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ADSORPTION OF METHYLENE BLUE BY APRICOT STONES-DERIVED – ACTIVATED CARBON

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ABSTRACT

Activated carbons from apricot stone shells were tested for their adsorption capacities towards methylene blue under equilibrium and kinetic conditions. Satisfactory Langmuir plots were obtained with monolayer capacity of 170-440 mg.g⁻¹.

Linear Freundlich relationships indicate favourable adsorption in the carbon/dye system. Some carbons show higher affinity and suitability for column application. Adsorption seems to be controlled by both mass transfer and intraparticle diffusion processes due to the high concentration involved and to the poor mixing conditions.

Highly adsorbing carbons are observed as inferred from the high dye removal capacity attaining $\ge 93\%$ at initial concentration up to 600 mg.dm⁻³.

INTRODUCTION

Activated carbons are the most efficient sorbents for use in gas and water purification [Mastral, et al. (2000) and Podkoscietny, et al. (2003)] and therefore they find wide applications in many processes such as wastewater treatment, air purification and solvent recovery [Barsal, et al. (1988) and Patrik, (1995)]. The most toxic organic pollutants include phenols [Kilduff & King (1997) and Girgis & Daifullah (1998)] and dyes [Yupeng Guo, et al. (2003) and Aitcheson, et al. (2001)], while the most hazardous inorganic pollutants are the heavy metals [Vlaoclimir & Danish (2003) and Youssef, et al. (2004)]. The organic and inorganic pollutants are conventionally removed via adsorption. Powdered activated carbons (PAC) and granual activated carbons (GAC) are the most effective sorbents for the removal of these contaminants from wastewater and potable water [Toles, et al. (1997)].

A current trend in the preparation of activated carbons is the use of various kinds of waste materials [Youssef, et al. (1994) and Hourich, et al. (1999)] and agricultural byproducts [Attia, et al. (2003) and Rashwan, & Girgis (2004)].

For controlling organics, the adsorbent can be passed through the water, or the water can be passed though the adsorbent. In practice PAC and GAC are good examples for each respective method [Grauford & Cline (1990)]. The effectiveness of an activated carbon for its use in industrial applications is determined by adsorption of different adsorbates; e.g. methylene blue, p-nitrophenol and iodine, from solution, are usually employed [Aydin & Tez (1990)].

In general, pore structure and high surface area are the most important characteristics of activated carbon in adsorption of materials from water [Rashwan, & Girgis (2004)]. Most of the surface available for adsorption by carbon is located in pores within the carbon particles created during the activation process and the major contribution to surface area is located in pores of molecular dimensions [Culp, et al. (1978)]. Such highly adsorbing carbons depend on both the carbon precursor, and the conditions of preparation.

In a previous communication [Philip & Girgis (1996)], it was found that apricot stone shells present a good precursor for activated carbon when treated with H_3PO_4 followed by carbonization up to 500°C.

The object of the present investigation deals with these carbons that deserve testing when contacted with a standard dye as methylene blue in solution (MB), under equilibrium or kinetic conditions, Langmuir and Freundlich isotherm plots were fitted to the data [Hsisheng & Chien (1998)], the mass transfer and pore diffusion coefficients evaluated, and the dye removal capacity established. These adsorption characteristics are correlated either to the conditions of preparation (% H₃PO₄, or temperature of carbonization) and /or to the pore structure parameters (specific surface area and total pore volume).

EXPERIMENTAL

1- Materials and methods.

Ten activated carbon samples, were prepared [Philip & Girgis (1996)] from apricot stone shells with different H_3PO_4 concentrations (20-50%) and carbonization at 300-500°C. The surface areas of these samples were determined by nitrogen adsorption at -196°C with the help of a conventional volumetric apparatus and applying the BET-equation. The prepared samples were tested for their adsorption capacities towards methylene blue (MB). Their notations and conditions of preparation are summarized in Table I.

