Original Article

Removal of health hazards causing acidic dyes from aqueous solutions by the process of adsorption

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Abstract:

The association of dyes, particularly acidic dyes with health related problems is not a new phenomenon. A lot of well established literature is already available on the role of dyes as a major cause in skin and respiratory diseases. The adsorbents which are of low cost, locally available and are relatively new for their acidic dyes removal capacity from aqueous solutions were investigated. Bagasse, cow dung, groundnut shells, pea shells, used tea leaves, wheat straw were used in their charcoal form whereas brick kiln ash and cement kiln ash adsorbents were used as such for the removal of acid violet 17, acid violet 49, acid violet 54, acid blue 15 and acid red 119. The effects of various experimental parameters, initial pH, dye concentration, sorbent dosage, ion strength, contact time were examined and optimal experimental conditions were decided. At initial basic pH more than 8.0, all the five dyes studied could be removed effectively. The isothermal data for adsorption followed the Freundlich and Langmuir models. The adsorption results in this study indicated that all the adsorbents were attractive candidates for removing acidic dyes from dye wastewater.

Key Words: Acidic dyes, Adsorbents, Health hazard

Introduction:

Synthetic dyes have been increasingly used in the textile, paper, rubber, plastic, cosmetics, pharmaceutical and food industries because of their ease of use, inexpensive cost of synthesis, stability and variety of colour compared with natural dyes.[1-3] Today there are more than 10,000 dyes available commercially.[4] most of which are difficult to biodegrade due to their complex aromatic molecular structure and synthetic origin.[5] The extensive use of dyes often poses pollution problems in the form of coloured wastewater discharge into environmental water bodies, which interferes with transmission of sunlight into streams therefore reduces photosynthetic activity.[6] In addition, some dyes or their metabolites are either toxic or mutagenic and carcinogenic. A lot of cases throughout the world are reported about the role of dyes in connection with variety of skin, lung, and other respiratory disorders.[7] Use of variety of dyes and chemicals in the dyeing processes causes considerable variation in the wastewater characteristics like pH, colour and chemical oxygen demand (COD). The presence of these heat and light stable, complex dye molecules in wastewater made the conventional methods of sewage treatment, such as primary and secondary treatment systems, unsuitable.[7] The adsorption process provides an attractive alternative treatment, especially if the adsorbent is inexpensive and readily available. Granular activated carbon is the most popular adsorbent and has been used with great success[8], but is expensive. Consequently, many investigators have studied the feasibility of using low cost substances, such as plum kernels[9], chitin[10], chitosan[11], perlite[12], natural clay[13], bagasse pith[14], fly ash[15], boiler bottom ash[16], bagasse fly ash[17], rice husk[18,19], peat[20], banana pith[21] orange peel[22], Eichhornia ash[23], saw dust[24,25] , walnut shells charcoal[26], etc. as adsorbents for the removal of dyes from wastewaters. Critical review of low cost adsorbents for waste and wastewater treatment has been represented by Pollard et al.[27], Mall et al.[28] and Bailey et al.[29]

The purpose of this work was to investigate the possibility of bagasse charcoal (BC), brick kiln ash (BKA), cement kiln ash (CKA), cow dung charcoal (CDC), groundnut shells charcoal (GNC), pea shells charcoal (PSC), used tea leaves charcoal (UTC), wheat straw charcoal (WSC) materials which were obtained from the local market. Brick kiln ash and cement kiln ash were used as such whereas all other adsorbents were used by making their charcoal for the removal of acidic dyes from aqueous solution. All the biomaterials are low cost agricultural waste residues and are easily available in large quantity in India. The dyes selected as sorbate were Acid Violet 17 (AV17), Acid Violet 49 (AV49), Acid Violet 54 (AV54), Acid Blue 15 (AB15) and Acid Red 119 (AR119). The effects of various operating parameters on biosorption such as initial pH and dye concentration, sorbent dosage, ion strength, contact time were monitored and optimal experimental conditions were decided.

