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Capacity of activated carbon in the removal of acid dyes subsequent to its thermal treatment

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Abstract

A commercial activated carbon (Prolabo) was subjected to thermal treatment at 400 and 600 °C for 2 h. Characterization of the parent, and heated products, was carried out by determining the $N_2/77$ K adsorption isotherms, FTIR spectra, acidic/basic sites, and adsorption of iodine, Acid Blue 74 (AB), Acid Red 73 (AR) and Acid Yellow 23 (AY) from aqueous solution. Thermal treatment resulted in some enhancement in the porosity characteristics (specific area and pore volume) particularly in micropores, although insignificant changes appear in the surface chemical properties. Two liquid-phase adsorption models are applied, the Langmuir and Dubinin–Radushkevich linear equations, which showed satisfactory fit to the adsorption data. Evaluated capacity and affinity parameters demonstrated that the uptake of the acid dyes decrease in the order of AB > AR >> AY, which may be ascribed to chemical structure differences in substituents and side chains leading to unavailable accessibility. Heated carbons exhibit a reduction in uptake of AB, and an increase in case of AY and AR dyes. Changes in surface chemical groups, under action of heat probably influence the adsorbate/adsorbent, adsorbate/liquid and adsorbent/liquid relationships leading to the apparent differences.

Keywords: Active carbon; Thermal activation; Dye removal; Adsorption isotherm

1. Introduction

Dyes are widely used, and discharged, in many industries such as textiles, rubber, paper, plastics, cosmetics etc. to colour their products. Dyes even in low concentrations affect the aquatic life and food web.

Acid dyes are organic sulphonic acids, the commercially available forms are usually sodium salts, which exhibit good water solubility. In sequence of their importance, acid dyes are mostly used with certain fiber types such as polyamide, wool, silk, modified acrylic, and polypropylene fibers, as well as blends of the aforementioned fibers with other fibers such as cotton, rayon, polyester, regular acrylic, etc.

According to their structure, acid dyes belong to the following chemical groups: azo, anthraquinone, triphenylmethane, pyrazolone, nitro and quinoline. Azo dyes represent the largest and most important group followed by anthraquinone and triarylmethane dyes [1].

Since many organic dyes are harmful to human beings, the removal of colour from process or waste effluents becomes environmentally important. Due to the large degree of organics and the stability of modern dyes, conventional physico-chemical and biological treatment methods are not completely effective for their removal. Besides the contaminating dyes, colour in drinking water is naturally objectionable, and is unacceptable due to ascetic reasons. The removal of colour from waste water is often more important than the removal of soluble colourless organics which normally contribute to the major BOD load.

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Nomenclature

C_{un}, C_{400}, C_{600}	Raw and 400, 600 °C-treated activated carbons
S_{BET}	Specific surface area evaluated by the BET-equation (m^2/g)
S_t^α	Total specific surface area evaluated from α_s-plot (m^2/g)
S_n^α	Non-microporous surface area from α_s-plot (m^2/g)
V_p	Total pore volume from liquid nitrogen held at $p/p^0 = 0.95$ (mL/g)
V_0^α	Micropore volume evaluated form α_s-plot (mL/g).
V_{meso}	Mesopore volume from $V_p - V_0^\alpha$ (mL/g)
Q_0	Monlolayer capacity from Langmuir model (mg/g)
K_L	Langmuir equation constant (L/g)
ΔG^0	Free energy change in adsorption from $-RT \ln K_L$
K_L	Characteristic Langmuir constant ($=1/(1+K_L C_0)$)
X_I	Fraction of surface covered by iodine (S_I/S_{BET})
S_I	Surface area covered by iodine from $Q_0 \times 0.948$
K_D	Distribution coefficient from (solute on solid)/(solute on solution) at equilibrium (L/g)
q_{DR}	Limiting adsorption in micropores (mg/g) from D–R equation
E_0	Characteristic energy of adsorption (kJ/mol) form D–R equation

Adsorption of dyes from different matrices has been reported [2–4] and the removal of various dyes categories have been discussed [5–12].

Lately, decolourization of textile effluents has received great attention, not only because of the toxicity that these effluents may have but also due to the physiological impact upon the population because their colour is often noticeable. Visual pollution, aggravated or not by toxicity questions is by itself a serious problem in water quality: it is not easy to accept red or brown rivers [13]. Colour in dye house effluents has always been a difficult problem to solve and the utilization of reactive dyes has made it even more serious.

Cooper [14] and Southern [15] summarized the technologies used until then in order to remove or at least reduce colour, mentioning that some of them have a certain efficiency: coagulation and/or flocculation, membrane technologies, (such as reverse osmosis, nanofiltration, and dialysis), chemical oxidation technologies (including Fenton's reagent with hydrogen peroxide, photocatalysis with UV radiation), biochemical oxidation and adsorbents utilization (e.g. activated carbon, inorganic adsorbents such as silica or clays, synthetic ion, exchange resins, chitin-based adsorbents and similar ones [16,18].