Using 150 ml stoppered conical flask, 0.2g of PAC are weighed, then 50ml of MB solution of various concentrations (200-1200mg.dm⁻³) are added. The stoppered conicals were immersed in a water bath thermo- stated at 35°C for 3 days, with occasional shaking. Residual concentration of the methylene blue (MB) in solution was determined by measuring its adsorbance at 670nm with the help of a Spectrophotometer (Unicom SP-500).

For kinetic experiments, 0.2g of the PAC was contacted with 50 ml of MB solution with the highest concentration of 1200 mg.dm⁻³. Successive determinations of the dye concentration were evaluated at suitable time intervals up to 80h.

2- Analysis of the equilibrium adsorption isotherms.

Langmuir isotherm is applied in its linear form:

 $C_e/q = 1/K_L q_m + 1/q_m - C_e,.....(1)$ where C_e is the equilibrium concentration mg.dm⁻³, q is the amount adsorbed (mg.g⁻¹) at the equilibrium concentration C_e , K_L is an empirical constant (dm³.g⁻¹) and q_m is the maximum adsorption capacity (mg.g⁻¹). A plot of C_e/q versus C_e would give K_L and q_m from the slope and intercept of the Langmuir plots.

Another adsorption model introduced by Freundlich is also applied in its logarithmic form:

 $Log q = Log K_F + 1/n Log C_c, \dots \dots \dots (2)$

q and C_e have been previously defined, K_F and n are Freundlich constants with n giving an indication of how favourable the adsorption process is, and K_F $(mg/g. dm^3/mg)^n$ is related to the capacity of the adsorbent.

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3- Analysis of the kinetic isotherm data.

Determination of the external mass-transfer coefficient is based on the work of Furusawa and Smith [Furusawa & Smith (1973) and Furusawa & Smith (1974)]. The change in the dye solution concentration with time is related to the fluid-particle mass-transfer coefficient by the equation:

 $dC_t/dt = -K_s S_s (C_t-C_s)$ with $C_t = C_o$ at t = 0....(3)If effective diffusion coefficients are neglected and as C_s approaches zero and C_1 approaches C_o as $t \rightarrow 0$, equation (3) becomes:

where C_t (mg.dm⁻³) is the liquid – phase concentration at time t, C_s (mg.dm⁻³) is the liquid – phase concentration at the outer surface of the particle, C_o (mg.dm⁻³) is the liquid–phase concentration at t=0, K_s (cm.s⁻¹) is external mass-transfer coefficient and S_s (cm⁻¹) is the outer surface area of adsorbent particles per unit volume of particle-free liquid.

Thus, equation (4) can be used for obtaining K_s by drawing a tangent to the curve C_t/C_o vs. t, but is only strictly valid for time t=0 [Mckay, et al. (1986) and El-Geundi (1990)].

RESULTS AND DISCUSSION

1-Textural properties

The textural properties (surface area, total pore volume and pore volume distribution) of an activated carbon are the most important parameters which determine its adsorption capacity both from solution and also from the gas phase. Columns 4-6 of Table I give the surface areas S_{BET} (m.²g⁻¹), the total pore volumes V_p (ml.g⁻¹) and mean pore radii r (Å) of the carbons investigated. The total pore volumes and the BET-surface areas depend on the percentage of H₃PO₄ used in the activation and also on the carbonization temperature. Surface areas between 642 and 1414 (m.²g⁻¹) have been measured indicating expanded surface for adsorption. The mean pore radii lie either in the microporous range (< 10 Å) or at the lower margin of mesoporous range (10.3 -12.3 Å) indicating that large pore fractions are of the microporous type. The adsorption of a bulky molecule such as MB from solution is propably dependent on the pore size particularly when micropores dominate. Table I depicted that q generally increases with the increase of S_{BET}

values. Evidently, q_m of carbon 53 is higher than that of carbon 52 although the S_{BET} of the former is lower. The relatively large mean pore radius of carbon 53 compared with carbon 52 may explain this trend. Another evidence may be observed when MB adsorption of samples 43 and 44 is considered.

