

PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON DERIVED FROM DATE PITS

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ABSTRACT

Date pits, an agricultural byproduct, was used as precursor in the preparation of activated carbon by chemical activation with $ZnCl_2$ at 773K. The influence of impregnation ratio on textural and chemical surface properties of the carbons obtained was studied. The textural properties of these carbons were characterized by adsorption of nitrogen at 77K. The nature of acidic surface groups of the prepared carbons was studied by FTIR spectroscopy and Boehm titration method. Equilibrium isotherms of methylene blue on the prepared carbons were obtained and the equilibrium data fitted well with the Langmuir model. The thermodynamic parameters related to the adsorption process, ΔH° , ΔG° and ΔS° were calculated and discussed. The results showed that the methylene blue adsorption capacity was positively correlated to the textural properties of the prepared carbons (BET area and total pore volume). The extent of the surface acidity is not a prominent factor in this particular study.

Key words:- Activated carbons , Preparation , Characterization

INTRODUCTION

Cationic dyes, known as basic dyes, are widely used in many industries such as textile, ceramic, paper printing and plastic in order to color their products. The wide use of dyes in these industries has led to a variety of environmental problems specially water pollution. The colored wastewater damages the aesthetic nature of water and reduces the light penetration through the water's surface and the photosynthetic activity of aquatic organisms due to the presence of metals, chlorides, etc. Due to the harmful impacts of such dyes on water, it is environmentally important to remove them from waste streams before discharge to public water sources. Several methods were designed to remove dye pollutants from wastewater such as coagulation and chemical oxidation, membrane separation process, electrochemical, filtration, reverse osmosis and aerobic and anaerobic microbial degradation [Crini 2006; Hartono *et al.* 2005 and Gurses *et al.* (2006)]. Among various treatment technologies, adsorption onto activated carbons is

generally regarded as an effective technique for the treatment of dye-containing wastewater because it has an excellent high surface area and high adsorption capacity for organic compounds [Cagnon *et al.* 2003 & Baklanova *et al.* (2003)]. Commercially available activated carbons are very expensive; for this reason many investigators have studied the feasibility of using many agricultural byproducts and wastes which are available at very little or no cost to prepare activated carbon such as olive stones [Baccar *et al.* 2009 and Martinez *et al.* (2006)], cherry stones [Duran *et al.* 2005 & Gomez-Serrano *et al.* (2002)], oil palm stones [Guo & Lua (2000)], apricot stones [Gergova & Eser (1996)], almond shells [Toles *et al.* 2000], sugar cane bagasse [Erlich *et al.* (2006)], cotton stalks [Putun *et al.* (2006)] and date stones [Haimour & Emeish (2006) and Girgis & El-Hendawy (2002)].

Date pits are among the most common agricultural byproducts available commercially in palm growing countries such as Egypt. Date pits constitute roughly 10% in weight of the fruit [Hmadaa *et al.* (2002)]. The excellent natural structure and low ash content of date pits [Barreveld (1993)] promotes the preparation of activated carbons with different surface properties from this precursor. In continuation of our previous work, on the preparation of activated carbon from low cost materials [Youssef *et al.* (1990); El-Nabarawy *et al.* (1997); Mostafa (1997) and Mohamed *et al.* (2006)], date pits based activated carbons were obtained via chemical activation by using $ZnCl_2$ as activating agent. The effect of impregnation ratio on textural and chemical surface properties of the products, thus obtained, are examined. The adsorption equilibrium of methylene blue was outlined and discussed.

EXPERIMENTAL

Preparation of activated carbons:

The starting date pits were obtained from local factory used to produce date pastry. First, the pits were washed several times with water and dried at 373 K for about 24 h. The dried pits were crushed and sieved, the fraction of particle sized between 0.5-2.0 mm being used for the preparation of activated carbon.

Non-activated carbon "CZ0" was obtained by carbonization the original date pits at 773 K in nitrogen atmosphere for 12 h in a horizontal furnace. Zinc chloride activated carbons CZI, CZII, CZIII, CZIV and CZV were prepared by soaking the dried date pits in zinc chloride solution where the ratios of $ZnCl_2$ to date pits were 1:3, 1:2, 1:1, 2:1, and 3:1 (w/w) respectively. The mixtures were left overnight at room temperature to incorporate all the chemical in the interior of the particles and then evaporated to dryness at 393K. The treated samples were then carbonized in nitrogen atmosphere at 773 K at a heating rate of 10°C/min. The samples were left at the final temperature for a holding time of 6 h before cooling to room temperature again. The carbonized zinc chloride treated products were washed with 0.1 M hydrochloric acid and then with distilled water until the resulting wash was Cl^- free. The prepared carbons were dried at 378K for 12 h under nitrogen flow rate and finally kept in tightly closed bottles.

