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Effect of Surface Area, Micropores, Secondary Micropores, and Mesopores Volumes of Activated Carbons on Reactive Dyes Adsorption from Solution

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ABSTRACT

The ability of six activated carbons to remove the problematic reactive dyes from textile solution was correlated to their physical characteristics. Specific surface area, micro-, meso-, and total volume were established using the Braunnaur, Emmet, and Teller (BET) and Dubinin–Radushkevich (D–R) equations. According to the N_2 adsorption method, five of the carbons exhibited type I adsorption characteristics with one carbon showing a type III isotherm. Activated carbon Filtrasorb 400

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outperformed the carbons under investigation by showing the presence of advanced primary and secondary micropores. A poor linear correlation between carbon surface area and performance was found. Adsorption properties of carbons were better related to their pore volumes. It was found that secondary micropore volume of carbons ($0.8\,\mathrm{nm} < D_{\mathrm{secondary\ micropore}} < 2\,\mathrm{nm}$) has an important role in reactive dyes adsorption.

Key Words: Activated carbon; Reactive dyes; Meso- and micropores; N_2 adsorption isotherms.

INTRODUCTION

Color removal of water-soluble reactive dyes is problematic: current methods rely on activated sludge systems and are not adequate, neither on site nor after dilution with domestic wastewater at sewage works. Despite extensive research into color removal from textile wastewater, problems of costly plant requirements or operating expenses; lack of effective color reduction, particularly for sulphonated azo dyes, and sensitivity to variable wastewater inputs are experienced in the application of the chemical and physical treatment methods in reactive dye wastewater treatment.[1]

Two properties of reactive dyes limit the rate of color removal: the highly soluble hydrolyzed azo structure and low biodegradability. Due to the chemical nature, molecular size, and structure, activated carbon appears to offer some prospects for effluent treatment. A number of investigations were conducted to evaluate adsorption of reactive dyes onto a range of natural and synthetic, organic and inorganic sorbents.[1] In these studies, no attempts were made to correlate the effect of the physical characteristics of the adsorbents to their adsorption tendency toward this group of dyes.

Within published research, the adsorption capacity of a certain material is generally related to the nature of the adsorbents surface functional groups. The surface area is also known to play an important role in the adsorption process.[2] Generally, carbon surface characteristics can be related to adsorption capacity from solutions. These characteristics include both chemical and physical properties. Chemically, carbon can be imagined as plates of carbon atoms arranged hexagonally in pasal planes.[3] Many chemical groups are assumed to be attached to that complicated geometry. Reactivity of carbon in solution can be attributed to strength and concentration of these chemical groups. Physically, on the other hand, carbon performance can be attributed to the presence of high surface area, channels, and porosity system. Such a structure can accommodate, or attract, adsorbate species onto the surface by different forces.[3]

Due to their commercial availability, ease of preparation from many natural resources, and high surface reactivity, activated carbons were considered in removing reactive dyes from wastewater. In this work, carbon texture properties (surface area and porosity) are correlated with their effectiveness for removing reactive dyes from solution. The principal reason for investigating a variety of carbons, which are prepared from different materials, was to test the effect of chemical and physical intrinsic properties on reactive dye adsorption. Once the total surface area and porosity are estimated, then carbon performances could be compared. It is worth mentioning that in addition to physical characteristics, chemical characteristics play a considerable role during adsorption of reactive dyes from solution. Therefore, to have a fair comparison between carbon performances, both physical and chemical characteristics should be related to carbon performance. Chemical characterization of these carbons is discussed elsewhere.[4]

Characterization of Carbon Texture Properties

Physical characterization of activated carbons and solid surfaces was first introduced by Brunauer and colleagues.[5] Nitrogen molecules were the first chemical substances used to identify the inner structure of carbons and the pore size distribution. This theory is mainly based on the elementary assumption of Langmuir's gas-adsorption theory.[6] Based on the Langmuir equation, the Braunnaur, Emmett, and Teller (BET) equation was derived:[6]

$$\frac{P}{\nu(P_0 - P)} = \frac{1}{\nu_m c} + \frac{c-1}{\nu_m c}\frac{P}{P_0} \tag{1}$$

where ν is the volume of the gas adsorbed (usually nitrogen) at pressure P; P_0 is the standard vapor pressure of the liquid at the temperature of experiment; ν_m is the volume equivalent to an adsorbed monolayer; and c is the BET constant, which is related to molar energy of adsorption in the first monolayer.

