

Evaluation of adsorption potential of adsorbents: A case of uptake of cationic dyes

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Abstract: Adsorption potential of a commercial activated carbon (FS300) has been evaluated for the uptake of cationic dyes namely methylene blue (MB) and rhodamine B (RB). Though, there are numerous studies in literature which report the sorption of MB (more than 40 studies) and RB (more than 10), however none of these use a common parameter to report the capacity of the sorbent. A protocol, based on the equilibrium dye concentration has been proposed to measure the sorption potential of a sorbent. The Langmuir model can very well describe the experimental equilibrium data for both dyes (coefficient of correlation > 0.999). MB ($Q_m = 312.5 \text{ mg g}^{-1}$) is more adsorbable than the RB ($Q_m = 144.9 \text{ mg g}^{-1}$). Molecular weight and chemical structure of dye molecules seem to affect the dye uptake. The effect of pH on dye uptake has also been evaluated by varying pH from 3 to 11. Uptake of MB increases with pH, wherein RB removal decreases with pH. Dyes could not be desorbed either by distilled water (0.06 and 0.11 % for MB and RB respectively) ,or by 0.1 N HCl (0.136 and 3.0% for MB and RB respectively) indicating, chemical adsorption type of adsorbent-adsorbate interactions.

Key words: Adsorption, Activated carbon, Cationic dye, Equilibrium isotherm and pH
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Introduction

The color contamination of the aqueous environment is not desirable from environmental, aesthetical and economical point of view. Industries like textile, paper and pulp, distillery, cosmetic, food and dye producing industry discharge colored wastewater (McKay *et al.*, 1999). Even a very low concentration of dye can make water unacceptable for various purposes. Presence of color in water prevents sunlight penetration in receiving water and consequently reduces biological activities and selfpurification capacity of water due to reduction in process photosynthesis. Some dyes are carcinogenic and mutagenic (Mathur and Bhatnagar, 2007), and causes many water born diseases, such as nausea, haemorrhage, ulceration of skin and mucous membrane, dermatitis, perforation of nasal septum and severe irritation of respiratory tract (Jogdand, 2000; Soni *et al.*, 2006; Mathur and Bhatnagar, 2007).

Several adsorbent have been reported in literature for dye removal such as commercial activated carbon, activated carbon prepared from waste materials in different conditions, fly ash and some organic material based adsorbents. Though, many of them have high uptake capacity, efficacy of an adsorbent in terms of high uptake capacity is not a guarantee for high removal capacity at lower concentration of dye. There is no universally acceptable method to evaluate various adsorbents. So, it is contemplated to propose a protocol, which could be used to compare the efficiency of different adsorbents. Methylene blue and rhodamine B, both are cationic dyes of different chemical class, has been selected as the candidate adsorbates. Parametric studies have also been carried out to study the effect of pH, ionic strength and type of regenerants.

Materials and Methods

The activated carbon FS300 (Chemviron GmbH) was used as the adsorbent. Before use, it was crushed in an electric ball mill and sieved through 600 μm sieve. The sieved adsorbent was kept in oven at temperature $40 \pm 1^\circ\text{C}$ for a period of 24 hr. Dried adsorbent was stored in a desiccator for use. Dyes namely methylene blue (MB) and rhodamine B (RB) were selected as representative sample of cationic dye. Aqueous dye solutions of concentration 100 mg l^{-1} were prepared in extraction water. The extraction water was prepared using ultra pure (millipore) water and adding 0.5, 0.3, 0.2 mM NaHCO_3 and $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, and $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ respectively. The pH of extraction water was adjusted to 7.5 ± 0.2 .