Surface characterization of prepared carbons:

The specific surface area and pore structure characteristics of prepared carbons were determined by nitrogen adsorption at 77K using automatic volumetric sorption analyzer (Quantachrome Nova 3200). Prior to gas adsorption measurements, the sample was degassed at 473 K in vacuum conditions for a period of at least 6 h. Nitrogen adsorption were measured over a relative pressure (P/P_0) range from 10^{-5} –0.995.

The analysis of acidic surface functional groups was carried out following the method proposed by Boehm [Boehm (1994)]. The acidic functional groups were neutralized with 0.1 mole/l NaOH, Na₂CO₃ and NaHCO₃ solutions. According to the method, the amount of NaHCO₃ reacted is equivalent to the amount of carboxylic groups, while NaCO₃ reacted with both carboxylic and lactonic groups. The amount of NaOH reacted minus the Na₂CO₃ value gives the amount of phenols present on carbons.

Chemical characterization of the prepared carbons was also examined by using FTIR spectroscopy. The infrared transmission spectra were recorded with a Jasco 410 spectrometer from 400 to 4000 cm⁻¹ using the KBr wafer technique. Wafers were prepared from the mixture of 20mg of carbon sample and 200 mg of KBr. This mixture was compacted in a manual hydraulic press at 59 MPa. Carbon yield, ash content and slurry PH of the prepared carbons were also determined according to ASTM tests D2866-94 and D3838-80, respectively [ASTM (1996)].

Equilibrium adsorption of methylene blue (MB):

Analytical reagent grade methylene blue (MB, M.W= 319.86 g/mole) was obtained from BDH Co. Equilibrium studies were conducted in a set of 250 ml stoppered glass bottles containing 0.25 gm carbon sample and 100 ml of MB solutions with varies initial concentrations at pH= 6.5-7. The bottles were agitated occasionally in a shaker at 25± 0.1°C to attain equilibrium. Supernatant solution was filtered and analysed spectrophotometrically at 660 nm by the help of a Spectronic 20 spectrophotometer. The amount of MB adsorbed q_e "mg/g" was calculated from the mass balance equation:

$$q_e = (C_i - C_e)V / m \dots\dots\dots(1)$$

Where C_i , C_e are the initial and equilibrium concentrations of MB (mg/l) respectively, V , is the volume of the solution (l) and m , is the mass of carbon (g).

RESULTS AND DISCUSSION**Textural properties:**

Chemical activation of date pits with ZnCl₂ at 500°C produces carbon sample characterized by their high carbon yields (41.0–43.5% of raw material) and low ash contents (1.2–2.2%). Moreover, the pH values of aqueous suspensions of these carbons fall in the acidic range (4.8–6.0).

The nitrogen adsorption-desorption isotherms at 77K of the prepared carbons are shown in Figure (1). The desorption branches were found to lie on the adsorption branches as the adsorption data indicating the absence of hysteresis characteristic of mesoporosity or specific interaction [Youssef (1976)]. Figure (1) clearly shown that the isotherms are Langmuirian in shape being typical type 1 in the BDDT classification [Brunauer et al. (1940)], which is characteristic of adsorption on microporous adsorbent. The conventional BET equation [Brunauer et al. (1938)] was applied over the relative pressure range $0.02 \leq p/p_0 \leq 0.35$ to determine the monolayer capacity V_m and hence the specific BET areas ($S_{BET} \text{ m}^2/\text{g}$) by adopting the value of 0.16 nm^2 for the cross-sectional area of the nitrogen molecule at 77K. The calculated S_{BET} values for the investigated carbons are listed in column 2 of Table (1). The total pore volume, V_T (ml/g) expressed as the volume of liquid nitrogen adsorbed per gram carbon at relative pressure of $0.98 p/p_0$ is listed in column 3 of Table (1). The mean pore radius r (nm) may be calculated from the relationship [Podkosiely et al. (2002)].

$$r(\text{nm}) = 2V_T \times 10^3 / S_{BET} \dots\dots\dots(2)$$

The mean pore radii of the carbons are given in column 4 of Table (1).