Adsorbent surface area can be calculated by using the ν_m value, which is easily determined from the linearization of above the equation. Surface area is a key parameter used to characterize porous solids like activated carbon. Surface area (S_{BET}) can be estimated from Eq. (2):[2]

$$S_{BET}(m^2 g^{-1}) = \frac{X_m}{M} \cdot N \cdot A_m \cdot 10^{-20} \tag{2}$$

where X_m is the amount of adsorbate (g) forming one molecular layer per gram solid; M is molecular weight of adsorbate; N is Avogadro's constant; and A_m (in Å^2) is the molecular cross-sectional area of adsorbate.

In spite of its limitations, the BET method is still the most widely used for surface characterization. In addition to its usefulness for surface area determination, gas adsorption data can be further used to estimate the microporosity, which is an important property in adsorbent characterization. Before monolayer completion, i.e., at the early stage of gas adsorption, total adsorbent microporosity (cm^3g^{-1}) can be determined. Dubinin and Radushkevich proposed a theory of adsorption equilibrium in microporous materials.[7] The amount of adsorbate which filled a sorbent micropore can be calculated from the Dubinin–Radushkevich equation:[7,8]

$$\frac{\nu}{\nu_0} = \exp\left[-B\left(\frac{T}{\beta}\right)^2 \log_{10}^2 \frac{P}{P_0}\right] \tag{3}$$

where ν is adsorbate (N_2 in this work) volume adsorbed at P/P_0 and ν_0, the total micropore volume. B and β are equation constants. Upon linearization, Eq. (3) becomes:

$$\log \nu = \log \nu_0 - D_{R-D} \log^2\left(\frac{P_0}{P}\right) \tag{4}$$

A plot of $\log \nu$ against $\log^2(P_0/P^2)$ should obtain a straight line with micropore volume as an intercept. In fact, higher micropore volume is an indication of microporosity and higher areas for adsorption.[6,8]

MATERIALS AND METHODS

The commercial activated carbon materials used in this research were Filtrasorb 400 (FS-400), EA207, C207, Centaur, lignite coke carbon, and Chilean lignite carbon. All carbons were selected due to their high adsorption in solution.

FS-400 and Centaur carbons were supplied by Chemviron Carbon, UK, and have a bituminous coal origin. C207, a material prepared from coconut shell, and EA207, a coal, were obtained from Sutcliffe Spekman Carbons Ltd., UK. Coke carbon supplied by Rheinbraun Brennstoff GMBH, Germany, was prepared from a lignite origin. Chilean lignite is locally produced carbonaceous material.

Three reactive dyes were studied, namely, Remazol Reactive Yellow, Remazol Reactive Black, and Remazol Reactive Red. Chemical structure of remazol reactive black is depicted in Fig. 1. The working solutions for the three dyes were prepared from standard concentrated solutions (33 to 55%) as received from the manufacturer (Bayer, Frankfurt, Germany).

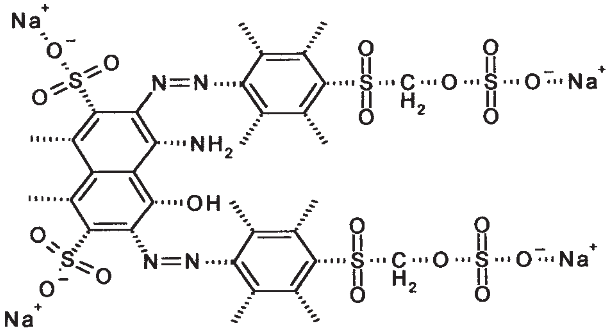

Figure 1. Chemical structure of Remazol Reactive Black.

Equilibrium Isotherm Determination

A concentration variation equilibrium method was carried out at 20 ± 2°C by shaking 0.050 ± 0.001 g of adsorbent in 50 cm^3 of an aqueous solution of preselected dye concentration range in glass bottles in a temperature-controlled shaker (L.H. Engineering Co. Ltd., England). Dye concentrations were varied from 0 to 1000 mg dm^{-3}. After 3 weeks, samples were removed and filtered through 0.45-μm Milipore cellulose nitrate filters and subsequently analyzed for postadsorption concentration using a Unicam spectrophotometer at maximum wavelength for each dye. The maximum wavelengths were 410, 520, and 597 nm for yellow, red, and black, respectively. The equilibrium concentrations were determined by converting the optical densities to concentration using a predetermined linear calibration graph.