The adsorption process has an edge over the other treatment methods due to its sludge free operation, and complete removal of dyes even from dilute solutions. Activated carbon is the most widely used adsorbent for this purpose. The wide usefulness of activated carbon is a result of its chemical and mechanical stability, high adsorption capacity and high degree of surface reactivity [3].

In case of adsorption from the liquid systems, the chemical properties of surface groups influence the adsorption equilibria to a large extent. A significant role is ascribed to the surface functional groups containing various heteroatoms (mainly oxygen). In case of modified carbons, these oxygen functional groups are either increased as a result of treatment with a wide range of oxidants (gaseous or solution), or reduced by thermal treatment processes as these groups are mostly thermally unstable. A substantial work has been carried out on the production of activated carbons with various texture and surface chemical nature depending basically on the precursor and activation technique. However, in several cases the after-treatment generates developed carbons suitable for specific uses such as metal removal.

Since the high removal capacity towards organic pollutants is essentially physical in nature and based on its high area per unit weight, thermal treatment is expected to inflict small effect on solute uptake. In case of acidic dyes containing various functional groups, an additional factor will be associated with the abundance of surface functional groups.

The present study aims to demonstrate and illustrate the following aspects: first the removal capacity of three different acid dyes – blue, red and yellow – by a commercial activated carbon (Prolabo product); second the impact of thermal pre-treatment on the adsorption capacity; third the corresponding adsorption parameters as evaluated by two adsorption models; and fourth the role of carbon surface modifications and structural differences of the dyes on the extent of dye uptake. Thus, it tries to postulate the possible impact of thermal treatment of a bottle-available activated carbon on its surface and adsorptive properties, and to what extent it enhances its removal capacity or retards it.

2. Experimental section

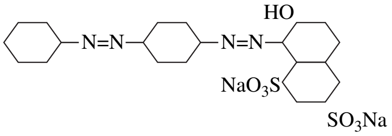
2.1. Materials

The absorbent used in this investigation was a commercially activated carbon [decolourizing powder

ADWIC (Alnasr Drug-chemicals) packed according to Prolabo] used untreated as such and on thermally heated at either 400 or 600 °C. The absorbates used in the experiments are acidic dyestuffs supplied by Aldrich company and used as the commercial salts.

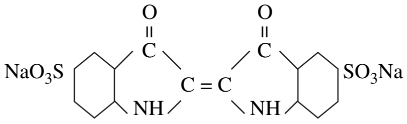
(1) Acid Red 73 (yellowish red): C.I 27290

C.I. Acid Red 73 (*Yellowish red*)



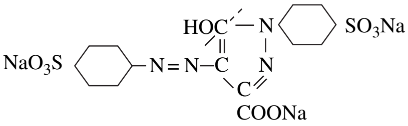
(2) Acid Blue 74 (greenish blue) C.I 73015

C.I. Acid Blue 74 (*Greenish blue*)



(3) Acid yellow 23 (yellow) C.I 19140

C.I. Acid Yellow 23 (*Yellow*)



and potassium iodide–iodine solution mixture.

3. Methods and procedures

The equilibrium isotherms were determined by contacting a mass of active carbon [0.1 g] with different concentrations of dye solution for 48 h at room temperature in a constant agitation shaker. After that the samples were filtered and the dye concentration in the supernatant solution was estimated by measuring absorbance with a Shimadzu UV–Vis 2401 PC recording spectrophotometer using a 1 cm light-path cell. The wavelength was selected so as to obtain maximum absorbance for each dye stuff and the λ_{max} values are given as follows for: Acid Blue, $\lambda_{max} = 607$, Acid Red, $\lambda_{max} = 509$, Acid Yellow, $\lambda_{max} = 426$ and iodine $\lambda_{max} = 474$ [19].

3.1. Liquid-phase adsorption

The amount of dye adsorbed on the carbons, q_e (mg/g) was calculated by mass balance relationship by Eq. (1)

$$q_e = [C_0 - C_e]\frac{V}{\omega} \tag{1}$$

where C_0 and C_e are the initial and equilibrium liquid-phase concentrations, respectively (mg/L), V the volume of the solution and W the weight of the carbon used (g) [19]. IR analysis was performed by using test scan Shimatzu-FTIR for untreated carbon and solids thermally heated at 400 and 600 °C, and spectra were scanned over the 4000–500 cm^{-1} range.