Bottle point isotherm experiments were carried out for the determination of dye uptake capacity of the adsorbent. Fifty ml dye solution was taken in a 100 ml wide mouth bottle (Duran, Germany) and varying dosages of adsorbent were added to each bottle. To study the effect of pH, the initial pH of the dye solution was varied from 3 to 11 using 0.1N HCl/NaOH. Dye solutions (50 ml) were contacted with varying amounts of adsorbents. In all experiments, the samples were agitated using a rotary shaker for a period of 48 hr at 30 rpm. Temperature was maintained $20 \pm 1^\circ\text{C}$. All samples were taken in duplicate. Upon equilibration, dye solution was separated from adsorbent using $0.45 \mu\text{m}$ membrane filter. The dye uptake by the sorbent was determined by subtracting the dye concentration remaining in the bottle after sorption from the initial dye concentration in the bottle. Initial and final dye concentrations were measured spectrophotometrically.



Desorption studies were carried out in two phases -

(a) **Sorption:** The calculated weight of adsorbents were added to 1000 ml dye solution (100 mg l⁻¹, pH-7.5±0.2, temperature 20±1°C) and kept on rotary shaker (30 rpm) for 48 hr. The equilibrium concentration of dye solution was measured. Adsorbents were separated from dye solution using 0.45 µm membrane filter. The filtrate was dried in oven at 40±1°C for 48 hr.

(b) **Desorption:** Equal amount of dye loaded adsorbents were added to 50 ml extraction water and 0.1 N HCl in duplicate and kept on rotary shaker for 48 hr. The equilibrium dye concentration of solution was measured.

Results and Discussion

Adsorption from aqueous solution involves transfer of adsorbate from the aqueous phase to the activate sorption sites on the solid phase. The adsorption isotherm obtained under experimental conditions (at temperature 20±1°C and pH 7.5±0.2) for the sorption of dyes namely MB and RB onto activated carbon is shown in Fig. 1. The shape of adsorption isotherm indicates type of interaction between adsorbate and adsorbent. Fig. 1 indicates that there is a steep increase in adsorption capacity with increase in effluent concentration. On further increase in effluent concentration, the shape of adsorption isotherm achieves a well defined plateau, indicating the saturation capacity of adsorbent. This type of adsorption isotherm may be classified as Type "1" or "H". Type "H" isotherm indicates a very high adsorptive affinity and strong adsorbent - adsorbate interaction (Avom *et al.*, 1997; Martin *et al.*, 2003).

Modelling of adsorption isotherm data: Though, a number of isotherm models have been proposed to describe the equilibrium distribution of an adsorbate between two phases (in this case, aqueous and solid). Langmuir model which is based on theoretical considerations, having 2 degrees of freedom, is mathematically easy to solve. Besides, due to its theoretical derivation, Langmuir model could be used to delineate the mechanistic aspects of the adsorbate - adsorbent interaction. So, the equilibrium isotherm data obtained from the experimental study have been modelled using most commonly available model - "Langmuir" model. Besides, the Langmuir isotherm model has been widely used to model dye adsorption data (McKay, 1996; Martin *et al.*, 2003), so it could be used for the ready comparison with the published data. The isotherm model can be expressed as:

$$Q_e = \frac{Q_m b C_e}{1 + b C_e} \quad (1)$$

Where Q_m (mg l⁻¹) and b (mg l⁻¹) are the isotherm constants obtained by linearised plot of $\frac{C_e}{Q_e}$ versus C_e for a particular adsorbate-adsorbent combination.

The isotherm constants for MB and RB are presented in Table 1. In all cases Langmuir model provides a good fit ($r^2 >$

0.999), indicating the applicability of this model for describing the sorption equilibrium. Langmuir isotherm is valid for monolayer adsorption on a surface containing a finite number of identical sites. It assumes uniform adsorption energy on the surface of the adsorbent and no transmigration of adsorbate in the plane of the surface (Namasivayam and Arasi, 1997). It means, once a dye molecule occupies a site, no further adsorption can take place at that site and a saturation value, *i.e.*, Q_m , is reached which corresponds to the completion of monolayer.