Nitrogen Adsorption Studies

Total surface area and micropore and mesopore volumes were determined using the N_2 gas adsorption method (Sorptomatic 1900 instrument). Prior to experimental measurement, carbons were out-gassed at 70°C under low vacuum pressure (10^{-5} to 10^{-6} torr). Adsorption isotherms, performed under a constant nitrogen flow rate to an adsorption cut-off p/p_0, required 10 hours to complete. Surface area and total micropore volumes were calculated using the BET equation and Dubinin–Radushkevich (D–R) equation, respectively. Mesopore volumes were calculated by subtracting the micropore volume (obtained from D–R equation) from the total volume of nitrogen adsorbed at p/p_0.

RESULTS AND DISCUSSION

Adsorption Capacities and Performance in Solution

To study the effect of the physical properties of the carbons on their performance in solution, the uptake capacities of each carbon was essentially determined. Adsorption capacity of each carbon was determined from batch isotherms that have reached equilibrium.

The adsorption isotherm curves showed a Langmurian shape, i.e., initial slope at the beginning and a plateau covering most experimental points. An example of the adsorption isotherms of reactive yellow is given in Fig. 2. Maximum adsorption capacities, as calculated from Langmuir isotherm, are summarized in Table 1. The activated carbon FS-400 showed an uptake capacity of 714 $\text{mg} \cdot \text{g}^{-1}$ for Reactive Yellow, followed by 278 $\text{mg} \cdot \text{g}^{-1}$ for Reactive Black, and the lowest recorded was a value of 213 $\text{mg} \cdot \text{g}^{-1}$ for Reactive Red. In addition to the high capacity of FS-400 for adsorption, it was found that C207 and EA207 were also effective carbons toward all studied dyes. On the other hand, Chilean lignite showed ineffectiveness toward the three reactive dyes studied, which is reflected in the modest adsorption capacity values obtained. Adsorption isotherm results were consistent with some reported data.[1,8,9] This was not surprising since FS-400 activated carbon is a well-known commercial adsorbent due to its high surface area and the well-developed structure. In fact, adsorption of dyes from solution onto activated carbon is controlled by many factors. Factors that depend on

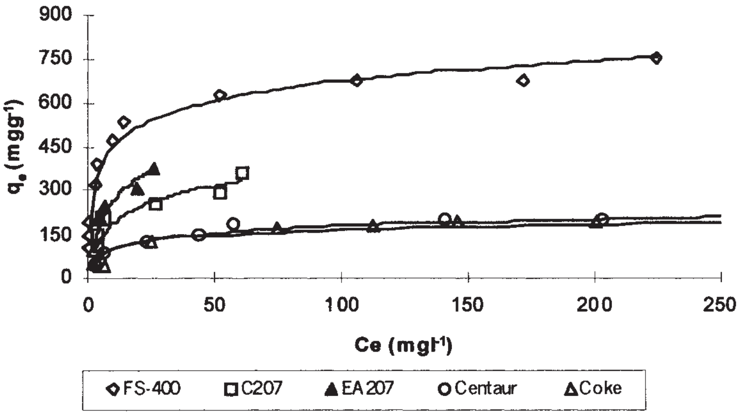

Figure 2. Experimental adsorption isotherms of reactive yellow on different types of activated carbon.

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Table 1. Maximum adsorption capacities of activated carbons.

Carbon	Reactive yellow ($mg\,g^{-1}$)	Reactive black ($mg\,g^{-1}$)	Reactive red ($mg\,g^{-1}$)
FS-400	714	278	213
C207	416	109	123
EA207	384	96	72
Coke carbon	190	130	80
Centaur	200	0	0

adsorbent characteristics include the magnitude of surface area and porosity. Other factors related to the chemical content of the carbon surface and the interaction of the sorbate to the surface are important. Within published research, mixed results have been reported relating activated carbon capacities and performance for various types of reactive dyes.[4,9,10] Under the same experimental conditions, the sorption efficiency of carbons under investigation decreased in the order as follows:

FS-400	R. Yellow > R. Black > R. Red
C207	R. Yellow > R. Red > R. Black
EA207	R. Yellow > R. Black > R. Red
Coke carbon	R. Yellow > R. Black > R. Red
Centaur	R. Yellow > R. Black, R. Red
Chilean carbon	No significant adsorption