Sample No.	Treatment		Characterization Data			Langinuir plots		Freundlich plots	
	%н,ро4	нтт("с)	S _{RF1} m ² .g ⁻¹	Vp ml.g ^{.1}	r (Á)	qm nig.g."	Ki dm. ³ g ⁴	n,	K _f
32	20	300	642	0.340	10.6	172	0.0363	3.57	37,10
33	30	300	87 B	0.395	9,0	280	0 0137	2.58	25,70
42	20	400	792	0.392	9,9	203	0.0308	3.68	41.70
43	30	400	946	0.448	9,5	283	0 0272	5.26	91 20
44	40	400	1135	0.495	8.7	275	0.0145	2.67	27 50
43	50	400	1285	0.665	10.3	292	0,0228	3.74	57.50
52	20	500	1308	0.619	9.5	312	0.0188	3.11	43.60
53	30	500	1231	0.641	10.4	361	0.0310	3.48	77,60
54	40	500	[4]4	0 758	10.7	441	0.0566	4.80	128.8
55	50	500	1140	0.700	12.3	362	0.0502	5.32	123.0

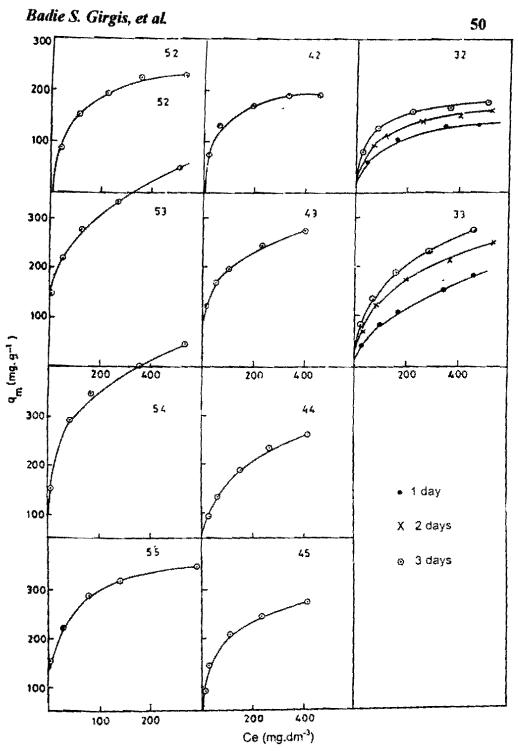
 Table I: Textural parameters and adsorption parameters of MBactivated carbon systems.

(HTT) = heat treatment temperature.

2- Equilibruim of MB on the activated carbons.

To facilitate the estimation of the adsorption capacities at various liquid-phase concentrations, the two well known equilibrium adsorption isotherm models, Freundlich and Langmuir, were employed. Although the Freundlich equation is an empirical one, yet it is employed to describe the adsorption data. The parameter K_F can serve as a measure of the relative adsorption capacity of different carbons at the same equilibrium concentrations.

For the adsorption isotherms of MB (Fig.1) the residual dye in solution was determined after three days. A representative set of data (carbons obtained at 300° C) are given in the same figure for equilibrium concentrations attained after 1,2 and 3 days. This indicates that adsorption of the dye is slow and that a period of 3 days may establish equilibrium conditions.





Adsorption-Methylene Blue-Activated Carbon.

According to Walters and Luthy [Walters & Luthy (1984)] it seems that some isotherms belong to the Freundlich type (Nos.33, 53 and 54) and that all the others exhibit Langmuir isotherms and attain distinct limiting values. Table I summarizes the evaluated Langmuir (q_m , K_L) and Freundlich (n_F , K_F) isotherm parameters. Except for carbons 32 and 42, all solids are characterized by high adsorption capacities (280-440 mg.g⁻¹). However, the K_L constants do not show any regular trend of values.