Dye removal capacity - An approach: Batch equilibrium isotherms could be used to determine sorption capacity of an adsorbent. Adsorption isotherms could be used to evaluate the capacity of an adsorbent (Gupta *et al.*, 2003). But, there is no universally acceptable method to obtain a single value that would be used to specify the capacity of the adsorbent for a given adsorbate-adsorbent system. For example, in case of Langmuir model, it is not clear that out of model constants, b and Q_m , which one should be used to measure the capacity of the biosorbent. Isotherm constants b and Q_m denote heat of adsorption and capacity of adsorbent respectively. The natural choice could be Q_m , since it represents capacity of the adsorbent. But, Q_m is saturation capacity or the ultimate capacity of the adsorbent for a given adsorbate-adsorbent system.

Recent data show that the Langmuir constants, monolayer saturation capacity Q_m , vary from 4.3 to 980.3 mg g⁻¹ and b from 0.0007 to 204 mg l⁻¹ for MB (Table 1). These constants for RB vary from 2.6 to 203.2 mg g⁻¹ for Q_m and 0.03 to 32 mg l⁻¹ for b (Table 2). While, in the present study, the monolayer saturation capacity is 312.5 and 144.9 mg g⁻¹, and b are 4.0 mg l⁻¹ and 8.625 mg l⁻¹ for MB and RB respectively. However, a comparison of the Q_m values for the adsorption of MB ten adsorbents out of 50 has sorption capacity more than FS300. Similarly for RB, the Q_m value of one adsorbent (coir pith) is more than the Q_m for FS300. It indicates, that Q_m may not be true representative of adsorption capacity of an adsorbent, as at low equilibrium concentration, the Q_m does not show same trend. Thereby, a comparison of capacities based on Q_m would be erroneous.

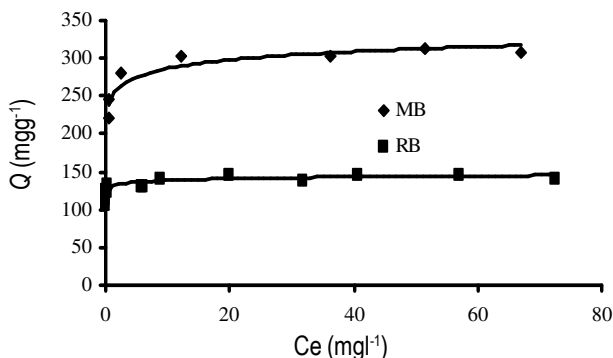
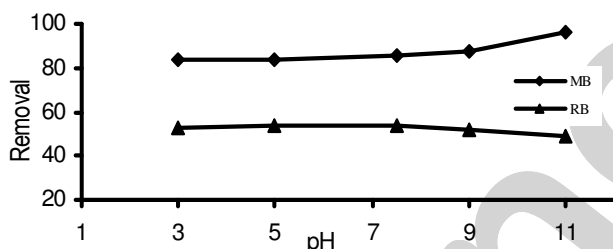
Langmuir constants for the adsorption MB and RB by a number of adsorbents are presented in Table 1, 2. These include the published values as well as the Langmuir constants obtained in the present study. Using these isotherm constants (Table 1, 2), adsorption capacities have been calculated at equilibrium adsorbate concentrations of 0.1, 0.5 and 1.0 mg l⁻¹, which are presented in columns 4, 5 and 6 of Table 1, 2. These dye concentrations have been selected on the basis of their ADMI values, since the colour of aqueous solutions could be represented in terms of ADMI values (APHA, 2005). ADMI values for dye concentrations of 0.1, 0.5 and 1.0 mg l⁻¹ have been determined as 24.7, 87.4 and 259.2 for MB; 113.1, 464.6 and 963.3 for RB respectively (Table 1, 2). Even at low dye concentrations (0.1 mg l⁻¹) the ADMI value for MB and RB are 24.7 and 113.4 respectively.