As observed, a consistent trend was exhibited by all carbons, that is, Reactive Yellow was adsorbed more than R. Black, while R. Red was the most difficult to adsorb, with the exception of C207 carbon, which showed slightly higher capacity for R. Red over R. Black. This clearly indicates the selective nature of the adsorbents towards Reactive Yellow. Of the corbons assessed, coke carbon was least effective with, uptake values of 80, 130, and 190 $mg\,g^{-1}$ of reactive red, black, and yellow, respectively. Carbon EA207 was next, with uptake values of 72, 96, and 384 $mg\,g^{-1}$ of reactive red, black, and yellow, respectively. Although Centaur carbon showed slightly higher affinity to reactive yellow, with an uptake value of 200 $mg\,g^{-1}$, it failed to remove any traces of both red and black. The adsorbent C207, with an uptake capacity of 416, 109, and 123 $mg\,g^{-1}$, for reactive yellow, black, and red, respectively, proved a potentially good carbon adsorbent for removing reactive dyes from the solution. It is believed that the surface charge of the carbon may play an important role in the ability to favorably adsorb a certain class of dyes. However, the trends and capacities of the activated carbons obtained in this study could not be explained entirely on the basis of the chemical properties of the activated carbon surface.[10] Therefore, characterization of the activated carbons was carried out in an effort to relate the capacities and performance in solution to the physical and textural properties of these adsorbents.

Relation to Surface Area

The total surface area (or BET surface area $m^2\,g^{-1}$) is one of the most widely known parameters associated with activated carbons.[11] It is often assumed that a material with a larger total surface area has more pronounced adsorptive properties, and will therefore perform better. However, the value of this concept is limited. In many applications, only part of the total surface area is accessible for the molecules to be adsorbed. Most of the total surface area is found in the micropores, where a typical carbon contains $1000\,m^2\,g^{-1}$ in micropores, 10 to $100\,m^2\,g^{-1}$ mesopores, and $2\,m^2\,g^{-1}$ macropores.[12] In addition, the magnitude of the surface area does not reflect the chemical strength of the carbon.[13] Most liquid phase applications involve the adsorption of high molecular weight materials, e.g., colored bodies or humic substances. Most of these are excluded from a large part of the micropore system. Here, a carbon with a high mesoporosity is required, and a high total surface area is of no use.[14] Ideally, the carbon should have a large number of pores that are just slightly larger than the size of the molecules to be adsorbed. Smaller pores are not accessible, and much of the larger pores provide relatively little surface area per unit volume. In practical liquid phase applications, materials with molecular masses roughly between 300 and 100,000 are encountered, corresponding with optimal pore radii between 0.5 and 4 nm.[6,8] As can be noted in Fig. 1, reactive dyes are large molecules and of high molar masses (about 1000 g/mol). For effective adsorption for reactive black, carbons of pore radius higher than 0.86 nm is required.

The surface area of carbon samples S_{BET}, was obtained by applying the BET equation to the N_2 adsorption isotherms and the values obtained are reported in Table 2. As indicated by these results, most carbons have a high surface area (S_{BET}), with the lowest value reported for $4\,m^2\,g^{-1}$. Again, this is reflected clearly by its inability to remove reactive dyes from solution.

The degree of correlation between carbon surface areas and uptake capacities of three reactive dyes is shown in Table 3. As shown, a poor linear correlation between surface area and carbon performances was obtained. It can be suggested that physical adsorption (nonelectrostatic attraction) was highly involved with reactive yellow since a slightly better correlation between surface area and capacity was obtained, while electrostatic adsorption (chemisorption) may be involved in the adsorption of reactive black and red. Since the surface area of the adsorbents fail to

Table 2. Surface area of investigated carbons.