The isothermal equilibrium data were analyzed by applying the Langmuir isotherm equation in its linear form

$$\frac{C_c}{q_e} = \frac{1}{K_L Q_m} + \frac{C_e}{Q_m} \tag{2}$$

where Q_m is the maximum amount of adsorption corresponding to complete monolayer coverage on the surface (mg/g), C_e the adsorbate equilibrium concentration (mg/L) and K_L the Langmuir constant (L/mg). A linearized plot of C_e/q_e versus C_e is obtained and both Q_m and K_L are computed from the slope and intercept [20]. One of the essential characteristics of the Langmuir isotherm could be expressed by a dimensionless constant called equilibrium parameter, R_L,

$$R_L = \frac{1}{1 + K_L C_0} \tag{3}$$

where C_0 is the initial maximum solute concentration. The values of R_L indicate the type of isotherm to be reversible ($R = 0$), favourable ($0 < R_L < 1$), linear ($R = 1$) or unfavourable ($R > 1$). The free energy of adsorption, ΔG^0_{ad} can be evaluated from the parameter K_L (mol/L) according to [20]

$$\Delta G^0_{ad} = RT/\ln K_L \tag{4}$$

The liquid-phase adsorption data can also be analyzed by the Dubinin–Radushkevich equation [19]. Deriving this equation for liquid-phase adsorption, the amount adsorbed corresponding to any adsorbate [21] concentration is assumed to be a Gaussian function of the Polanyi potential [21,23] ε,

$$q_e = q_{Dr}[\exp(-B\varepsilon)]^2 \tag{5}$$

with

$$\varepsilon = RT\ln(1 + 1/C_e) \tag{6}$$

where q_{DR} is the maximum amount of adsorbate that can be adsorbed in carbon micropores, B is a constant related to the energy, and C_e is the equilibrium concentration in units of g (adsorbate) (g solution)$^{-1}$. If the carbon surface is heterogeneous and an approximation to a Langmuir isotherm is chosen as the local isotherm on each homogeneous subregion [21], then the most probable free energy of adsorption is

$$E=(2B)^{-1/2} \tag{7}$$

Therefore, the D–R equation can be used to estimate not only the maximum adsorption capacity, but also an average free energy value unique to the adsorbate system. Eq. (6) can be linearized and expressed as

$$\ln q_e = \ln q_{DR} - B\varepsilon^2 \tag{8}$$

A linear plot of $\ln q_e$ against ε^2 would give the values of q_{DR} and B (thus the mean free energy of adsorption, E) from the intercept and slope.

A different parameter was additionally evaluated which indicates adsorption affinity, that is the distribution coefficient, K_D, calculated by

$$K_D = \frac{q_{ei}}{C_{ei}}$$

(where i = number of data points) in L/g units. It indicates distribution between sorbent and aqueous solution [22].

3.2. Characterization of carbons

The activated carbons were characterized by N_2/77 K adsorption using a Gemini 2375 V3.03 [24] and the isotherms analyzed by application of the BET and α_s-methods [12]. Several textural parameters were evaluated: S^α (total specific area), S^α_n (non-microporous area), total pore volume, V_p (amount adsorbed at a relative pressure of $p/p^0 = 0.95$), micropore volume, V^α_0, and mesopore volume, V_{meso} (from volume at $p/p^0 = 0.10–0.95$) [25].

4. Results and discussion

4.1. Characteristics of parent and thermally treated activated carbons

4.1.1. Textural modifications

Figs. 1–3 present N_2/77 K adsorption isotherms, pore-size distributions and α_s-plots for the three carbons. Thermal treatment at 400 and 600 °C seems to inflict no observed changes in the shape of either relationship. The adsorption isotherms are typical type II of the BDDT classification, with limited adsorption at high pressure indicating a micro/mesoporous character. This is corroborated by both the pore-size distribution (two maxima around 25–30 and 80–100 Å) and the type α-2 of the α_s-plots associated with same trend of wide distribution of narrow and wide micro/mesoporosity [26].