From the Freundlich isotherm plots (Fig.2), satisfactory linear plots are observed in the whole range of concentration. The estimated n_{F} -values indicate that most of the tested carbons show high affinity in removing the dye molecules (n=2.5-5.3) and appear to be concentration-independent [Fornwalt & Hutchhins (1966)].

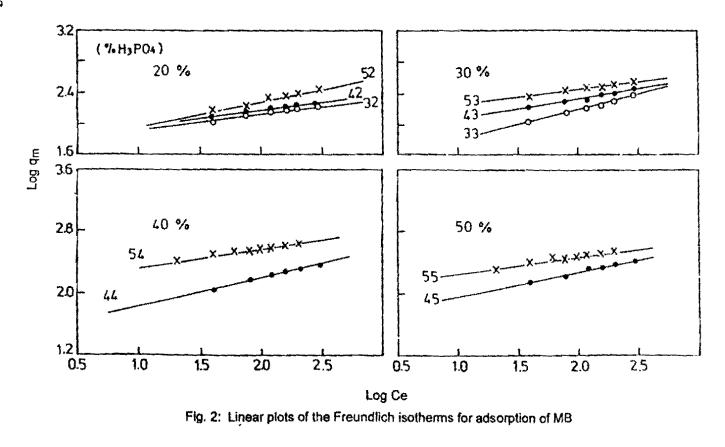
It is reasonable to conclude that carbons 54 and 55 would be the most efficient in achieving batch treatment. Their four corresponding parameters are the highest (q_m , K_L , n_F and K_F). These carbons are treated at the highest temperature, 500°C, and by the highest H₃PO₄ concentrations (40 and 50%). Such results will be further illucidated with the help of the kinetic experiments.

3- Kinetic adsorption of MB by the activated carbons.

[A] Parameters of the external mass transfer process.

It is generally agreed that the adsorption process is very rapid and does not represent the rate-limiting step of the uptake or organic compounds [Zogorski, et al. (1976)].

With respect to intraparticle diffusion, good linearization of adsorption data was reported with porous adsorbents by plotting the amount of solute adsorbed per unit weight versus the square root of time $(t^{1/2})$ [Zogorski, et al. (1976); Morris & Weber (1964); Weber & Morris (1964) and Snoevink (1968)]. Its slope has been described as a relative rate constant. However, Zogorski et al [Zogorski, et al. (1976)] proposed to characterize the slope of the cumulative removal versus $t^{1/2}$ as "removal rate". From S-shaped uptake curves, they linearized the intermediate portion to evaluate this diffusion constant.

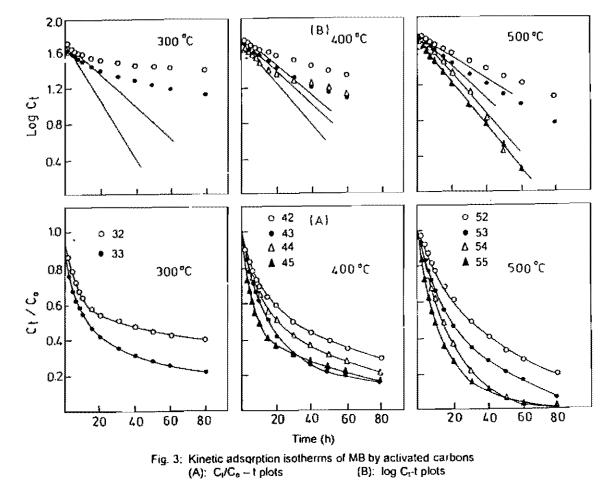


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Bhaskar and Bhamidimarri, [Bhaskar Later on, & Bhamidimarri (1992)] suggested another approach to calculate the external mass-transfer coefficient. Since the intraparticle diffusional resistance is negligible in the initial stages of adsorption, thus, the slope of the bulk concentration (C_1) for the first few minutes on a semi-log graph gives the film mass-transfer coefficient directly [Peel, et al. (1981)]. The plot indicates that the mass-transfer is controlling in the early stages and the deviation thereafter is due to the development of intraparticle diffusion resistance. The present study is based on the work of Furusawa and Smith [Furusawa & Smith (1973) and Furusawa & Smith (1974)].