Carbon	Surface area $(m^2 \cdot g^{-1})^a$	Surface area $(m^2 \cdot g^{-1})^b$
FS-400	1100	1020
Centaur	—	767
EA207	950	900
C207	1050	900
Chilean	—	4
Coke carbon	300	288

[a] Manufacturers data based on the BET method.
[b] Estimated data based on the BET method (this work).

explain the uptake capacities of the various reactive dye–carbon systems, attention is given to the inner pore structure of these carbons. Hence, carbon capacities for reactive dyes were correlated to their mesopore surface area since mesopore area is more predominant for dye adsorption.[14]

Carbon Pore Volume Characterization

Porosity can be predicted from N_2 adsorption isotherms.[6] The results obtained from N_2 adsorption isotherm experiments are shown in Figs. 3 and 4. Apart from Chilean lignite, which had a type III isotherm, all carbons exhibited type I isotherm. Type I, which represents reversible isotherms, are characteristics of microporous solids; i.e., the five activated carbons

Table 3. Correlation coefficient, r^2, of adsorption capacity of reactive dyes on activated carbons with surface area, micropore, mesopore, and secondary micropore volumes.

Dye	r^2 Obtained for surface area	r^2 Obtained for micropore volume	r^2 Obtained for mesopore volume	r^2 Obtained for secondary micropore volume
R. Yellow	0.70	0.60	0.56	0.90
R. Black	0.27	0.24	0.52	0.60
R. Red	0.38	0.32	0.49	0.60

Note: r^2 values were calculated by correlating adsorption capacities of dyes on activated carbons with their corresponding surface area, micropore, mesopore, and secondary micropore volumes.

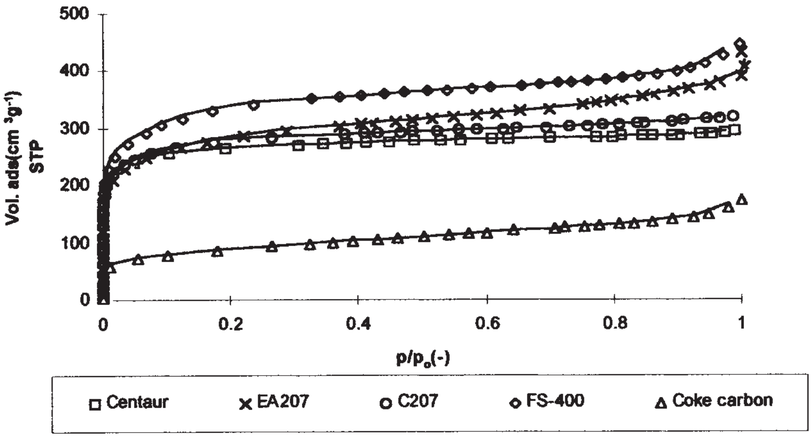

Figure 3. The N_2 (77 K) gas adsorption isotherms for carbons.

have mainly microporous structure.[8] In this type, N_2 uptake reaches a limiting value as p/p_0 tends to 1, indicating that uptake is dependent on micropore volume accessibility rather than surface area. On the other hand, type III isotherms are uncommon and are usually associated with water vapor adsorption interactions.[11] Generally in this type, the adsorbent–adsorbate interactions are weak as compared with the adsorbate–adsorbate forces.[11]

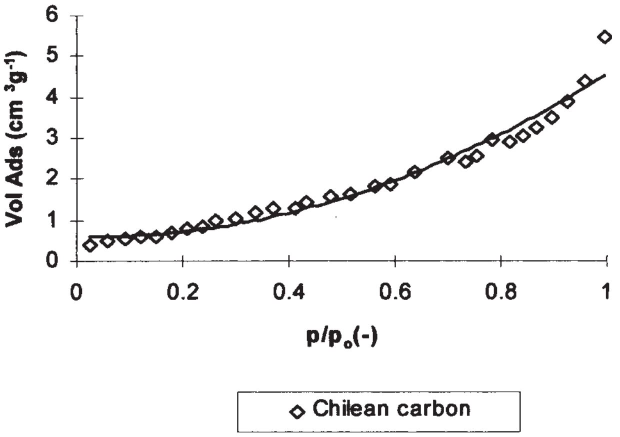

Figure 4. The N_2 (77 K) gas adsorption isotherm for Chilean carbon.