Table - 1: Comparison adsorption capacity for methylene blue at different equilibrium concentration

Adsorbent	Langmuir constant		Equilibrium concentration, mg l ⁻¹			Reference
	Q _m (mg l ⁻¹)	b (mg l ⁻¹)	0.1 mg l ⁻¹	0.5 mg l ⁻¹	1 mg l ⁻¹	
AC (E.Merk, India)	980.3	0.479	44.809	189.417	317.487	Kannan and Sundaram (2001)
AC prepared from bamboo dust	143.2	0.12	1.698	8.106	15.343	„
AC prepared from coconut shell	277.9	0.091	2.506	12.094	23.180	„
AC prepared from groundnut shell	164.9	0.128	2.084	9.919	18.712	„
AC prepared from rice husk	343.5	0.358	11.872	52.151	90.554	„
AC prepared from straw	472.1	0.389	17.677	76.872	132.215	„
AC Chemviron GW	215.1	0.67	13.507	53.976	86.298	San Miguel <i>et al.</i> (2002)
AC Hydrodarco-C	156.7	1.39	19.123	64.252	91.135	„
AC prepared from waste tyre rubber	49.0-448.4	0.05-0.6	0.243-19.397	1.195-79.778	2.333-128.114	„
AC prepared from activated sludge	85.47	1.487	11.064	36.448	51.103	Graham <i>et al.</i> (2001)
AC prepared from activated sludge and coconut husk	76.92	1.262	8.620	29.759	42.915	„
AC prepared from activated sludge and peanut shell	95.24	3.182	22.990	58.482	72.466	„
Banana peel	20.8	16.5	12.951	18.551	19.611	Annadurai <i>et al.</i> (2002)
Orange peel	18.6	19.9	12.379	16.901	17.710	„
Chemically treated psidium Guyava leaves (30°C - 60°C)	133.33-175.40	0.29-0.38	3.757-6.421	16.884-28.00	29.973-48.298	Singh and Srivastava (1999)
Sunflower stalks	205.4	0.0502	1.026	5.029	9.818	Sun and Xu (1997)
Dead fungal biomass <i>A. niger</i> (pH10)	54.67	0.14	0.755	3.577	6.714	Fu and Viraraghavan (2000)
Chitin (at different temperature and particle size)	304.62-396.57	0.0021-0.0037	0.091-0.113	0.455-0.563	0.91-1.123	Annadurai and Krishnan (1996)
Chitosan (at different temperature and particle size)	320.51-713.79	0.001-0.0032	0.103-0.032	0.160-0.512	0.320-1.022	Annadurai <i>et al.</i> (1997)
Untreated and treated sawdust	15.4-30	0.0182-0.031	0.028-0.093	0.139-0.458	0.275-0.902	El-Nabarawy and Khedr (2000)
Untreated and treated water hyacinth	4.3-15.3	0.0628-0.0582	0.113-1.25	0.511-4.711	0.914-7.204	„
Cotton waste	277.77	0.0093	0.258	1.286	2.559	McKay <i>et al.</i> (1999)
Rice husk	312.25	0.0172	0.536	2.662	5.280	„
Bark	914.58	0.0173	1.579	7.843	15.553	„
Coal	323.68	0.0036	0.116	0.582	1.161	„
Hair	158.23	0.0194	0.306	1.520	3.011	„
Peat	-4.04	-2.47	1.325	-21.231	-6.788	Ramakrishnan and Viraraghavan (1997)
Bentonite	46.3	36	36.235	43.863	45.049	„
Slag	-4.86	-0.005	0.002	0.012	0.024	„
Fly ash	54.05	0.0007	0.004	0.019	0.038	„
Fly ash	54.05	0.01	0.054	0.269	0.535	Viraraghavan and Ramakrishnan (1999)
Boiler bottom ash (25°C)	3.984	1.695	0.577	1.828	2.506	Mall and Upadhyay (1995)
Boiler bottom ash (30°C)	4.732	1.631	0.664	2.126	2.933	„
Boiler bottom ash (40°C)	5.153	1.582	0.704	2.276	3.157	„
Boiler bottom ash (50°C)	6.113	1.546	0.819	2.665	3.712	„
Raw kaolin	13.99	27.49	10.258	13.041	13.499	Ghosh and Bhattacharya (2002)
Pure kaolin	15.55	91.87	14.024	15.219	15.383	„
Calcinee raw kaoline	7.59	13.44	4.352	6.607	7.064	„
Calcined pure kaolin	8.88	56.31	7.541	8.575	8.725	„
NaOH treated raw kaolin	16.34	204	15.576	16.181	16.260	„
NaOH treated pure kaolin	20.49	122.01	18.938	20.160	20.323	„
<i>Fomes fomentarius</i>	232.73	0.140	3.213	15.225	28.581	Maurya <i>et al.</i> (2006)
<i>Phellinus igniarius</i>	204.38	0.184	3.693	17.219	31.762	„
FS300	312.5	4	89.286	208.333	250.000	Present study