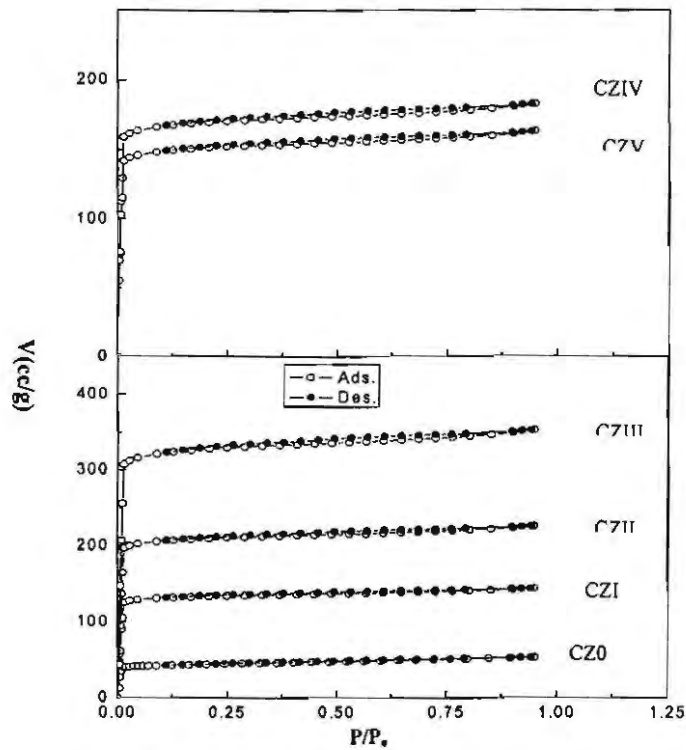


Fig. (1): Nitrogen adsorption-desorption isotherms at 77K of the prepared carbns.

Two other independent methods were also applied to analyze the nitrogen adsorption isotherms, i.e. the t -method [Lippens *et al.* (1964)] and the α_s -method [Sing (1968)]. In the first method the amount of nitrogen adsorbed V_1 (cc/g) is plotted against the multilayer thickness t (nm) as measured on standard non-porous material of comparable values of BET-C constant. The t -method allows the determination of specific surface area S^t as determined from the slope of the solid line passing through the origin Fig.(2). The micropore volume V_{mic}^t is obtained from the intersection of the dotted line passing through the plateau of the V_1 - t plot i.e., at zero thickness. The surface located in non-micropores S_n^t could also be calculated from the slope of the dotted line. S^t (m^2/g), V_{mic}^t (ml/g) and S_n^t (m^2/g) values are listed in columns 5,6 and 7 of Table (1) respectively.

