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SORPTION BEHAVIOR OF CATIONIC AND ANIONIC DYES FROM AQUEOUS SOLUTION ON DIFFERENT TYPES OF ACTIVATED CARBONS

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ABSTRACT

The effect of dye molecular charges on their adsorption from solution was investigated by using different types of activated carbon adsorbents. Two types of model systems were used representing cationic and anionic dyes. Screening investigations using single point tests were used throughout the study. Cationic dyes, of which Methylene Blue is an example, showed a higher adsorption tendency towards activated carbon over anionic dyes represented by an azo-type reactive compound. Of the number of activated carbons tested, only one of the adsorbents showed an exception to this behavior, and a good relation was observed between Methylene Blue capacity and activated carbon performance. The high capacity of cationic dyes in comparison to anionic dyes was also evident in the results obtained by a preliminary kinetic study carried out on the selected systems. Surface net charge of activated carbon and

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the nature of attractions between the molecules were suggested to be one of the reasons attributed for this behavior.

Key Words: Adsorption; Activated carbons; Cationic and anionic dyes.

INTRODUCTION

Water pollutants are diverse and varied in nature. They basically include organic and inorganic substances. Generally, inorganic pollutants are represented by heavy metals, where as organic pollutants extend from small molecules such as chloroform to macrocylic or hetreocyclic compounds such as dyes. Dyes are cyclic organic compounds with relatively high molecular mass. They are classified in a number of ways according to their use or the properties of their chemical structures. From chemical standpoint, dyes are considered to be either cationic (carrying a positive charge) or anionic (carrying a negative charge) in nature (1). Though not particularly toxic, dyes have an adverse aesthetic effect because they are visible pollutants. In some cases less than 1 ppm of dye concentration produces obvious water coloration. Adsorption of dyes from wastewater is considered to be an effective and economic process for cleaning coloured water. A number of references outlined the applicability of adsorbents for removing dyes from effluent. These adsorbents may be of natural (2) or synthetic origin (3). For example, activated carbon, peat, lignite, clays, and coal are some of the most widely used natural adsorbents. The high surface area, porosity and reactive surface chemistry of these materials are the main reasons that make them effective for removing dyes from solution. Furthermore, it is suggested that the adsorbate charge is likely to have a controlled effect on the adsorption process (4). According to some studies, it was suggested that adsorbents may have high tendency toward positively charged dyes (cationic dyes) over negatively charged ones (anionic dyes) (5–11).

In this work, adsorption capacities of six activated carbons were studied in relation to the removal of two types of dyes from aqueous solutions. Two anionic dyes (Remazol Reactive Black and Remazol Reactive Red) and one cationic dye (Methylene Blue) were considered. The effect of dye molecular charge on carbon capacities is discussed and the adsorption capacities are compared.

EXPERIMENTAL

Carbon Adsorbents

Activated carbon materials used in this study were: Filtrasorb 400(FS-400), EA207, C207, Centaur, and Lignite Coke carbon. Table 1 contain some of the most

Table 1. Surface Properties for Studied Carbons

Carbon[a]	Origin	Surface Area (m^2/g)[b]	Surface Area (m^2/g)[c]	Bulk Density (g/cm^3)
FS-400	Bituminous coal	1020	1200	0.425
Centaur	Bituminous coal	767	710	—
EA207	Coal	900	986	0.50
C207	Coconut shell	900	1072	0.49
Chilean Lignite	Lignite	4	80	—
Coke Carbon	Lignite	288	170	0.45

[a] FS-400 and Centaur carbons supplied from chemviron carbon, UK. C207 and EA207 supplied by Sutcliffe Spekman Carbons Lt, UK, and Coke carbon supplied from Rheinbraun Brennstoff GMBH, Germany.
[b] Calculated according to the standard B.E.T Method.
[c] Calculated from Methylene adsorption data (26).

important characteristic values supplied by the manufacturers. All carbons were selected due to their high adsorption ability in solution for a number of organic and inorganic contaminants. The samples were sieved using standard British sieves (Endecotts Ltd, London, England) to obtain desired particle size distribution. Adsorptive capacities for the carbons were evaluated for the size range of 600–710 μm. To eliminate the effect of long storage times (i.e., to minimize the oxidation of carbon surfaces with air), all samples were kept in dry sealed glass bottles. All carbons were used as received from the manufacturers without any further treatment.