Materials and Methods:
Reagents

The following reagents, procured from different companies were used as needed for the determination of COD and other experimental purposes:

Potassium dichromate (99.8%, S.D. Fine Chemicals, India), Ferrous ammonium sulphate (99.9%, BDH Laboratory Reagent, India), 1,10-Phenanthroline (99.9%, SISCO Research Laboratory, India), Silver sulphate

(99.5%, BDH Laboratory Reagent, India), Mercuric sulphate (99.5%, Ranbaxy, India), Sulphuric acid, HCl and NaOH (AR grade, Ranbaxy, India)

Preparation of different adsorbents

All the adsorbents used in this study were obtained from a local market. The collected adsorbents; bagasse, dry cow dung, groundnut shells, pea shells, used tea leaves, wheat straw were extensively washed with tap water to remove soil and dust, sprayed with distilled water and then dried in an oven at 80°C. Then the adsorbent materials were burnt in the absence of free excess of air to get their respective charcoals. The different charcoal obtained were sieved to different particles sizes and then their particles in the size ranges between 200 –250 microns were preserved in the desiccator for further studies. However, brick kiln ash and cement kiln ash were obtained from respected plants and then sieved through sieves to get particles of sizes in ranges between 200-250 microns.

Preparation of dye solutions: The dyes used in this study are listed in Table 1. Their chemical structures are shown in Fig.1.
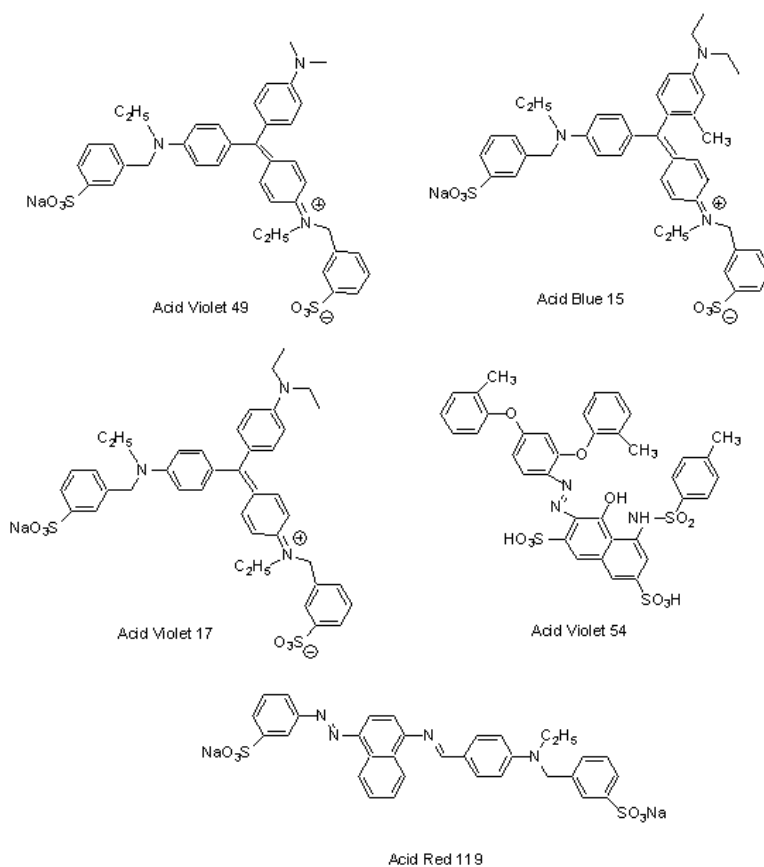

Fig. 1 The chemical structures of the acidic dyes used in this study

Five dyes (Acid Violet 17, Acid Violet 49, Acid Violet 54, Acid Blue 15, Acid Red 119) obtained from a local dye manufacturing company in commercial purity, were used without further purification. The dye stock solutions were prepared by dissolving accurately weighed dyes in distilled water to the concentration of 100 mg/l. The experimental solutions were obtained by diluting the dye stock solutions in accurate proportions to needed initial concentrations. The initial pH of each dye solution was adjusted with 0.1M HCl or NaOH using pH meter to its effective adsorption pH value.

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Table 1: The general data of the dyes used in this study

Commercial name	C.I. No	F.W.
Acid Violet 17	42650	761.8
Acid Violet 49	42640	733.4
Acid Violet 54	-	789.8
Acid Blue 15	42645	775.9
Acid Red 119	-	673

Experimental methods and measurements

Adsorption experiments were carried out in a rotary shaker at 150 rpm and 20 ± 2°C using 250 ml shaking flasks containing 100 ml of different concentrations at initial pH values of dye solutions. The initial pH values of solutions were previously adjusted with 0.1 M HCl or NaOH using pH meter. Different doses of sorbent were added to each flask and then the flasks were sealed to prevent change in volume of the solution during experiments. After shaking the flasks for predetermined time intervals, the samples were taken out from the flasks and the dye solutions were separated from the sorbent by filtration with 200 mesh stainless steel sieve and then centrifuged. Dye concentrations in the supernatant solutions were estimated by measuring COD, adopting standard APHA method [30].