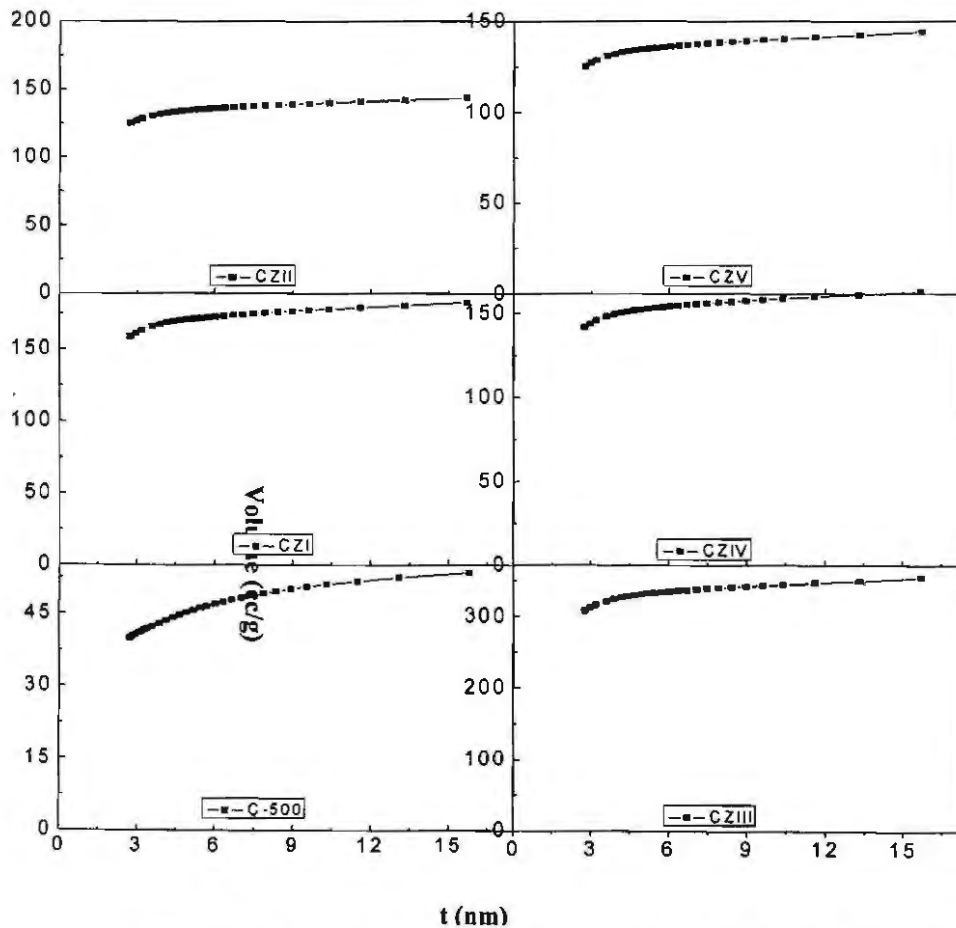


Fig. (2): V_1 - t of nitrogen adsorbed on non-activated and zinc chloride activated carbons.

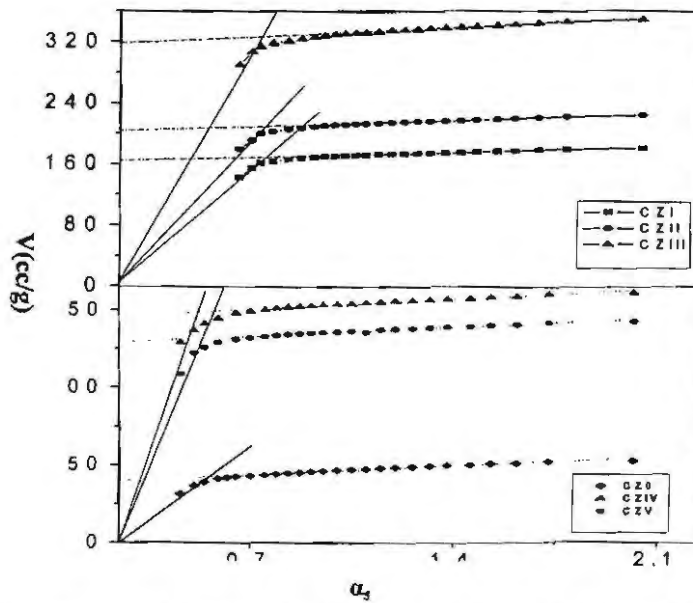


Fig. (3): α_s -plots of nitrogen adsorbed on non-activated and zinc-chloride activated carbons.

The second method plots the volume of nitrogen adsorbed versus the reduced isotherm (α_s) determined on a standard non-porous material of the same chemical composition, thus the adsorption data (V_a ml/g) were plotted against the α_s values reported by Sellen-Perez and Martin-Martinez [Sellen *et al.* (1991)] for the nitrogen adsorption on a non-porous carbon material Fig.(4). The linear section connecting to the origin corresponds to the specific surface area (S^a) and the linear relation at and beyond $\alpha_s \geq 1.0$ describes the non-microporous surface area (S^a_n) whereas its intercept with the V_a axis gives the micropore volume (V^a_{mic}). S^a (m²/g), V^a_{mic} (ml/g) and S^a_n (m²/g) are listed in columns 8-10 of Table (1) respectively.

Table (1): Textural characteristics of the prepared carbons.