Adsorbate

Two anionic dyes were used in this study: namely, Remazol Black and Remazol Red supplied from Bayer, Frankfurt, Germany. Reactive Dyes, of which these are an example, represent about 20–30% of the dyes used worldwide (12). Their usage has increased by approximately 15% per year since 1980 (12). They are characterized by nitrogen to nitrogen double bond (N=N) azo bonds. The color of the azo dyes is due to this azo bond and associated chromophores. On the other hand, Methylene blue was used as an example of cationic dyes.

Equilibrium Isotherm Experiments

1000-ppm dye stock solutions were prepared from their corresponding concentrated solutions supplied by the manufacturers. Methylene Blue was prepared from analytical reagent grade standard solutions (BDH, England). All dye solutions were kept in cool darkened conditions for further use. Lambda maximum and liner calibration graphs were established for each dye by using a spectrophotometer (Unicam model). Lambda$_{max}$ values of 520, 597, and 630 nm were obtained for Reactive Red, Reactive Black, and Methylene Blue, respectively. The maximum capacities of dyes on various activated carbons were determined by carrying out equilibrium isotherm tests. In these tests, an accurately weighed ±0.01 g of each carbon was mixed with a fixed volume of dye solution in sealed glass bottles. The ratio of carbon mass to dye solution was kept constant for all experiments. Carbon particle diameter of 600–710 μm was chosen for all sorbents. The bottles were agitated using a mechanical shaker at room temperature. A constant shaking speed was maintained throughout the experiments.

In order to determine when adsorption equilibrium had been reached, liquid phase samples were taken at intervals between one and five weeks. The samples were removed and filtered through 0.45-μm Millipore cellulose nitrate filters and subsequently analyzed for postadsorption concentrations using Unicam spectrophotometer at maximum wavelength for each dye. The equilibrium concentrations were determined by converting the optical densities (densities above 1.00 were diluted) to concentration using the predetermined linear calibration graphs. All isotherms were done in duplicate, and results are reported as an average of these. Blanks were also included. These procedures gave reproducible results and were found to agree within 4%.

To study the rate of dye uptake, a fixed amount of carbon was added to a 2000-mL plastic vessel. This adsorption solution was stirred with a six-blade stirring rod attached to a mixer (IKA Labortechnik model, Germany). 4-mL solution samples were withdrawn from the adsorption beaker at specific times. Removed samples were diluted and analyzed by using the spectrophotometer. As the total removed volumes were relatively small (60–65 mL) to initial volume (1700 mL), no correction was required.

Surface Acidity and Basicity

Both the acidity and basicity of carbons were determined using titration methods (13). The carbon samples were sieved through a 100-μm sieve, washed several times with distilled water, and then dried at 100°C for 24 h prior to titration. All chemical reagents used in the titration were of analytical grade and supplied by BDH (Poole, UK).

Surface Acidity

50 g of carbon was prepared as described above and put into contact with 50 cm^3 of 0.01 *N* NaOH. The samples were agitated at 25°C using an automatic shaker. The mixture was filtered and then back titrated with 0.01 *N* HCl. The

concentration of acid substitute groups on the surface was determined and expressed in mequi or mmol/g carbon.

Surface Basicity

The total surface basicity of a carbon sample was quantified by mixing 0.5 g of carbon with 50 cm^3 of 0.1 *N* standard solution of HCl. The suspension was agitated for 24 h at 25°C. An aliquot of the supernatant was filtered and back titrated with 0.1 *N* NaOH solution.

pH of Carbon Solution

The pH value of carbon samples was measured after 3 h stirring of a 10% (w/v) mixture of activated carbon and distilled water.

RESULTS AND DISCUSSION

The maximum adsorption capacity of a carbon to adsorb a dye was evaluated from the well-known Langmuir equation:

$$q_e = (X_m C_e a_l)/(1 + a_l C_e) \tag{1}$$

where q_e is the carbon surface concentration (mg/g), X_m is maximum surface concentration (mg/g), and b is the isotherm constant (L/mg).

The amount of dye adsorbed after time t (q_t mg/g) was calculated by using the mass balance formula:

$$q_t = (C_o - C_t)V/m \tag{2}$$

Where V, m, C_o, and C_t are the volume of solution (mL), carbon mass (g), and the initial and concentration at time t, respectively.