The amount of dyes sorbed by the adsorbent was calculated using the following equation

$$q = (C_o - C_e) V / W$$

where W (mg/g) is the amount of dye sorbed by adsorbent, C_o and C_e (mg / l) are the initial and equilibrium liquid phase concentrations of the dye, respectively. V (l), the initial volume of dye solution and W (g), the weight of the adsorbent material.

The experiments were conducted in duplicate and the negative controls (with no sorbent) were simultaneously carried out to ensure that sorption was by adsorbent material and not by the container.

Results and Discussion
Effect of Contact Time

The values of equilibrium time of all the five acidic dyes with various adsorbent materials for COD reduction were estimated and are given in Table 2. It was observed that the rate of removal of acidic dyes increases with increase in contact time to some extent. Further increase in contact time does not increase the uptake due to deposition of dyes on the available adsorption site on adsorbent material.

Table 2: Equilibrium time of various dyes with different adsorbents

S.No.	Adsorbents	Equilibrium time
1	Bagasse charcoal	4 hours
2	Brick kiln ash	4 hours
3	Cement kiln ash	3 hours
4	Cow dung charcoal	4 hours
5	Groundnut shells charcoal	5 hours
6	Pea shells charcoal	4 hours
7	Used tea leaves charcoal	4 hours
8	Wheat straw charcoal	5 hours

Preliminary investigations on the rate of uptake of various dyes on the different adsorbent material indicated that Bagasse charcoal taken 4 hours in removing 78% of AR 119 and 74 % of AB15 whereas for other dyes removal is lesser than 50%. In case of Brick kiln ash 100% of AR119, 97% of AB15 and AV54, 98% of AV17 and 92% of AV49 was removed after 4 hours. Whereas, a time period of 3 hours was taken by cement kiln ash for removing 99% of AR119 & AV49, 90% of AV17, 65% of AB15 and 53% of AV54. Cow dung charcoal, however, was found comparatively lesser efficient as it took 4 hours and removal was only in between 40 and 60%. Groundnut shells charcoal took longer period of 5 hours in removing various acidic dyes and removal of 78 and 74% was observed respectively for AR 119 and AB15, however for other dyes, removal was less than 50%. Pea shells charcoal removed more than 90 % of dyes in each case in 4 hours. Used tea leaves charcoal took 4 hours in removing 92, 89, 72, 70 and 68% of AR119, AV54, AV 17, AB15 and AV 49, respectively. Wheat straw charcoal taken 5 hours in removing 87% of AV54, 70% of AB15, 58% of AV49, 41% of AV17 and 22% of AR119.

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Effect of pH

Because the initial pH of solution can significantly influence adsorption of dyes, the effects of pH on dye adsorption by the bagasse charcoal was studied first. The value of pH used ranged from 4 to 11.
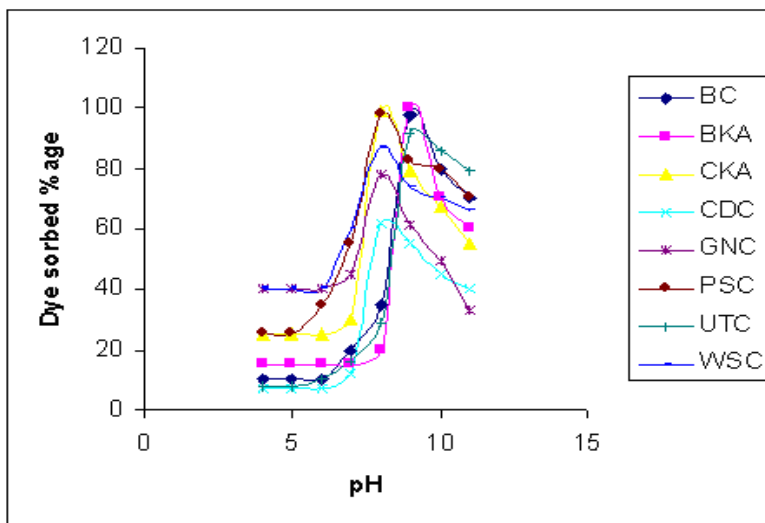