Sample	S_{BET} (m ² /g)	V_T (ml/g)	r^- (Å)	S^t (m ² /g)	V^t_{mic} (ml/g)	S^a_n (m ² /g)	S^a (m ² /g)	V^a_{mic} (ml/g)	S^a_n (m ² /g)
CZ0	134.77	0.083	12.2	134	0.018	18	122.73	0.051	21.47
CZI	390.93	0.223	11.4	495.1	0.024	26.1	477.2	0.219	20.41
CZII	614.49	0.350	11.43	614.4	0.03	32.4	592.24	0.276	31.55
CZIII	959.06	0.547	11.85	958.55	.047	50.55	926.4	0.446	45.93
CZIV	495.75	0.283	11.38	472.3	0.022	23.32	426.4	0.199	49.66
CZV	442.33	0.252	11.36	411	0.02	20.0	377.2	0.166	57.55

It is evident from **Table (1)** that comparable areas and total pore volumes for each carbon sample are calculated using BET equation and t-method. These values are slightly higher than those calculated from α_s method. **Table (1)** reveals also that the surface area and the total pore volume of the resultant activated carbons increase with the increase of the amount of zinc chloride used in activation to reach a maximum at zinc chloride /carbon ratio 1:1 and then decreases with further increase in the ratio from 2:1 to 3:1 (i.e. samples CZIV and CZV). However, this was not accompanied by any appreciable change in pore dimension. Generally, all carbons measured mean average pore radius of 11Å. This obvious increase in both surface area and total pore volume of activated carbons may be taken as an evidence for the creation of new pores upon Zinc chloride activation up to zinc chloride /carbon ratio equal (1:1). Further increase in the amount of zinc chloride to higher activation ratios (i.e. CZIV and CZV) may causes shrinkage and /or collapse of the microporous structure of CZIV and CZV carbons which leads to reduce partially both of total surface area and total pore volume. Thus zinc chloride / carbon ratio 1:1 was recommended to prepare an effective activated carbon at the minimum consumption of zinc chloride. **Table (1)** reveals also that the prepared carbons exhibit high degree of microporosity as indicated by their higher content of microporosity ($V_n/V_T \geq 84\%$) and low mesoporosity ($V_n/V_T = 5-16\%$).

Surface chemistry of activated carbons:

The acidic surface functional groups are important characteristics of activated carbons, since these groups have significant applications on their behaviors as ion exchangers, adsorbents, catalysts and catalyst supports [Budinova *et al.* (2006)]. The FTIR spectra of the prepared carbons (not illustrated) show indications of various surface functional groups. According to these spectra, the studied carbons exhibited intensive peaks at $1720 - 1630 \text{ cm}^{-1}$, $1300 - 1000 \text{ cm}^{-1}$, $3000 - 2750 \text{ cm}^{-1}$ and at 3500 cm^{-1} which are identical to the presence of $\text{C}=\text{O}$ (in carboxylic, anhydride, lactone and ketone), $\text{C}=\text{O}$ (lactonic, ether, phenol), $\text{C}-\text{H}$ (alkane) and $\text{O}-\text{H}$ (hydroxyl) functional groups [Starsinic *et al.* 1983 and Fanning & Vannice (1993)]. The titration results of the surface functional groups of the prepared carbons are presented in **Table (2)**. Inspection of **Table (2)** reveals that all the carbons possess oxygen functionalities in the form non-carbonyl, i.e. carboxylic, lactonic and phenolic groups. The concentration of acidic groups in non-activated carbon (CZ0) is significantly lower when compared to those obtained from ZnCl_2 activation. The total concentration of these groups slightly increased with the increase of the amount of ZnCl_2 used in the activation method. **Table (2)** indicated also that progressive activation with ZnCl_2 decreases the concentration of lactonic groups, whereas their phenolic functionality content remains relatively constant. Carboxylic groups were found to increase with increasing severity of zinc chloride activation.

Table (2): Acidic surface functional groups of investigated carbons.