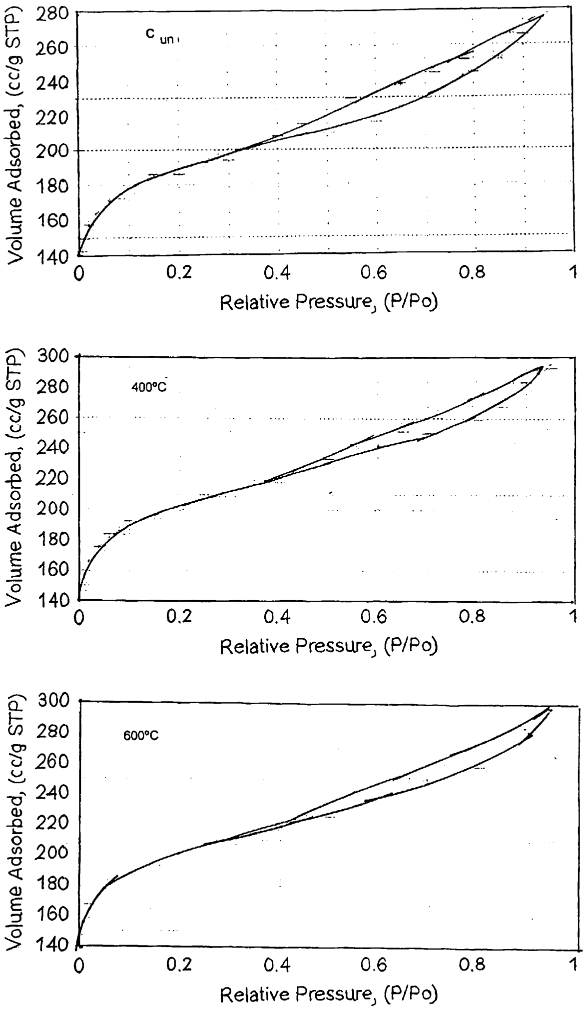


Fig. 1. Adsorption isotherms of N_2/77 K for the three carbons.

Table 1 shows the porous characteristics and displays the porosity changes consequent to the thermal treatment up to 600 °C. The parent carbon exhibits a moderately developed porosity, with specific surface $S_{BET} = 685$ m^2/g and total pore volume of 0.429 mL/g, calculating an average micropore radius of 12.6 Å. Upon considering the ratio of mesoporous surface $[S_n/S_t = 0.275]$ it is clear that the tested carbon is essentially microporous, whereas the micropores $[V_0/V_p]$ contribute to 47% of total pore volume. Thus, it is a mixed porosity carbon with appreciable contribution in volume of mesopores. Heating at 400 °C slightly enhances porosity [~7% increase in surface both total surface area and pore volume]. This rise seems to be related to creation [or clearing] of micropores [viz. $S^\alpha_{mic} = S^\alpha_t - S^\alpha_n$ and V^α_0].

Further raising of temperature is not accompanied by any perceptible change in porosity parameters.

Accordingly, thermal treatment of a commercially available carbon at 400 °C leads to a small modification in both its porous characteristics and the relationships

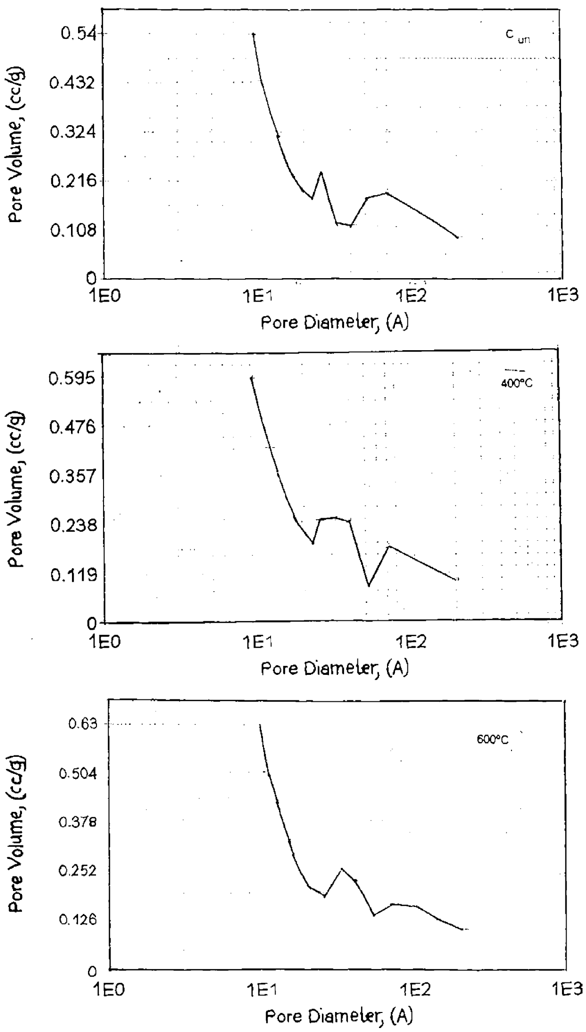


Fig. 2. Pore-size distribution of the tested carbons.

already present in the original adsorbent [micro/mesoporosity]. Heating up to 600 °C is expected to desorb already adsorbed gaseous matter accumulated during processing and storing. Their release clears the internal porosity and thus generates the observed small changes. It is equally probable that under action of heat, several surface oxygen functionalities are decomposed as will be demonstrated next.