Fig. 2 Effect of initial pH on adsorption of acidic dyes by various adsorbents

As shown in Fig. 2, for all five dyes, the dye removal ratios were maximum when the initial pH medium was kept basic. The ratios of dyes sorbed decreased as the initial pH was decreased from 11 to 4, and, the dye removal ratios were maximum at pH 9 and then kept constant beyond pH 5. For this reason pH 9, where maximum removal was observed, was selected for all other experiments with bagasse. Similarly optimum working pH were found for other sorbents. It was found that pH of 8.9, 8.1, 8.5, 8.3, 8.2, 9.0 and 7.9 were optimal for brick kiln ash, cement kiln ash, cow dung charcoal, groundnut shells charcoal, pea shells charcoal, used tea leaves charcoal and wheat straw charcoal materials, respectively.

Effect of Dye Concentration

The influence of dye concentration on adsorption percentages of dyes was estimated. The effect of dye concentration was studied by keeping the adsorbent dose constant at 1 g. For adsorption study, in the optimized period of contact time, the rate varies directly with concentration of adsorbate. The activities of all the adsorbent materials fall sharply with an increase in the initial concentration of dye. It is well known fact that rate of exchange adsorption controlled by diffusion through a hydrostatic boundary layer called film diffusion control or through the pores of the region matrix called particle, diffusion control. The rate of exchange adsorption is mainly controlled by film diffusion under the conditions of small resin particle, dilute solution and mild stirring and vice-versa in case of pore or particle diffusion. More practically, both processes control it.[31] The three distinct steps involves in the adsorption of an organic/inorganic compound onto the pores of adsorbent material are:

1. The adsorbent molecules must be transferred from the bulk phase of the solution to the external surface of the adsorbent particles is called as 'film diffusion'.
2. Transfer of adsorbent molecules to an adsorption site on the inside of the adsorbent particle is known as pore or 'particle diffusion'.
3. The adsorbate particle must become attached to the interior surface of the adsorbent particle that is 'adsorbed'.

In case of particles diffusion external transport > internal transportation and in film diffusion external transport < internal transportation and if external transportation is approximately equal to internal transportation, the transport of ions to the boundary may not be possible at significant rate.
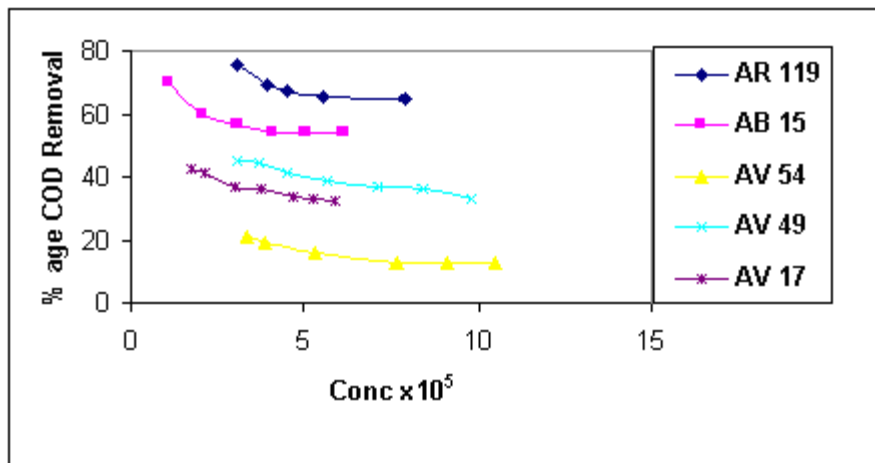
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Fig. 3 Influence of dye concentration on adsorption of various dyes by bagasse charcoal