Fig. 3A illustrates the rate experiments plotted as C_t / C_o versus time t. Such a plot represents the rate of change in residual dye in solution [Mckay, et al. (1986)]. By drawing a tangent to the obtained curves, the external mass transfer coefficient, K_s is evaluated as t tends to zero (Table II). In all cases the initial rapid stage of adsorption is observed then gives way to a very slow approach to equilibrium. During the initial fast adsorption the intraparticle diffusional resistance is negligible, thereafter it becomes active.

Fig. 3B shows the plots of log C_i versus t, as suggested by Bhaskar et al [Bhaskar & Bhamidimarri (1992)]. A short linear section appears in the early few hours, followed by a longer linear portion that covers the remaining period up to 80 h. Mass transfer coefficients determined from the early stage are given in Table II. Another constant was estimated from the latter long linear section which could be assumed as associated with the intraparticle diffusion process [Bhaskar & Bhamidimarri (1992)].



Samala Na	<u>Mass 1</u>	Uptake – t ¹⁷² plots			
Sample No.	C _t /Co-t plot	Log C	D _s		
	Ks	(K _s)L	(K _s) _h	mg/g.h ^{1/2}	
32	0.055	0.0193	0.0015	34.4	
33	0.103	0.0225	0.0041	45.8	
42	0.034	0.0154	0.0048	26.1	
43	0.062	0.0185	0.0055	35.6	
44	0.023	0.0176	0,0062	33.9	
45	0.143	0.0226	0.0059	46.8	
52	0.023	0.0120	0.0084	33.5	
53	0.037	0.0165	0.0111	44.0	
54	0,042	0.0211	0.0287	49.7	
55	0.067	0.0255	0.0255	53.5	

Table II: Mass transfer coefficients (K_s) and relative inraparticle diffusion rate constants (D_s).

(L) denotes low section values.

(h) denotes high section values.

Mass transfer seems to be more active with carbons prepared at low temperatures. Thus, for example, carbon 32 gives $K_s = 0.055$ which is considerably higher than K_s of carbon 42 which in turn is higher than that of carbon 52. The same is also true when carbon 33, 43 and 53 are considered. The decrease of K_s with the rise of the carbonization temperature may be attributed to the fact that the rise of the carbonization temperature always gives less contaminated porous carbons. Apparently in most cases K_s continuously increases with the increase of percentage impregnation. This may be attributed to the existence of traces of the activating agent (H₃PO₄) when high concentrations of this agent were used.

A comparison between the mass transfer coefficients obtained from C_t/C_o versus t (K_s) and from log C_t versus t taken from low isotherm values (K_s)_L indicates that both exhibit the same trend of change as a function of conditions of preparation.

[B] Intraparticle diffusion coefficients.

The following equation [Ozer, et al. (1997)] allows the determination of the intraparticle diffusion constant or the removal rate (D_s) .

$$q_m = D_s t^{1/2}$$

 D_s is obtained by plotting q_m versus $t^{1/2}$. Representative intraparticle diffusion plots are depicted in Fig. (4). The plots show satisfactory straight lines covering the early points followed by bending at higher values of $t^{1/2}$. The relative intraparticle diffusion constants, or the "removal rate" values are given in Table II. Generally, the removal rate was found to increase with H₃PO₄ concentration.

At high values of $t^{1/2}$, the "removal rate" becomes slower as it takes place through the intricate system of micropores consisting the internal texture of the activated carbons.

Apparently, the carbons characterized by high intraparticle diffusion constants (33,45,54 and 55) exhibit, likewise, highest mass transfer coefficient.