4.1.2. Physico-chemical changes

According to the procedure of Boehm [27], the total acidity and basicity are determined by titration with either 0.1 N NaOH or 0.1 N HCl, respectively. The parent carbon showed a low content of acid groups [1.1 mmol/g] which decrease to 0.91 mmol/g for the 400 °C-product and slightly reincrease to 1.24 mmol/g for the 600 °C-product. The basic sites showed no variations, 0.33 mmol/g, with thermal treatment. A pH value of 7.2–7.5 was recorded for the carbon/water suspensions. These data are supplemented by recording the FTIR spectra of the three carbons shown in Fig. 4. The three spectra show exactly the same absorption bands with negligible variations, except in intensity for the 600 °C-product. The band at 3140 cm^{-1} is usually assigned to hydrogen-bonded O—H, whereas the broad band noticed at 3420 cm^{-1}, assigned to absorbed water, is destroyed by heating. A weak absorption at 2360 cm^{-1} is usually ascribed to single C—H bonds, that at 1640 cm^{-1} is ascribed to aromatic ring stretching or highly conjugated C═O stretching. The sharp prominent absorption at 1395 cm^{-1} is assigned to carbonyl-carbonate or carboxylic salt or metal-carbonate. Other broad absorption around at 1120 cm^{-1} is ascribed to C—O stretching in alcohol or ether or hydroxyl groups [28].

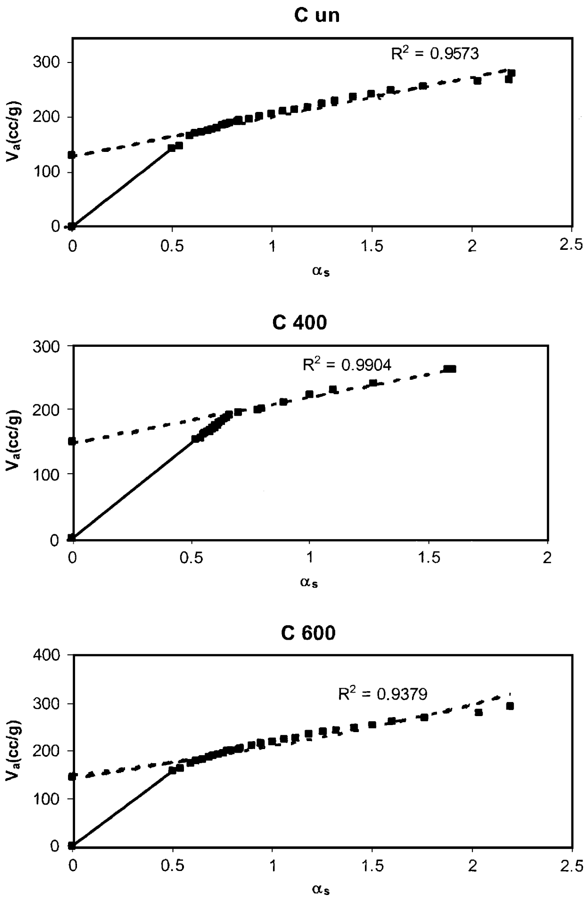


Fig. 3. α_s-Plots for the three tested carbons.

Table 1
Porous characteristics of parent and thermally treated activated carbons

Sample	S_{BET} (m^2/g)	V_p (mL/g)	$\bar{r}$ (Å)	S_t^{α} (m^2/g)	S_n^{α} (m^2/g)	V_0^{α} (mL/g)	V_{meso} (mL/g)
C_u	683	0.4291	12.6	765	211	0.202	0.228
C_{400}	732	0.4584	12.5	802	180	0.235	0.223
C_{600}	728	0.466	12.6	838	205	0.222	0.237