As shown in Fig. 3, there was regular decrease in percentage of COD removal when the concentration of dyes were increased, for example in case of bagasse the percentage of COD removal were decreased from 78 to 70% in AV119, 74 to 58% in ABV15, 48-38% in AV49, 44-36% in AV17 and 20-10 in AV54 . Similarly, in case of brick kiln ash the percentage of COD removal decreased from 100 to 93%, 98 to 92%, 97 to 89%, 97 to 88% and 92 to 80% for AR119, AV17, AB15, AV54 and AV49 respectively. In case of cement kiln ash the percentage of COD removal decreased from 99 to 90%, 99 to 88%, 90 to 80%, 65 to 58% and 53 to 49% for AR119, AV49, AV17, AB15 and AV54, respectively. And in case of cow dung charcoal the percentage of COD removal decreased from 60 to 50%, 62 to 48%, 52 36%, 37 to 30% and 44 to 38% for AR 119, AB15, AV54, AV49 and AV17, respectively. In case of groundnut shells charcoal the percentage of COD removal decreased from 78 to 70%, 74 to 68%, 22 to 12%, 50 to 38% and 42 to 35% for AR 119, AB15, AV54, AV49 and AV17, respectively. In case of pea shells charcoal the percentage of COD removal decreased from 97 to 93%, 98 to 92%, 90 to 82 %, 93 to 81 and 98 to 95 % for AR 119, AB15, AV54, AV49 and AV17, respectively. In case of used tea leaves charcoal the percentage of COD removal decreased from 92 to 88%, 70 to 55%, 89 to 78%, 68 to 48% and 72 to 58% for AR 119, AB15, AV54, AV49 and AV17, respectively. In case of wheat straw charcoal the percentage of COD removal decreased from 22 to 18%, 70 to 60%, 87to 80%, 58 to 48% and 41 to 38% for AR 119, AB15, AV54, AV49 and AV17, respectively.

With the data for Freundlich and Langmuir equations were employed to study the sorption isotherms of dyes.

The Freundlich equation was linearized as follows:

$$\log \frac{x}{m} = \log k + \frac{1}{n} \log Ce$$ [32]

where x/m is amount of acid dye adsorbed (x) per unit weight (m) of adsorbent, Ce, is the equilibrium concentration, k and n are empirical constants and their values were obtained from the intercept (log k) and slopes (1/n) of linear plots of log x/m versus log Ce.

The Langmuir equation is shown as follows:

$$\frac{Ce}{X} = \frac{Ce}{Cm} + \frac{1}{bCm}$$ [32]

Where Ce (mg/l) is the concentration of the dye solution at equilibrium, X (mg/g) is the mass of dye adsorbed (x) per gram (m) of adsorbent. Cm is the mass of dye that 1 gm of adsorbent can adsorb when the monolayer is complete and b is the isotherm constant for particular adsorbate adsorbent combination.

The *Cm* and *b* values were calculated from the slopes (*1/Cm*) and intercepts (*1/bCm*) of linear plots of *Ce/X* versus *Ce*.

Table 3: n and k values for various dyes on treatment with different adsorbents

Sr.No.	Adsorbent	n & k values	Dyes				
			Acid Red 119	Acid Blue 15	Acid Violet 54	Acid Violet 17	Acid Violet 49
1.	Bagasse charcoal	n	5.13	1.46	5.97	1.16	6.56
		k	0.051	0.791	0.007	0.007	0.009
2.	Brick kiln ash	n	2.45	0.56	5.22	11.33	3.86
		k	0.105	0.0002	0.007	0.007	0.014
3.	Cement kiln ash	n	2.76	2.45	1.88	2.17	2.89
		k	0.09	0.18	0.35	0.25	0.13
4.	Cow dung Charcoal	n	3.28	2.13	4.70	3.49	1.79
		k	0.03	0.16	0.02	0.05	0.42
5.	Groundnut shells charcoal	n	1.67	2.21	2.44	1.55	1.71
		k	0.45	0.13	0.11	0.45	0.26
6.	Pea shells charcoal	n	1.45	3.83	1.27	3.03	1.34
		k	6.85	0.06	14.04	0.13	7.62
7.	Used tea leaves charcoal	n	1.89	1.47	4.93	2.05	5.03
		k	0.25	0.76	0.01	0.005	0.008
8.	Wheat straw Charcoal	n	2.53	0.50	4.98	0.007	3.56
		k	0.11	2.53	0.01	0.001	0.02

Table 4: *Cm* and *b* values for various dyes on treatment with different adsorbents