The weak adsorption capacities of Chilean carbon can be anticipated from the lower N_2 adsorption capacity values compared to the other carbons. The total nitrogen adsorption capacity for Chilean lignite was calculated from the BET equation as $6\,cm^3\,g^{-1}$. This value is considerably lower than the estimated values of other carbons. Values of 446, 431, 320. 295, and $173\,cm^3\,g^{-1}$ were calculated for FS-400, EA207, C207, Centaur, and coke carbon respectively. Close inspection of the uptake capacities of these carbon shows that carbon performance in solution can be related to its capacity to the N_2 gas where better performance is expected with higher N_2 capacity carbons. The micropore volumes were evaluated by applying the D–R equation [Eq. (4)]. This equation is normally applied only to 0.3 p/p_0 since above this value, relative pressure filling of mesopores usually starts.[15] Figure 5 shows D–R plots of N_2 gas adsorption results related to the carbons under investigation. Micropore volumes for activated carbons were estimated from the intercepts of the straight lines shown in this figure.[7] Mesopore volumes obtained from the adsorption isotherms of N_2 were calculated by subtracting the value of V_0 (N_2) from the amount adsorbed at $p/p_0 = 0.95$.[15] In type I isotherms, the amount adsorbed at p/p_0 of 0.95 corresponds to the total amount adsorbed in both the micropores (filled at low pressures) and the mesopores (filled by capillary condensation at pressures above 0.2). Consequently the subtraction of V_0 from the total amount will provide the mesopore volume. This parameter is very useful when comparing the porous texture of activated carbons and results are reported in Table 4.

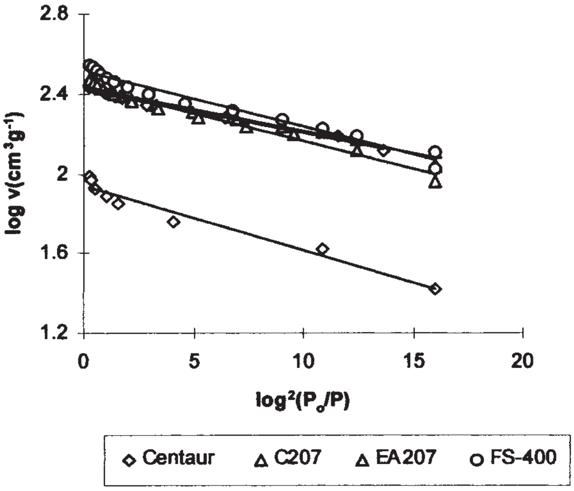

Figure 5. Dubinin–Radushkevich (D–R) plots of N_2 adsorption at 77 K.[7]

Table 4. Total pore volume, primary micropore, secondary micropore, micropore, and mesopore volumes calculated from N_2 adsorption isotherm.

Carbon	Total pore volume ($cm^3\,g^{-1}$)[a]	Primary micropore volume ($cm^3\,g^{-1}$)	Secondary micropore volume ($cm^3\,g^{-1}$)	Micropore volume ($cm^3\,g^{-1}$)[b]	Mesopore volume ($cm^3\,g^{-1}$)
FS-400	0.70	0.35	0.20	0.50	0.13
Centaur	0.46	0.31	0.11	0.42	0.03
EA207	0.58	0.31	0.15	0.44	0.14
C207	0.50	0.34	0.11	0.43	0.06
Chilean	0.01	0	0.001	0.002	0

[a] Calculated from N_2 adsorption data.
[b] Calculated from N_2 data by using D–R equation.

Due to potential importance of secondary micropores to dye adsorption, N_2 adsorption data were further treated to obtain the primary (pore diameter <0.8 nm) and secondary micropores ($0.8\,\text{nm} <$ pore diameter $< 2\,\text{nm}$). Allocation of primary and secondary micropores involved subdividing the adsorbed N_2 amounts in the range of relative pressure 0 to 0.01 and 0.01 to 0.40, corresponding to primary and secondary micropores.[16] Most carbons showed a good microporosity. Such microporosity was inferred earlier from their adsorption isotherms. Furthermore, results in Table 4 indicate that five carbons have large mesopore volumes with EA207, FS-400, and coke carbon having the largest. Except for Chilean carbon, all other carbons have secondary micropore volumes. FS-400 and EA207 have the highest secondary micropore volumes (0.20 and $0.15\,cm^3\,g^{-1}$ respectively).

The correlation between adsorption capacity of carbons and mesopore volume is shown in Table 3. A better correlation was obtained for reactive black and reactive red adsorption, while reactive yellow adsorption was better correlated to surface area than to mesopore volume. Newcombe and coworkers[16] obtained high correlation coefficients ($r^2 = 0.98$) between mesopore volume and adsorption capacity to humic substances. In a different study, Moreno-Castilla and coworkers[17] studied the efficiency of five activated carbons toward five phenolic pollutants. A high correlation coefficient ($r^2 > 0.90$) was observed between total surface area and mesopore volume. The investigators conclude that activated carbon, with the highest surface area and the most developed porosity, has the highest adsorption capacity in solution. The correlation analyses used in this work were mainly based on the work of Othman et al., 2000.[18]

In fact, 2-nm pore diameter (the upper limit of micropores) is relatively large and it is highly possible for some large molecules to get inside. Therefore, it is important to correlate the total micropore volume to the adsorption capacities. The correlation between adsorption capacity of dyes and micropore volume is shown in Table 3.

