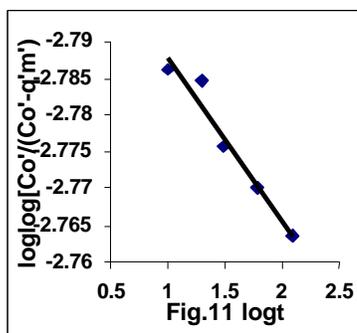


Fig. 11. Bangham plot for Rhodamine 6G

5. Conclusions

Palm Shell Powder is a promising adsorbent for removal of the cationic dyes, Methylene Blue and Rhodamine 6G from water. The surface of the PSP particles was heterogeneous, non-specific and non-uniform in nature. Different operational parameters observed during the process of investigations reveal that the pH, temperature, contact time, adsorbent dose and concentrations of the adsorbate govern the overall process of adsorption. The batch studies clearly demonstrate that there is > 94 % removal for concentrations as high as 600 ppm. The exhaustive capacities determined are 121.5 and 105.0 mg/g of adsorbent for MB and Rh6G respectively. The equilibrium adsorption is achieved in 120 min. The adsorption of Methylene Blue on PSP was exothermic. However Pseudo second order model is the best fit for the adsorption of dyes onto PSP suggests that the adsorption of dyes by PSP may involve not only physisorption but also chemisorption. PSP was found to be more effective than CAC for the removal of MB and Rh6G. In addition it is a low cost adsorbent. PSP was found to be effective for the removal of Rh6G from effluent.

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