Sr.No.	Adsorbent	Cm (mol l^{-1} x 10^5) b (1 mol^{-1})	Dyes				
			Acid Red 119	Acid Blue 15	Acid Violet 54	Acid Violet 17	Acid Violet 49
1.	Bagasse charcoal	Cm	0.194	0.159	2.929	5.027	-
		b	8607	16685	14566	40143	-
2.	Brick kiln ash	Cm	10.598	-	2.562	4.983	3.309
		b	3876	-	10568	40729	8238
3.	Cement kiln ash	Cm	3.230	3.070	2.930	6.540	4.230
		b	56984	35390	89134	24305	18778
4.	Cow dung ash	Cm	4.750	3.050	3.090	6.030	2.240
		b	2844	3278	8992	0970	6377
7.	Groundnut shells charcoal	Cm	13.200	9.500	7.800	13.300	8.400
		b	2414	2814	5510	991	1986
8.	Pea shells Charcoal	Cm	5.830	0.810	5.510	1.590	4.140
		b	1243	18643	3048	9570	3362
11.	Used tea leaves charcoal	Cm	10.990	6.736	2.879	4.873	3.084
		b	2975	1009	11773	80258	11894
12.	Wheat straw charcoal	Cm	10.818	-	2.890	4.721	3.703
		b	5303	-	10911	38209	7165

Table 3 and 4 gives the the *k* and *1/n* values in Freundlich equation, *Cm* and *b* values in Langmuir equation. The correlation coefficients in every case were found in between 0.92 -0.98. The adsorbing capacities of different adsorbing material varied very much in removing different dyes. The maximum sorption capacities (Cm) for Bagasse charcoal was 38.32 mg/gm of absorbent for AV 17, brick kiln ash has shown maximum monolayer concentration 80.88 mg/gm of absorbent for AR 119, cow dung charcoal has shown maximum monolayer concentration 50.30/gm mg of absorbent for AV 17, groundnut shell charcoal has shown maximum monolayer concentration 100.57 mg/gm of absorbent for AV 17, pea shells charcoal has shown maximum monolayer concentration 44.48 mg/gm of absorbent for AR 119 and used tea leaves for has shown maximum monolayer concentration 126.53 mg/gm of absorbent for AB 15, wheat straw charcoal has shown maximum monolayer concentration 72.81 mg/gm of absorbent for AR 119. From the results in Table 3, it could be concluded that the sorption isotherms of all the five dyes followed the Langmuir and Freundlich models.

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Hall et al[33], have suggested a dimensionless equilibrium parameter namely R, in order to reveal the essential characteristic of Langmuir isotherm relating R with Langmuir constant b and the initial concentration of the adsorbate solution, C_o i.e. $R = 1/(1+bC_o)$ the value of R were determined in all cases, the values of R were between zero and one which indicate highly favourable adsorption and applicability of Langmuir isotherm also.

R value	Type of isotherm
R > 1	Unfavourable
R = 1	Linear
R = 0 – 1	Favourable
R = 0	Irreversible

Rate constant values of all the systems were also calculated by using the equation i.e. $\ln C_o/C_e = kt$, alongwith $t \frac{1}{2}$ ($t^{\frac{1}{2}} = 0.693/k$) values. The surface residence times were large indicating strong binding to the surface active site indicating a very favourable adsorption phenomenon in all the cases.

Effect of sorbent dose

The effect of sorbent dose on the removal ratios of dyes are shown in Fig. 4 The percentages of dyes sorbed increased as the sorbent dose was increased over the range 1.0 – 10 g/l. The adsorption of dyes increased from 29 to 98.9%, from 33 to 99.5%, 16 to 98.7%, 40 to 99.0% and 25 to 97.7% in AR 119, AB15, AV54, AV49 and AV17, respectively using rick kiln ash (Fig. 4).
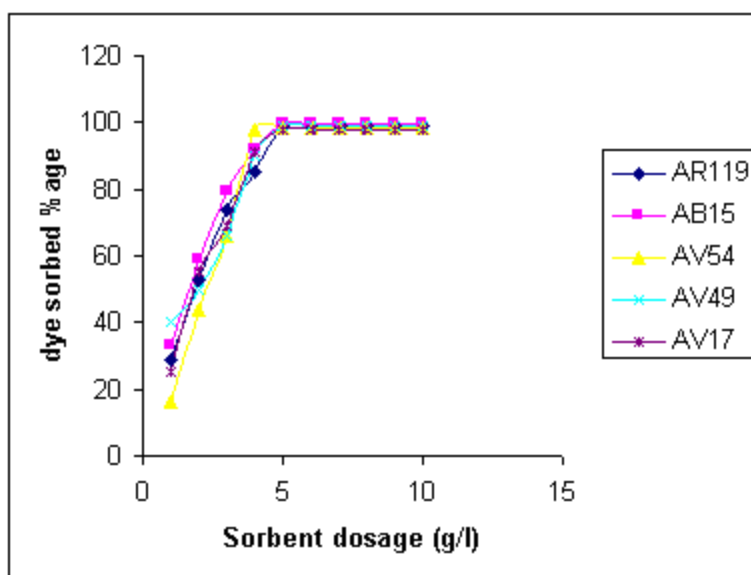