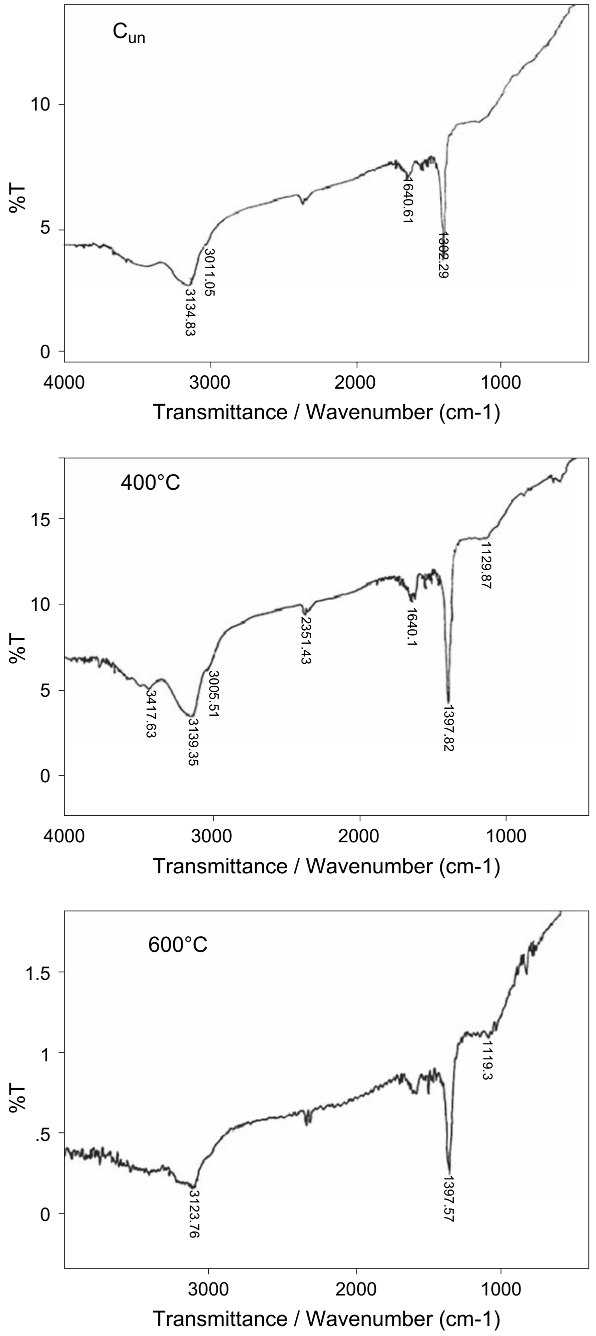


Fig. 4. FTIR spectra of the parent and treated carbons.

It appears that, in general, thermal treatment of the investigated activated carbon results in very slight modifications in the surface chemical structure indicating stability of the tested amphoteric carbon, the case with steam-activated carbons. The apparent small changes seem to be only in the porosity characteristics.

4.2. Adsorption characteristics from the liquid phase

Equilibrium isotherm data are obtained for iodine, Acid Blue (AB) Acid Red (AR) and Acid Yellow (AY) onto the three tested carbons: virgin, 400 and 600 °C-products [Fig. 5]. Satisfactory linear relations were obtained by the Langmuir adsorption model, but in case of the D-R model the low equilibrium concentration values were off the obtained linear relations. Most of the evaluated adsorption capacities, form both models, are generally concordant, although the thermodynamic parameters are different [viz. $\Delta G^0 = 5-8$ kJ/mol and $E^0 = 11-18$ kJ/mol] as they denote different functions. Only in case of iodine they are comparable. The dye removal capacities are also presented in another presentation i.e. the (K_D) against initial solute concentrations [Fig. 6].

4.2.1. Adsorption of iodine from aqueous solutions

This test is usually complimentary to the N_2/77 K adsorption isotherms, and assumed to measure the surface area in micropores within pore sizes ≥ 10 Å [29]. Assuming the currently employed area of $40A^2$ as the cross sectional area covered by an adsorbed iodine molecule, this permits evaluation of the surface areas (S_I) shown in Table 2. The virgin carbon seems to contain appreciable narrow microporosity unavailable to iodine where the amount adsorbed of iodine covers only 0.86 of the total surface area $\left(S_t^\alpha\right)$. Thermal treatment at 400 and/or 600 °C clears this ultraporosity that becomes completely accessible to the relatively larger iodine as compared to nitrogen molecules ($X_I \sim 1.0$).

The constants of the two adsorption models, Langmuir and D–R are slightly different inspite of their different theoretical basis. The monolayer capacities (Q_0 and q_{DR}) are nearly the same, and both energy parameters [ΔG^0 and E^0] are somewhat smaller in case of the latter model. Accordingly, the low characteristic energy of adsorption [$\sim$6.0 kJ/mol] indicates the pure physical nature of iodine adsorption by the three activated carbons, but it slightly increases by heating at 600 °C. It is noticeable that the uptake of iodine by the tested carbons is highly favourable from solutions containing up to 200 ppm as evident from the high distribution coefficients, although the parent carbon exhibits the highest affinity $K_D \sim 38$ in comparison to ~ 21, shown by the heated carbons.

4.2.2. Uptake of the acid dyes by virgin carbon

Removal of the tested three dyes exhibits characteristic different capacities in the order AB > AR >> AY (Table 3). This trend is clearly evident from the various evaluated model parameters: Q_0 (or q_{DR}), K_D (at 100, 200 ppm) and a reverse function of energy of adsorption. The widely different adsorption capacities cannot be ascribed to apparent differences in adsorbent properties, since we are dealing with one and the same carbon. It is characterized by a good specific surface area within a micro/mesoporous texture and appreciable mesoporosity (S_n^α and V_{meso}, Table 1).