The assumption given by El-Geundi that the slope of the high time range of the log C - t plot **[El-Geundi (1990)]** could be correlated with the intraparticle diffusion, seems to be unjustifiable. Since upon comparison with the Ds values estimated from the usual C- $t^{1/2}$ plots, no correlation seems to hold between the two estimates. The slope of such plots only indicates an increase with respective increase in H₃PO₄ concentration, at every temperature of carbonization.

4- Correlation between colour removal capacity and conditions of preparation.

Figs.5 and 6 summarize representing results for the limiting dye removal capacity $\%R = C_0-C_e/C_0 \times 100$ shown by the carbons after 3 days of contact. At 20 and 30% H₃PO₄, a continuous increase in uptake is observed with increasing temperature of carbonization. Impregnation with either 40 or 50% H₃PO₄ exhibit an anomalous behaviour showing a minimum removal capacity by carbons prepared at 400°C. Probably the dehydrated H₃PO₄ acid deposited at such temperature inside the pores or at their entrance blocks a proportion of the internal adsorption space. Evidently Fig. 6 indicates that carbon prepared at 500°C is accompanied by a regular increase in percent dye removal as a function of acid concentration. Carbons obtained at 400°C show a drop in uptake at 40% H₃PO₄, at and beyond 200 mg.dm⁻³ of initial dye concentration. The highest removal capacity, in most cases ,appear at 30 % H₃PO₄.

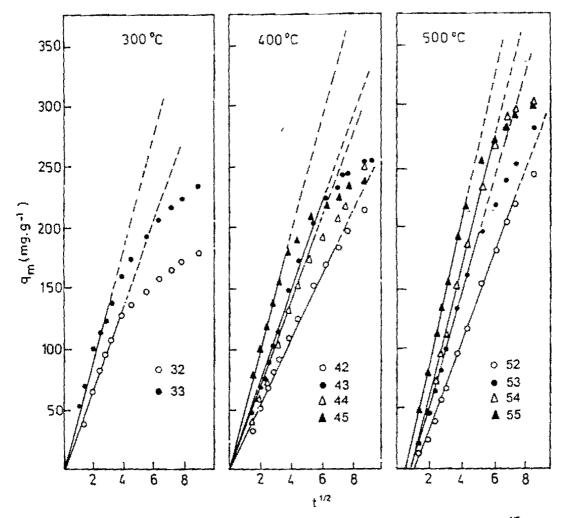


Fig. 4: Kinetic adsorption isotherms of MB plotted as amount adsorbed vs $t^{1/2}$

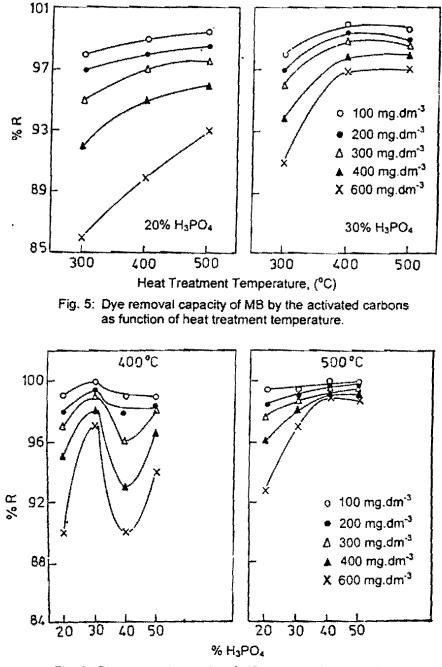


Fig. 6: Dye removal capacity of MB by the activated carbons as function of impregnating acid concentration.

Adsorption-Methylene Blue-Activated Carbon.