Sample	Carboxylic (meq./g)	Lactonic (meq./g)	Phenolic (meq./g)	Total acidity (meq./g)
CZO	0.08	0.06	0.04	0.18
CZI	0.16	0.28	0.22	0.66
CZII	0.24	0.22	0.26	0.72
CZIII	0.30	0.22	0.22	0.74
CZIV	0.36	0.18	0.24	0.78
CZV	0.42	0.12	0.26	0.86

Adsorption of methylene blue:

The adsorption isotherm indicates how the adsorbed molecules distribute between the liquid phase and the sorbent phase when the adsorption process reaches an equilibrium state. Figure (4) typically shows the adsorption isotherms of "MB" at 298K on the prepared carbons where the amount of "MB" dye adsorbed (q_e , mg/g) was plotted against the equilibrium concentration (C_e , mg/l). The analysis of the isotherm data by fitting them to different isotherm models is an important step to describe how solute molecules interact with adsorbents and is critical in optimizing the use of adsorbents [El-Guendi (1991)]. Several models have been published in the literature to describe the experimental data of adsorption isotherms. In the present study Langmuir model was used to describe the relationship between the amount of dye adsorbed (q_e , mg/g) and its equilibrium concentration (C_e , mg/l). The model assumes uniform energy of adsorption onto the surface and the uptake occurs on homogeneous surface by monolayer of one molecule thick without interaction between sorbed molecules. The linear form of Langmuir isotherm is represented by the following equation [Langmuir (1918)]:

$$\frac{C_e}{Q_o} + \frac{1}{bQ_o} = \frac{C_e}{q_o} \dots\dots(3)$$

Where Q_o and b are Langmuir constants related to maximum adsorption capacity (monolayer capacity) and energy of adsorption, respectively. Satisfactory straight lines were obtained by plotting C_e/q_e vs C_e Fig.(5) which indicated the homogeneous nature of date pits carbon surface. Values of Q_o and b were calculated from the intercept and slope of the linear plots and are listed in Table (3).

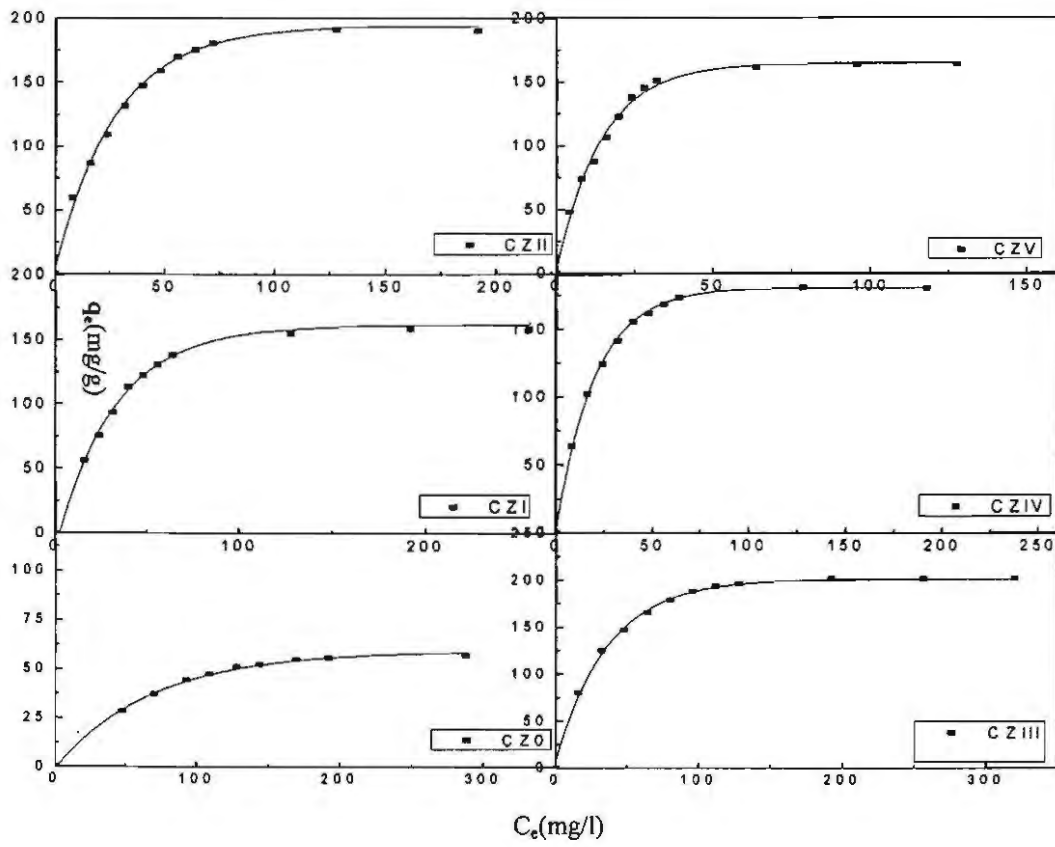


Fig.(4): Isotherms for the adsorption of methylene blue at 298K on the investigated carbons.