Adsorption capacities of each sorbent were determined from batch isotherms that have reached equilibrium. Equilibrium was reached when each sorbent ceased to show a significant increase in solid phase uptake. This was obtained by plotting solid phase uptake [q_e(mg/g)] against the residual phase concentration [C_e(mg/dm^3)]. Once equilibrium capacities were determined, it was possible to compare the uptake performance of the sorbents under investigation. Plots of complete adsorption isotherm curves for Methylene Blue and Reactive Black on FS-400 and Centaur carbons are shown in Figures 1 and 2.

The maximum capacities (mg dye/g carbon) of all carbons to uptake dyes are shown in Table 2. As this study is mainly concerned with comparing cationic and anionic dye capacities on different activated carbons, single-point tests were

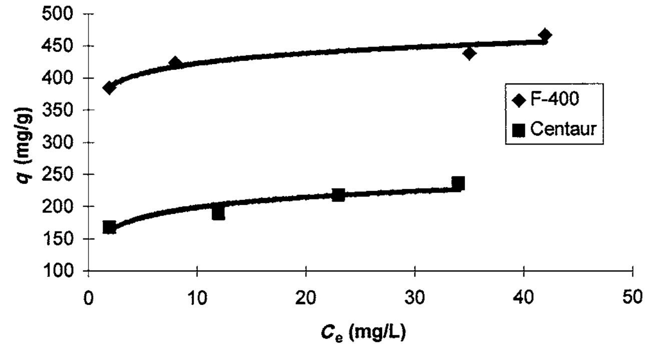

Figure 1. Adsorption isotherms of Methylene Blue (cationic dye) on FS-400 and Centaur; temperature: 21°C; agitation time: 3 weeks.

used to give approximate values of maximum capacities of carbon. The maximum saturation values of Methylene Blue calculated according to Equation (1) were 454 and 232 mg/g for FS-400 and Centaur carbon, respectively. The Methylene Blue value reported here is higher than the value of 281 mg/g reported for FS-400 by Bacaoui et al. (14). On the other hand, single-point tests gave values of 533 and 314 mg/g for the same dyes and carbon combination (Table 2). It is worth mentioning that the maximum capacities obtained from complete isotherms were more accurate than single-point tests; however, Miguel and coworkers (15) used the single-point test for comparing the adsorbing capacity of carbons toward some reactive dyes. A keen inspection through Table 2 shows a preferred adsorption of cationic dye over anionic ones. FS-400, EA207, C207, Centaur, and Chilean lignite showed a high tendency to adsorb cationic dye, in this case Methylene Blue, in comparison to anionic dyes represented by Reactive Black and Reactive Red. Of

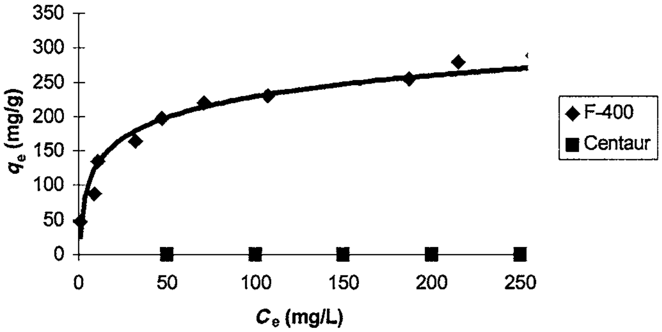

Figure 2. Adsorption isotherms of Reactive Black (anionic dye) on FS-400 and Centaur Carbons; temperature: 21°C.

Table 2. Maximum Capacities of Carbons (mg/g)[a]

Carbon	Methylene Blue (mg/g)	Reactive Black (mg/g)	Reactive Red (mg/g)
FS-400	533	325	279
EA207	436	94	68
C207	474	108	105
Centaur	314	0	0
Chilean Lignite	35	0	0
Coke Carbon	74	124	82

[a] Average of two experiments with standard deviation of ±4%.