Table - 2: Comparison adsorption capacity for rhodamine B at different equilibrium concentration

Adsorbent	Langmuir constant		Equilibrium concentration, mg l ⁻¹			Reference
	Q _m (mg l ⁻¹)	b (mg l ⁻¹)	0.1 mg l ⁻¹	0.5 mg l ⁻¹	1 mg l ⁻¹	
Banana pith	8.5	0.034	0.029	0.142	0.279	Namasivayam et al. (1993)
Orange peel	3.23	0.049	0.016	0.077	0.151	Namasivayam et al. (1996)
Coir pith	203.2	0.03	0.608	3.003	5.918	Namasivayam et al. (2001)
Carbonized coir pith	2.6	2.74	0.559	1.503	1.905	Namasivayam et al. (2001b)
Banana peel	20.6	32.6	15.764	19.409	19.987	Annadurai et al. (2002)
Orange peel	14.3	32	10.895	13.459	13.867	„
<i>Fomes fomentarius</i>	25.12	1.290	0.125	0.613	1.196	Maurya et al. (2006)
<i>Phellinus igniarius</i>	36.82	4.010	0.099	0.490	0.968	„
FS300	144.9	8.625	67.101	117.625	129.845	Present study

**Fig. 1:** Adsorption isotherm for the uptake of methylene blue (MB) and rhodamine B (RB) by FS300**Fig. 2:** Effect of pH on dye uptake

In actual application of an adsorbent, the desirable concentration of the adsorbate in aqueous phase is almost zero. Specifically, in processes related to color removal, where dyes (like MB and RB) have high tinctorial values, very low equilibrium aqueous phase concentration of dyes is desirable. Thus, capacity of adsorbents at low equilibrium concentrations should be compared. At equilibrium concentration 0.1 mg l⁻¹, the adsorption capacity varies from 0.004 to 44.809 mg g⁻¹ for MB and 0.019 to 189.417 mg g⁻¹ for MB and 0.016 mg g⁻¹ to 15.764 mg g⁻¹ for RB (Table 1, 2). While at this equilibrium concentration activated carbon FS300 has a capacity of 89.286 mg g⁻¹ for MB and 67.101 mg g⁻¹ for RB. Thus, in comparison to other adsorbents, activated carbon FS300 provides high adsorption capacity at lower effluent concentration.

Differences of MB and RB adsorption: The monolayer saturation values are 312.5 and 144.9 mg g⁻¹ for MB and RB

respectively. MB is more adsorbable than RB. It may be due to smaller ionic radius and lighter molecular weight of MB than RB. The ionic radius of MB and RB are 15 Å and 23.3 Å respectively (Haglund et al., 1996). So only pores, larger than the 23.3 Å (ionic radius of RB at pH 7) are accessible to RB. Tamai et al. (1999) reported that pore size is more important than the Brunauer, Emmett, Teller (BET) surface area. Walker and Weatherly (2001) explained, only 14% of the BET surface area of commercial activated carbon (FS400) was available for dye adsorption. Besides molecular size, difference in molecular structure of these two dyes may be other reason for the variation in the dye adsorption capacity. MB contains three benzene rings in linear structure having no side chain, while RB contains four benzene rings having one side chain with acidic colligative i.e., a carboxylic group (-COOH). RB is more aromatic compound. MB is grouped as cationic dye having wholly basic nature, while RB is known as cationic dye having acidic colligative (Gurr, 1971). RB molecule also contains a carboxylic group (-COOH), which has pH equal to 4.2. As pH is raised above 4.2, there is a decrease in the overall charge of RB. Thus at pH 7.5, at which isotherm experiments have been carried out, RB is almost neutral, whereas MB carries net positive charge. Thus, from the present discussion, it seems that presence of carboxyl group and higher number of benzene rings and side chain of benzene ring in RB molecule make it less adsorbable than MB. McCreary and Snoeyink (1980) have also found similar results.