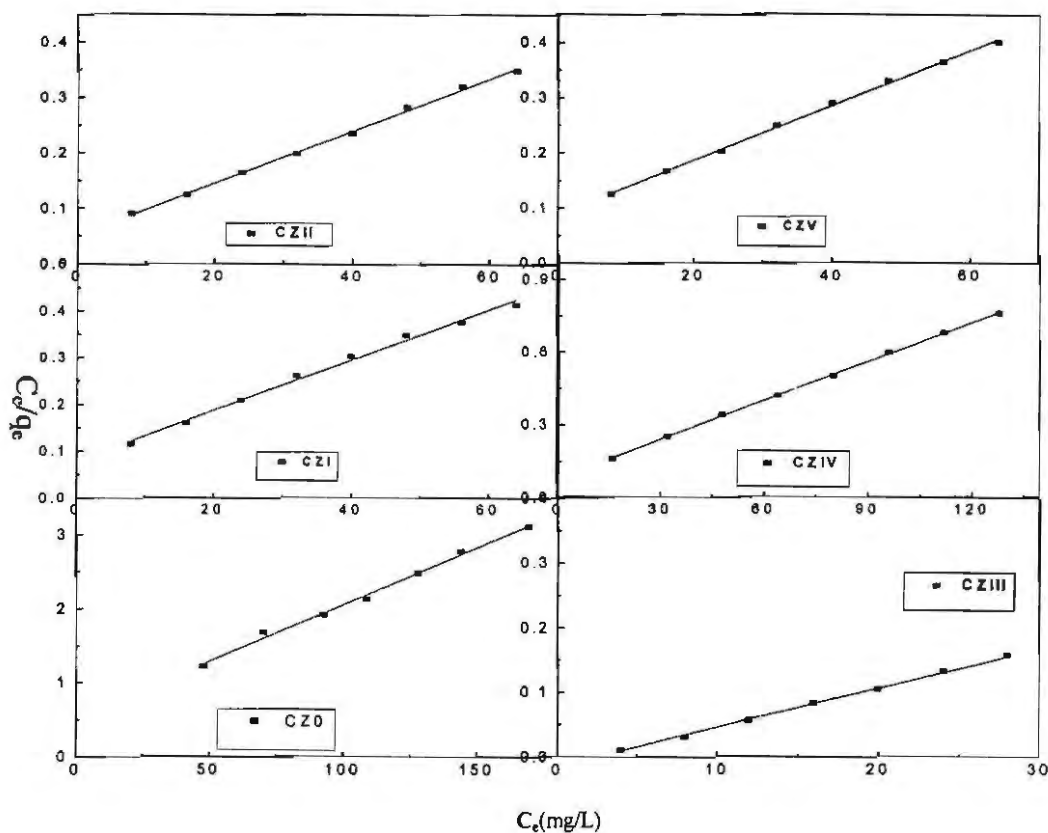


Fig. (5): Langmuir plots for the adsorption of methylene blue on the investigated carbons.

As seen from Table (3), the adsorption capacity of the prepared carbons increases in the order $CZI < CZII < CZIV < CZII < CZIII$, i.e. with the increase of both surface area and total pore volume of the carbon samples. The results indicate also that the amount of methylene blue adsorbed seems to be independent of the total acidity of the investigated carbons. Two evidences support this conclusion:

- (i) CZII, CZIII and CZIV carbons which measure comparable values of total acidities showed different "MB" adsorption capacities.
- (ii) CZV carbon of relatively higher total acidity shows lower "MB" adsorption capacity as compared with the previously mentioned carbons.

The thermodynamic parameters, ΔH° , ΔG° and ΔS° for the adsorption of methylene blue by the prepared carbons were calculated from the following equations:

$$\Delta G^\circ = -RT \ln K_a \dots\dots\dots(4)$$

$$\Delta S^\circ = \frac{\Delta H^\circ - \Delta G^\circ}{T} \dots\dots\dots(5)$$

The equilibrium constant K_a of the adsorption process, which is equal to the product $Q_o \cdot b$ as graphically determined from the linear form of the langmiur model, was measured at an additional temperature namely 315K. The values of K_a at 298K and 315K were used to calculate the enthalpy change " ΔH° " by using Clausius-Clapeyron equation [Gu *et al.*(1994)].