the carbons used in this study, Coke carbon showed an exception to this behavior. Although the above trends may describe a large number of carbon-dye systems, few studies reported a high tendency of certain adsorbents toward anionic dyes (15,16). The results also showed that although Centaur carbon had a good capacity to adsorb Methylene Blue (more than Coke carbon), it showed no capacity to adsorb anionic dyes. Similarly, Chilean Lignite carbon failed to adsorb anionic dyes and has, at the same time, the lowest capacity among the carbons under investigation for the removal of Methylene Blue. Methylene Blue adsorption capacity can be adopted as a criterion to test the total performance of the carbon adsorbents in solution (17). Based on this criterion, FS-400 showed the highest capacity toward all the dyes used in this investigation, and the experimental results recorded the highest value for Methylene Blue. This was not surprising, as FS-400 activated carbon is a well-known commercial adsorbent due to its high surface area and well-developed structure. In fact, adsorption of dyes from solution onto activated carbon is controlled by many factors. Some of these factors depend on adsorbent characteristics such as the magnitude of surface area and porosity, and others are related to the chemical content of the carbon surface and the interaction of the sorbate to the surface. Within literature, mixed results have been reported relating activated carbon capacities and performance for various types of dyes. Furthermore, a number of proposed mechanisms were reported (2,7,10,18,19) to explain this behavior. These mechanisms are based, mainly, on the electrostatic attraction between the dye charge and adsorbent surface charge during the sorption process. El-Geundi (2) studied the adsorption behavior of cationic and anionic dyes on maize cob. The author attributed the preferred adsorption of cationic dyes to electrostatic attraction between these dyes and the negatively charged surface. Sethuraman et al. (7) observed a high kinetic capacity of two minerals for adsorbing cationic dye in comparison with anionic dye. The preferred capacity for these minerals toward cationic dyes was attributed, according to the authors, to the nature of exchange sites on the their surfaces. Low et al. (18) have generalized that most biological sorbents (maize cob, bagasse pith, and peat wood) are more efficient in attracting cationic than anionic dyes. This is due mainly to colombic attraction between the negative surface of the sorbents and the positively charged dyes.

As mention earlier, few sorbents in addition to their good capacity for cationic dyes, manifested higher capacity for anionic dyes (i.e., have amphoteric behavior). Lee et al. (19) studied the sorption capacity of Bleaching Earth, a waste material from palm oil, by using six dyes (two cationic and four anionic). The comparative study showed that Bleaching Earth has a better capacity toward cationic compared to anionic dyes. The authors attributed this behavior to the negatively charged surface of Bleaching Earth, which has higher affinity for cationic dyes. In a similar type study, Mckay et al. (6) screened the removing behavior of eight dyes (two cationic and six anionic) by using six different natural adsorbents. It was inferred that, with exception of one adsorbent, all adsorbents showed high capacity for cationic dyes. In a different study, McKay et al. (20) have reported similar observations by using Fuller Earth. Juang et al. (10) studied the effect of dye charge on adsorption onto activated carbon. The main finding of the study was that a higher capacity and faster adsorption kinetics were obtained for cationic dyes. The authors attributed this preference to electrostatic attraction between electron-acceptor groups (i.e., negative surface charge) on carbon surface and the positive charges on dyes. In fact, it has been reported that activated carbon adsorbents, generally, carried a net negative surface charge in solution, and it is believed to be of significance for adsorption process (21). Accordingly, it seems that the acquired negative charged formed on carbons has a significant roll for preferred cationic dye adsorption behavior.

The high reactivity of activated carbon toward organic pollutants may also rise from the complexity of chemical surface groups compared to other surfaces. The surface of inorganic solids, such as metal oxides, for example, consists mainly of oxygen atoms and hydroxyl groups. It is primarily the hydroxyl groups that determines the chemistry (acid-base character) and the reactivity of these surfaces (13). In contrast, the activated carbon surface can contain not only one but at least five markedly different types of surface groups such as carboxylic, lactonic, phenolic, carbonyle, and etheric groups (22). This diversity of surface groups make the chemistry (acid-base character) much more versatile than that of other adsorbents (22).

Even though the basic properties of many solid carbon surfaces in solution have long been recognized, the nature of the sites that are responsible for such interaction is not fully understood. Carbon surfaces acquire a basic character on high temperature (>973 K) heat treatment (23). Most carbons contain a small amount of inorganic impurity that can influence their acid/base response in solution (24). Nevertheless, the pure organic portions alone have been shown to manifest their surface basicity in several ways (13), such as a pH value of above 7, a positive external surface charge, and acidic uptakes.