Fig. 4 Effect of sorbent dose on adsorption of acidic dyes using brick kiln ash

Similar increasing trend from minimum 20 to maximum 99.7% was observed with all other adsorbents. Increase in adsorption with the sorbent dose could be attributed to increased surface area and the availability of more sorption sites. This observation is consistent with Langmuir hypothesis of an increasing competition among adsorbent particles for organic substances with increasing number of adsorbent particles per unit volume.[34] In all the dyes, the ratios of dye sorbed had approached maximum values when sorbent dose of 5 g/l was used. So, in other parameter experiments, adsorbent amount of 5.0 g/l was chosen.

Effect of ion strength

The effect of ion strength on adsorption of dyes were tested by the addition of sodium chloride to the solution. The concentration of NaCl used ranged from 0 to 0.5 M. As seen in Fig. 5, (which is for study on brick kiln ash) increasing the ion strength of solution caused decrease in adsorption percentages of dyes. This could be attributed to inhibition for the nearness of dye molecules and sorption sits.
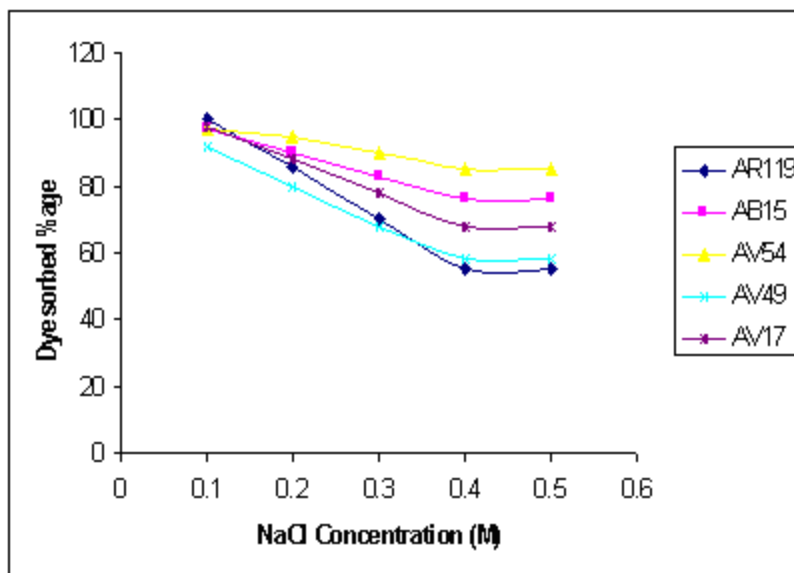
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Fig. 5 Effect of ion strength on adsorption of acidic dyes using brick kiln ash

From the results in Fig. 5, it could be concluded that the smaller the dye molecule weights, the larger the effects of ion strength on dye adsorption. Similar type of interference on the percentage adsorption was observed with other adsorbents also.

Economical Analysis

All the adsorbents used in the present study are of low cost. If the cost analysis of these adsorbents is performed then it can be seen that all these adsorbents are available in large amount in India and that too free of cost. The cement kiln and brick kiln plants are finding it difficult to dispose off their ash, so a large amount of ash can be collected from these plants free of cost. The other adsorbents are also available as waste in the market. The method of manufacturing charcoal from the adsorbents is very simple. A simple closed iron box with small hole can be used, in which the adsorbent material can be filled and then slowly heated on fire in low supply of oxygen. So from economical point of view the use of these adsorbents is very beneficial and effective in removing acidic dyes from the aqueous solutions.

Conclusion

The present study showed that all the materials are promising adsorbents for the removal of acidic dyes from aqueous solutions, since all the raw material were easily available in large quantity and the treatment methods of biomaterial and other adsorbents seemed to be economical. At initial basic pH all the five acidic dyes studied could be removed effectively. The isothermal data of adsorption followed both Langmuir and Freundlich models. The adsorption capacities of all the adsorbents were found to be so good that their use for removal of acidic dyes could be effective and economical.

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