Effect of pH: The effect of pH on the percent removal of the dyes by activated carbon is shown in Fig. 2. The removal (%) of dyes is studied at pH 3.0, 5.0, 7.5, 9.0 and 11.0. The removal of MB is favorable at higher pH. As pH is raised from 3.0 to 7.5, there is negligible increase in removal of MB. Above pH 7.5, the removal increases sharply with pH. The maximum removal of MB is observed at pH 11. Though RB is also a cationic dye, it shows a reverse trend. Percent removal is more in acidic range.

Above pH 7.5, removal of RB decreases with increase in pH. pH effect is negligible in pH 3.0-7.5. The variation in the uptake of MB and RB with pH can be understood by considering the difference in the structure of the dye molecules along with the change in zeta potential of the activated carbon.

The pH_{ZPC} (pH of zero zeta potential) of commercially available activated carbons FS400, 781-A, chemviron GW and N-72 are 7.2, 6.2, 6.5 and 5.7 respectively (Huang and Wu, 1977; Dai, 1998; Martin *et al.*, 2003; Al-Degs *et al.*, 2000). Therefore, it can be assumed that pH_{ZPC} of FS300 will also has in the same range *i.e.* pH 5.5 to 7.5. Al-Degs *et al.* (2000), have reported that surface of the activated carbon N-722 is negatively charged above the pH_{ZPC} , and positively charged below the pH_{ZPC} . The magnitude of surface charge of activated carbon decreases with increase in pH (Martin *et al.*, 2003). Thus, electro negativity of activated increases with increase in pH. The lower removal of positively charged MB dye at acidic pH range is probably due to the presence of excess H^+ ions competing with the dye molecule for the adsorption site. At alkaline pH, adsorbent surface gets net negative charge; therefore, there is an attractive electrostatic force between MB molecule (positively charged dye molecules) and adsorbent. The decrease in removal of RB with increase in pH above pH 7.5 can be attributed to the presence of an acidic group. The acidic group (carboxylic group) may dissociate as the pH increases, giving rise to a net negative charge on RB dye molecule (Gupta *et al.*, 2000).

Desorption studies: Pre saturated adsorbents with dyes namely methylene blue and rhodamine B were subjected for desorption in 50 ml extraction water and 0.1N HCl solution. Desorption studies can aid in elucidating the dye adsorption mechanism. Desorption of dyes by distilled water and 0.1N HCl give the value of desorption and regeneration of adsorbent (McKay *et al.*, 1987). The desorption value is 0.06 and 0.11% and regeneration is only 0.136 and 3.0% for MB and RB respectively. Desorption and regeneration values are very less for both dyes, indicating perhaps, there is strong chemical bonding between the dye molecule and activated carbon.

Performance of commercial activated carbon FS300 for the removal of cationic dyes namely methylene blue (MB) and rhodamine B (RB) has been evaluated by carrying out the equilibrium isotherm study at 20°C and pH 7.5. For the mathematical modeling of experimental data, Langmuir model is used. Langmuir model fits experimental data with high value of correlation coefficient ($r^2 > 0.99$) for both the dyes. The dye adsorptive capacity of the FS300 activated carbon has been compared with several adsorbents. The proposed methodology to compare the adsorption potential of an adsorbent seems better compared with the comparison of model coefficients, which represent the capacity of the adsorbent. FS300 shows moderate Langmuir monolayer saturation capacity, but it showed relatively high adsorption capacity at low effluent concentration than the other adsorbents. The removal of MB is favourable at higher pH whereas RB is favourable at lower pH under present study. Presence of salt improves the MB uptake. Desorption and regeneration values are very less for both dye. Thus, there is a strong chemical bonding between the dye molecule and activated carbon.

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