Table 3. Surfaces Acidity, Basicity, and the pH of Carbon Solutions

Carbon	Surface Acidity (mequi/g)	Surface Basicity (mequi/g)	pH (10%)
Centaur	0.33	1.12	6.11
Chilean	0.87	1.24	5.95
EA207	0.14	1.25	8.00
C207	0.20	1.40	8.77
FS-400	0.25	1.11	8.30

In an effort to gain a better insight into the effect of surface chemistry on the selectivity of the carbons towards anionic and cationic dyes, the surface acidity and basicity for the carbons were estimated using the experimental procedure described earlier and results are given in Table 3. As shown in Table 3, EA207, C207, and FS-400 produced an alkaline suspension and showed higher surface basicity. On the other hand, Centaur and Chilean lignite carbon seem to produce an acidic suspension in pure water and have higher surface acidity compare to other three carbons. The experimental results for FS-400 (FS-400 considered because it showed the highest adsorption capacity of all) showed a pH value of 8.3, which is consistent with observations reported by Huang and Wu (25). FS-400 also showed an amphoteric property by adsorbing both H^+ and OH^-. FS-400 was found to have a surface acidity of 0.25 mmol/g and 1.11 mmol/g carbon as surface basicity. Because FS-400 has a high surface basicity it is expected to show a high adsorption affinity for cationic dyes. The results were consistent with findings reported by Gyzel (26). Both C207 and EA207 showed the same aqueous chemistry and the same adsorption behavior exhibited by FS-400. As can be observed from Table 3,

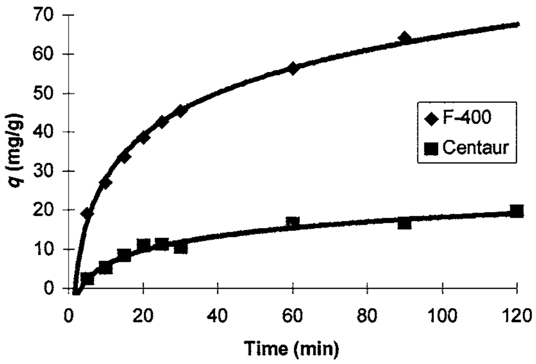

Figure 3. Kinetic capacity of FS-400 and centaur carbons for adsorbing Methylene Blue.

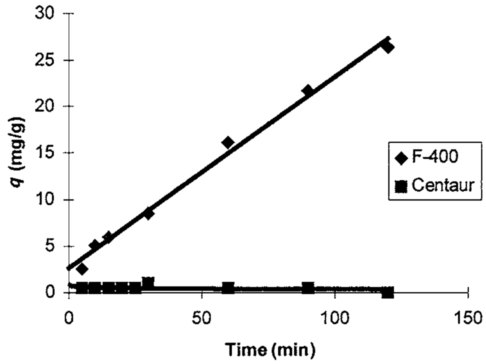

Figure 4. Kinetic capacity of F-400 and Centaur Carbons for adsorbing Reactive Black.

pH of carbon solutions may have an effect on carbon performance; alkaline suspension carbons have better performance compared to acidic suspension carbons.

The results of kinetic study of Methylene Blue and Reactive Black on carbon surfaces are shown in Figures 3 and 4. Under similar experimental conditions and after 90 min of agitation, FS-400 adsorbed 64 mg Methylene Blue/g carbon, whereas it adsorbed a lesser amount of anionic reactive black (22 mg/g). On the other hand, Centaur carbon adsorbed 17 mg/g of Methylene Blue and failed to adsorb Reactive Black. This result agrees with the test point result. Unlike Figure 3, Figure 4 shows a slower kinetic behavior of anionic dye adsorption. Actually, a similar observation, by using activated carbon adsorbent, was reported by Juang et al. (10).

CONCLUSIONS

The results of this study showed that cationic dyes (dyes carrying positive charge) had, in general, a higher tendency to adsorb on different adsorbent surfaces in comparison to anionic dyes. Five out of six carbons (with different origins) have a preferred capacity of cationic over anionic dyes. Some of these adsorbents also showed some ability to adsorb cationic dyes in addition to their high capacity for anionic dyes (i.e, showed an amphoteric behavior). Methylene Blue capacity can be considered as a criterion to describe carbon performance in solution. FS-400 was found to have the highest Methylene Blue value that reflected the high capacity to adsorb anionic dyes in general. The higher surface basicity of carbons was found to have a significant role in attracting cationic dyes from solution. It can be concluded from both equilibrium and kinetic results that cationic dyes adsorbed in large extent and in the same time have fast adsorption kinetics.

The net surface charge of adsorbents acquired in solution played an important role in explaining the differences in adsorptive capacities of the carbons under investigation.

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