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After 3 days of contacting the present carbons, all samples took up $\ge 90\%$ of the dye present in solution at all initial concentrations (100-600 mg.dm⁻³). Only the carbon obtained under the mildest conditions (No. 32) shows lower uptake at higher concentrations. At a level of 100 mg. dm⁻³ all carbons remove more than 90 % of the dye after one day. Carbons 54 and 55 appear superior as adsorbing carbons since they remove more than 95 % of the dye at all concentration levels. Carbon 45 comes next as it is likewise effective up to 400 mg. dm⁻³. These deserve testing in column experiments to establish their suitability in typical dyecarbon system.

In general, H_3PO_4 intensively modifies the structure of the precursors [Rodriguez & Molina (1992) and Molina, et al. (1995)]. The action of phosphoric acid in the activation of carbon samples may be visualized as to take place in three stages in the process of preparation : impregnation, pyrolysis and leaching of the impregnant. During the course of soaking, the acid introduced into the lignocellulose produces chemical changes and structural alterations. These involve dehydration and redistribution of biopolymers possibly by partial dissolution in the acid solution, together with the cleavage of linkages between the lignin and cellulose, followed by recombination reactions in which larger structural units are formed.

In the pyrolysis stage, the introduced acid dehydrates gradually with continuation of the chemical changes involving crosslinking, water elimination and polymerization. The dehydrated impregnant reduces tar and volatile formation, inhibits shrinkage and collapse of the particles, and develops an extensive pore structure. Porosity development in this stage is ascribed to the phosphoric oxides formed at the carbonization temperatures, which acts as local oxidizing agents performing controlled gasification.

CONCLUSIONS

Apricot stone shells appear to be a good precursor for the production of useful activated carbon. Activation of the raw shells was preformed by pre-impregnation with H_3PO_4 (20-50 wt %) followed by carbonization at 300-500°C. Adsorption of methylene blue was slow and limiting capacities increase as function of both acid concentration and temperature. A good relationship seems to hold between the Freundlich isotherm parameters and both the mass transfer and the intraparticle diffusion coefficients. Carbons characterized by highest values in above

parameters show highest dye removal capacity, that attain beyond 95 % removal within initial concentrations up to 600 mg.dm⁻³. Equilibrium experiments were supported by kinetic data. Adsorption capacity of the carbons appear to be dependent on the extent of surface area and total adsorption space.

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امتزاز صبغة المثيلين الزرقاء على الكريون المنشط والمحضر من نوى المشمش

تم فى هذا البحث تحضير عينات من الكربون المنشط من نوى المشمش وتم اختبار قدراتهما الامستزازية لصمبغة المثيليمن الزرقاء تحت الظروف الحركية وكذلك فى ظروف الاتزان.

تبيسن توافق نتائج الامتزاز مع نموذج لنجمير وتم حساب السعات الامتزازية للطبقة الاحاديسة للعيسنات حيسث وجد ان هذه السعات الامتزازية تتراوح بين ٤٤٠،١٧ مجم/ جم كذلك تم الحصول على علاقات خطية مرضية عند تطبيق معادلة فرونيدلن على نتائج امتزاز صسبغة المثيليسن السزرقاء بالنمسبة لجميع العينات ويفيد ذلك فى أن امتزاز الصبغات على الكربون المنشط موات وواعد. هذا وقد أظهرت بعض العينات قابلية عالية وملاءمة واضحة للاستخدام فى اعمدة الأمتزاز .

هـذا وقـد تبين ان امتزاز صبغة المثيلين الزرقاء على عينات الكربون المنشط قيد الدراسـة يخضع لاليتى الانتقال الكتلى والانتشار بين ثنايا جسيمات الصلب الماز ويعزى ذلك الـــى زيادة تركيز الصبغة وعدم كفاية ظروف الخلط . ولقد اتضح ان بعض عينات الكربون المنشـط عالـية القـدرة الامتزازية يمكن ان تتتزع اكثر من ٩٣% من الصبغة من محاليلها المائية التى تحوى تركيز للصبغة يصل الى ٦٠٠ مجم/ لتر .