The free energy change ΔG° and entropy change ΔS° were determined from equations (4) and (5). The calculated thermodynamic parameters are listed in Table (3). The negative values of the free energy change " ΔG° " confirm the spontaneous nature of the adsorption process. The negative values of the enthalpy change " ΔH° " indicates that the adsorption process is exothermic and these values also prove that the adsorption follows a physisorption mechanism in nature. The positive values of entropy change; " ΔS° " corresponds to an increase in the degree of freedom of the adsorbed species; the relatively lower values of ΔS° obtained for activated carbons indicate more ordered adsorbed molecules than those adsorbed on non- activated carbon.

Table (3): Langmuir constants and thermodynamic parameters for the adsorption of methylene blue on the prepared carbons.

$-\Delta H^\circ$ (KJ mol ⁻¹)	ΔS° (J mol ⁻¹)		$-\Delta G^\circ$ (KJ mol ⁻¹)		K_a		b (L mg ⁻¹)		Q_o (mg g ⁻¹)		Sample
	315K	289K	315K	298K	315K	298K	315K	298K	315K	298K	
27.55	81.0	80.1	51.06	51.39	1.004	1	0.045	0.022	22.0	54.0	CZO
21.3	20.1	22.2	27.88	27.54	12.77	14.28	0.114	0.094	112.0	152.0	CZI
24.25	15.2	15.7	29.21	28.93	14.4	16.34	0.106	0.086	136.0	190.0	CZII
22.4	21.7	21.7	29.26	28.89	14.48	16.28	0.102	0.081	142.0	201.0	CZIII
23.66	10.0	9.0	27.23	26.4	12.03	12.8	0.094	0.072	128.0	178.0	CZIV
18.15	23.1	23.0	25.6	25.21	10.38	11.41	0.088	0.07	118.0	163.0	CZV

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تحضير و توصيف عينات من الكربون المنشط من نوى البلح

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يهدف البحث الي تحضير عينات من الكربون المنشط من نوي البلح الذي يتوافر في جمهورية مصر العربية كأحد المخلفات الزراعية وقد استخدمت طريقة التنشيط الكيميائي باستخدام كلوريد الزنك عند درجة حرارة ٧٧٣ كلفن في مجال حامل باستخدام غاز النيتروجين وقد تم تحضير خمس عينات من الكربون المنشط باستخدام نسب مختلفة من كلوريد الزنك: نوي البلح الجاف (٣:١، ٢:١، ١:٣، ١:٢، ١:١) بالإضافة للطبقة المكونة عند درجة حرارة ٧٧٣ كلفن.

تم دراسة الخواص السطحية للعينات المحضرة باستخدام تقنية امتزاز غاز النيتروجين عند درجة حرارة ٧٧ كلفن، بينما استخدمت تقنية FTIR لتحديد كيميائية سطح العينات المحضرة بالإضافة لاستخدام طريقة المعايرة ل Boehm لتقدير تركيزات المجموعات الحمضية على سطح الكربون. اشتملت الدراسة أيضا علي تعيين قدرتها علي امتزاز صبغة المثلين الأزرق من محاليلها المائية.

هذا وقد نوقشت نتائج امتزاز غاز النيتروجين عند درجة ٧٧ كلفن بطرق حسابية مختلفة من خلال تطبيق معادلة BET وطريقة α وطريقة V_t-t وأوضحت النتائج أن الخواص السطحية للعينات (مساحة السطح - حجم المسام الكلي - امتصاص المسام) تعتمد علي نسبة كلوريد الزنك : نوي البلح وأن العينات تحتوي بشكل كبير علي مسام من النوع المايكرووالدقيق وأن القدرة الامتزازية للعينات تجاه امتزاز صبغة المثلين الأزرق تزداد بزيادة كلا من المساحة السطحية وحجم المسام الكلي ولا تعتمد علي حمضية سطح هذه العينات .

هذا وقد تم تعيين قيم دوال الديناميكا الحرارية (ΔH° , ΔG° , ΔS°) لعملية امتزاز صبغة المثلين الأزرق علي العينات المحضرة حيث بينت هذه القيم الطبيعية المتجانسة لسطح عينات الكربون وأن العملية الامتزازية من النوع الطارد للحرارة .