It seems that the total micropore volume has little effect compared to mesopore volume during reactive dye adsorption. In fact, a better representation for three dyes systems was obtained when carbon capacities were correlated to the secondary micropore than with total micropore volume, (see Table 3). This was predicted since not all the volume of micropores was involved during dye adsorption, only the secondary micropore volume was probably involved in that process. Hence, even if some carbons have the same micropore volumes, different magnitudes of secondary micropore volumes

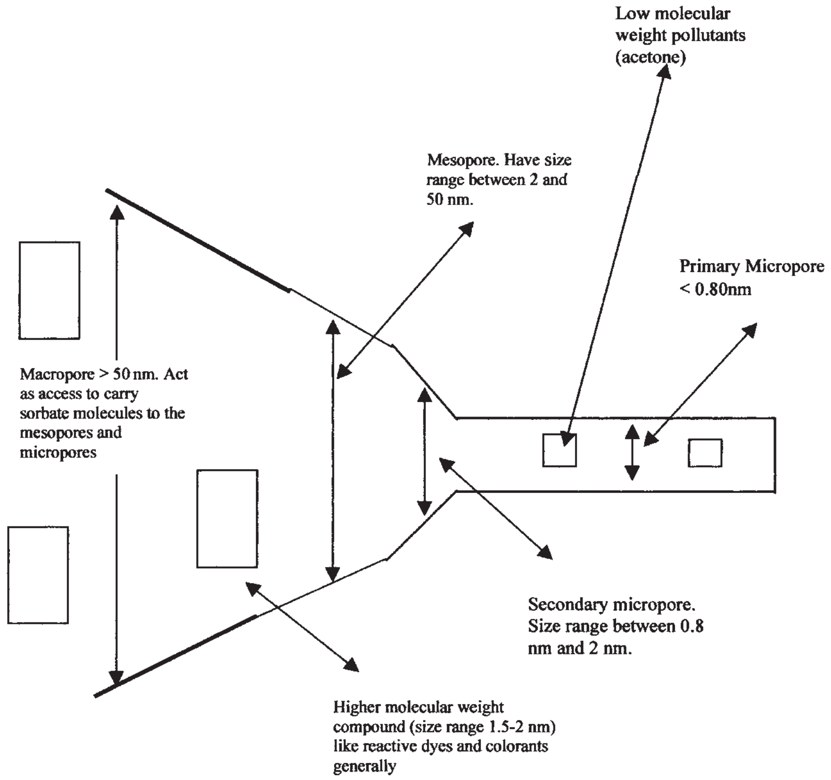

Figure 6. Visualization of the adsorption of small and large molecules by developed structure activated carbon.

may be found. The unexpected fit between secondary micropore volume and carbon capacities indicates that dye interaction with carbon occurred mainly at the areas that connected mesopore with micropores, i.e., secondary micropores. At this stage, and based on the N_2 data analysis, it may be suggested that reactive dye adsorption onto carbon surfaces may proceed partially with physical interaction, i.e., nonelectrostatic interaction. Moreover, mesopore and secondary micropore volumes were highly involved with this nonelectrostatic interaction.[16] In summary, the adsorption of reactive dyes can be visualized as shown in Fig. 6. The figure shows the dimensions of micro-, primary micro-, secondary micro-, meso- and macropores. In addition, it shows the dimensions of adsorbate molecules frequently removed by activated carbons.

CONCLUSION

Physical characterization and texture studies of six activated carbons were carried out as part of a complete study of the effect that these characteristics have on the adsorption behavior of these problematic reactive dyes from solution. The nitrogen adsorption studies proved useful in estimating various pore size distributions of the carbons. With the exception of Chilean lignite, all carbons showed a type I adsorption isotherm as well as a high developed porous structure. FS-400 carbon showed the highest degree of structure complexity, with a well-developed micro- and mesopore availability.

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