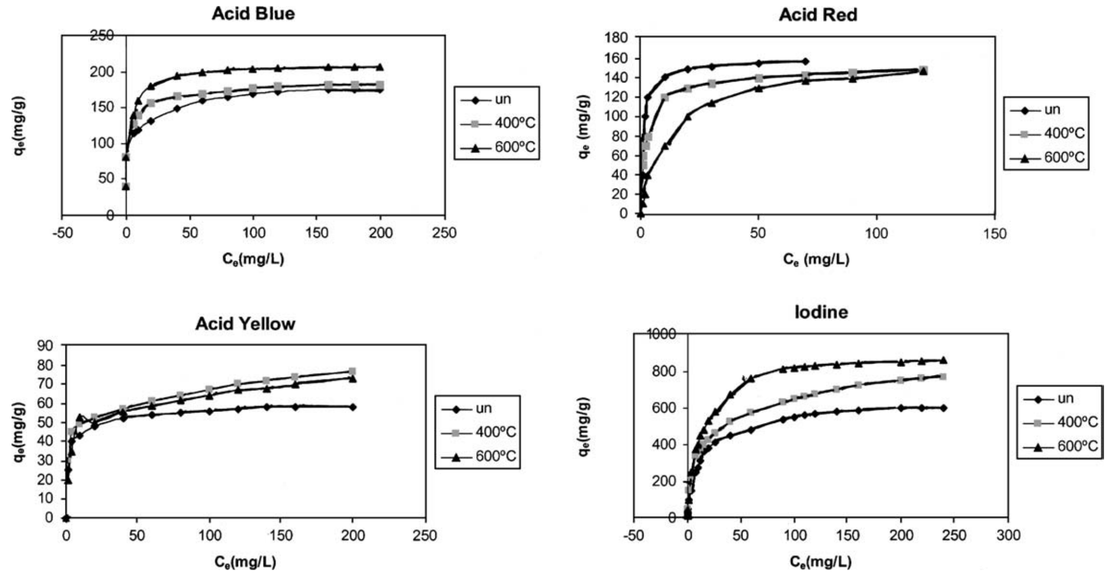


Fig. 5. Adsorption isotherms of iodine and three acid dyes with parent carbon (C_{un}) and 400 and 600 °C of treated carbons.

The chemical structures of the dyes could be responsible for the apparent different capacities. The very low uptake of AY could be due to its chemically different structure with side chain and presence of both the sulfonic and azo groups which might lead to the formation of large aggregated molecules that hinder their free diffusion to the internal porosity. The straight chain composition of AB may explain its high uptake value, whereas the AR contains an azo entity with a phenolic ring in the side chain which could be responsible for its relatively lower adsorption. An additional factor that might add to discrepancy in the removal capacity of the three dyes could be the surface oxygen functionalities which are greatly reduced [or decomposed] by thermal treatment, which will be demonstrated next.

4.2.3. *Uptake of the acid dyes by the heat-treated activated carbons*

Tables 4 and 5 show the adsorption characteristics of the 400 and 600 °C-treated carbons. The three dyes are adsorbed in the same order, mentioned above, as deduced from their removal capacity and distribution coefficients, that is AB > AR >> AY. The free energy and energy of adsorption change slightly but not in a regular trend. However, whereas the specific area is increased by heating, yet the uptake capacity for AB is reduced by about 20%, whereas that for AR and AY is

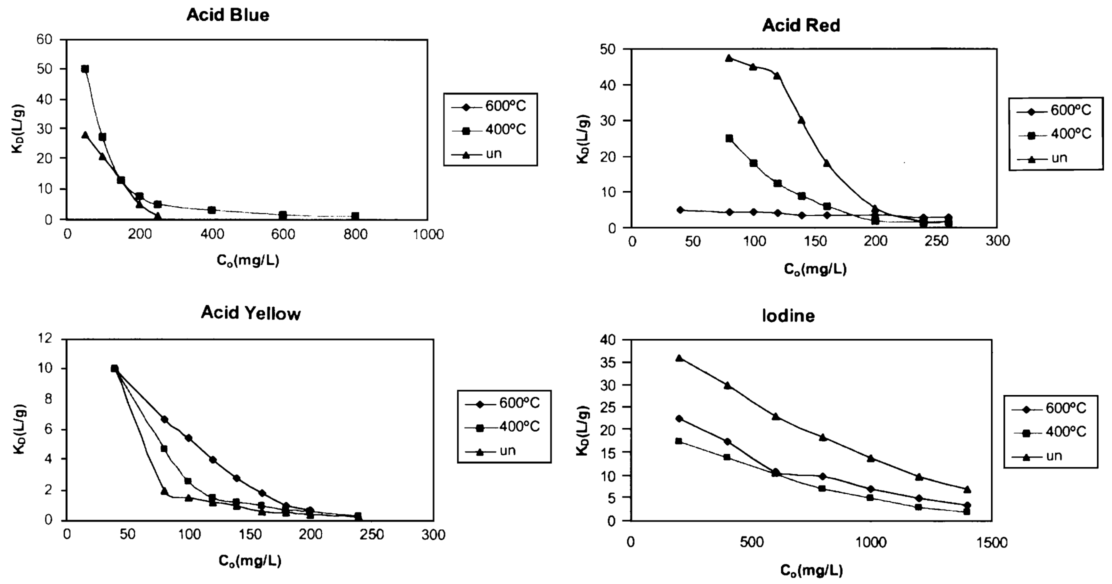


Fig. 6. Variation in the distribution coefficients as function of initial solute concentration.

Table 2
Characteristics of iodine adsorption from solution

Model	Parameter	Sample		
		C_u	C_{400}	C_{600}
Langmuir	Q_0 (mg/g)	694	833	892
	S_I (m^2/g)	658	789	845
	K_L (L/g)	0.065	0.041	0.44
	ΔG^0 (kJ/mol)	−6.80	−7.95	−7.77
	R_L	0.0941	0.0152	0.0138
	X_I	0.860	0.984	1.008
D−R	q_{DR} (mg/g)	663	794	913
	E_0 (kJ/mol)	5.94	5.59	6.66
K_D	At 100 ppm (L/g)	40.0	19.5	25.0
	At 200 ppm (L/g)	36.4	17.5	22.5
Site uptake (μmol/m^2)		4.000	4.479	4.824

Table 4
Adsorption characteristics of the 400 °C-treated active carbon towards the three acid dyes

Model	Parameter	Solute		
		AB	AR	AY
Langmuir	Q_0 (mg/g)	174	155	75.0
	K_L (L/g)	0.145	0.0921	0.1333
	ΔG_0 (kJ/mol)	−4.84	−5.94	−5.01
	R_L	0.019	0.042	0.029
D−R	q_{DR} (mg/g)	182	154	71.5
	E_0 (kJ/mol)	15.9	13.3	16.9
K_D	At 100 ppm	16.0	12.5	2.8
	At 200 ppm	4.5	2.0	0.5

enhanced by 20% for AR and 40% for AY. These changes could be associated with generated porosity, however, still the yellow dye is removed in the lowest amounts compared to its counterparts, that is the blue and red acid dyes. It means that thermal treatment of the present activated carbon results in the decrease of adsorption capacity of Acid Blue as noticed from the values of monolayer capacity and distribution coefficients [especially at low concentration $\leq$ 100 ppm]. But the same treatment improves appreciably the low uptake of Acid Yellow, based on the same parameters. Fig. 6 illustrates the demarcation of removal of the three dyes (as well as iodine) under effect of thermal treatment in terms of distribution coefficients. Thus, generally, iodine and Acid Red affinities are reduced markedly by thermal treatment irrespective of their apparent total removals. Acid Blue and AY affinities are remarkably increased, particularly at low dye loading ($\leq$100 mg/L).

5. Conclusion

Thermal treatment of a commercially activated carbon [Prolabo] was carried out to assess the impact of this process on the internal porosity and removal capacity towards three acid dyes, Acid Blue (AB), Acid Red (AR) and Acid Yellow (AY). The parent carbon exhibited a moderately developed porosity [as well as iodine uptake] within the micro/mesoporous ranges. Thermal activation leads to a small enhancement in microporosity due to the removal of adsorbed gases acquired during processing and storing.

A small reduction in surface acid sites appears as a result of heating, with no change in the basic sites, and associated with same FTIR absorption spectra. Removal of the three acid dyes shows the same pattern, irrespective of thermal treatment, that is decreasing capacity in the order AB > AR >> AY. This was ascribed to the different chemical natures of the three dyes, with respect to substituents and/or branched side chains that increase bulkiness and reduce diffusion ability. Solute properties are here the main factor determining the adsorption capacity, since the adsorbents are the same in each case. Heat treatment of the tested commercial activated carbon results in two different effects either an increase in uptake of AY and AR or a decrease in case of AB, as function of heat-treatment temperature.

Table 3
Adsorption characteristics of parent active carbon towards the three acid dyes

Model	Parameter	Solute		
		AB	AR	AY
Langmuir	Q_0 (mg/g)	230	143	56.5
	K_L (L/g)	0.0869	0.191	0.171
	ΔG_0 (kJ/mol)	−6.08	−4.12	−4.41
	R_L	0.044	0.021	0.023
D−R	q_{DR} (mg/g)	224	167	59.7
	E_0 (kJ/mol)	15.4	17.3	18.6
K_D	At 100 ppm	22.0	50	1.5
	At 200 ppm	9.0	5.50	0.5

Table 5
Adsorption characteristics of the 600 °C-treated active carbon towards the three acid dyes

Model	Parameter	Solute		
		AB	AR	AY
Langmuir	Q_0 (mg/g)	190	174	72.5
	K_L (L/g)	0.1316	0.0441	0.0984
	ΔG_0 (kJ/mol)	−5.05	−7.76	−5.77
	R_L	0.029	0.083	0.031
D−R	q_{DR} (mg/g)	189	173	70.5
	E_0 (kJ/mol)	17.3	10.5	17.7
K_D	At 100 ppm	49	4.0	5.5
	At 200 ppm	6.5	2.0	0.5

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