REMOVAL OF PENTACHLOROPHENOL FROM AQUEOUS SOLUTIONS BY ADSORPTION ON DATE-PITS AVTIVATED CARBON

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ABSTRACT

Adsorbable organic halide (AOX) compounds known with their toxic effect, produced as by-product for drinking water disinfection. The present work studied the removal of pentachlorophenol (PCP) as a model AOX compound in water. The use of a locally prepared date-pit activated carbon (DPAC) as adsorbent of pentachlorophenol from aqueous solutions was examined over pH range (2-11) and at different temperatures 25, 35, 45 and 55°C. Langmuir, Frendulich, Temkin and Redlich-Peterson isotherm models were tested for modeling the adsorption isotherm at equilibrium. date-pit activated carbon examined by Elemental Analysis and FTIR.

Keywords: Drinking water treatment; Pentachlorophenol; Date-pits activated carbon.

INTRODUCTION

Chlorine has been used for drinking water disinfection since the beginning of the last century and it still remains as the major chemical for this purpose around the world. It is introduced in water as a gas or as the sodium Vol.33, No.2, June, 2016 143

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or calcium hypochlorite (Praus, 2003). Chlorination generates a wide variety of chlorinated organic compounds, collectively called disinfection by products DBPs, mainly halogenated compounds that originate by the reaction of free chlorine and/or bromine with precursors or natural organic matters (NOM). Adsorbable organic halides (AOX), represents the organically bound halogens - chlorine, bromine and iodine (but not fluorine) contained in a sample which can be adsorbed on activated carbon. Chlorophenolic compounds including pentachlorophenol (PCP) constitute a major component of AOX (Ghosh *et al.*, 2011). These compounds causes liver and kidney damage, reduce gonad development and known to induce hormonal disorders in many aquatic species. (Sumathi and Hung 2004; Savant et al., 2006). The by-product control strategy in water treatment plants includes removing the DBP precursors, changing disinfectants, and/or removing DBPs themselves (Praus, 2003) and for the removal of AOXs, adsorption is regarded as a promising method. The preparing of activated carbon utilizing natural wastes adsorbents has an environmental and commercials advantage over other removal methods. Date-pits are common agricultural waste available in large quantity in the palm-growing countries such as the Kuwait. Banat et al. (2002) used raw date-pits and date-pit activated carbons for the adsorption of zinc and copper ions from water. Activated date-pit carbons were also used for the adsorption of cadmium ions (Banat et al., 2003). Date -pit activated carbon also used as adsorbent of Aluminium (Al-Muhtaseb et al. 2008), Cobalt (Al-Jlil, 2012) Phenolic compounds and Methylene Blue (Abdulkarim et al., 2002), organic compounds from water (Merzougui and Addoun, 2008),

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heavy metal (Esmael *et al.*, 2014) while Abdel-Ghani *et al.*, (2015) used activated carbons prepared from corn wastes to remove PCP adsorption from aqueous solution.

The focus of the present work is to evaluate the adsorption potential of date-pits based activated carbon in removing of Pentachlorophenol as a model of AOXs from aqueous solutions at different temperatures and pH values. However, to the best of the authors' knowledge, there is no published literature for the adsorption of PCP on date-pit activated carbon.

EXPERIMENTAL

1 Materials and Methods:

1.1 Pentachlorophenol solutions:

Stock solutions were prepared by dissolving pentachlorophenol (Sigma-Aldrich Chemical Company 99.0%) in deionized water followed by serial dilutions.

1.2 Preparation of activated carbon

The preparation of activated carbon from the palm-date pits involves three steps: boiling, soaking and a combined step of carbonization and activation of pits (Al-Attas, 2003 and Esmael et al., 2014). In the first step, the pits were boiled for about three hours then; the boiled pits were dried in an drying oven at 110°C for 24 hours and crushed. The second step is soaking. The solution commonly used in the chemical treatment process of producing activated carbon is phosphoric Acid (85% H₃PO₄) at 25 °C and for 12 hours, then dried in an oven at 120°C for one hour. The final step is the process of carbonization and activation. The date pits were placed on a

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metallic plate and subjected to an average temperature of 400°C for 2 hours under constant flow of nitrogen. The product was put in a flask and distilled hot water was added. Then, the pits were washed with distilled boiling water until the pH was achieved (6.7), and then the product was dried in an drying oven at 120°C overnight in order to remove any undesired moisture within the particles.

2 Characterization of Activated Carbon:

2.1 Surface Analysis:

The specific surface area and total pore volume were measured using a multipurpose apparatus Nova 2000 analyzer, Quantachrome Instruments (Japan) the method employed was adsorption of high-purity nitrogen by the Brunauer–Emmett–Teller (BET) method.

2.2 FTIR:

Fourier Transform Infrared Spectroscopy (FTIR) analysis of about 1 mg of DPAC were carried. The samples were grinded and milled with 100 mg KBr to form a fine powder. This powder was then compressed into a thin pellet under7 tons for 5 minutes. The sample was then analyzed using Shimadzu 6100A spectrometer and the spectrum was recorded in a spectral range of 400-4000 cm⁻¹.

3 AOXs Analysis:

AOX analyzer multi X2000-Germany was used for the determination of AOX in the collected samples according to Method No. ISO 9562:2004.

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4 Effect of solution pH:

The effect of pH on adsorption capacity of PCP onto DPAC, was evaluated. Batch experiments were performed at pH 2,4.7, 9 and 11 using an adsorbent dose of 0.1 g.dm⁻³ and PCP solutions volume 0.05 dm³ at room temperature for 240 min, where the initial concentration of PCP is 300 μ g. dm⁻³. The pH of the initial solution was adjusted by using 0.1 M HCl and 0.1 M NaOH

5 Effect of Contact Time:

Effect of contact time on the adsorption capacity evaluated by contacting a constant mass (0.1g) of date-pit activated carbon with a fixed volume (50ml) of two concentrations (500 and 1000 μ g. dm⁻³) for different intervals of contact time until equilibrium was reached

6 Adsorption Isotherm:

Batch adsorption experiments were carried out using bottle-point method (El-Geundi, 1991). In this method stock solution of PCP ($5000 \ \mu g.dm^{-3}$) was subsequently diluted to the required initial concentrations over wide range ($50-2750 \ \mu g.dm^{-3}$). Adsorption capacity of the activated carbon towards PCP was determined by contacting a constant mass (0.1g) of date-pit activated carbon with a fixed volume ($50 \ m$) of different initial concentrations of PCP solution in sealed glass bottles. The experiments were done at different temperatures: 25,35,45 and 55° C. After shaking in isothermal water-bath shaker for 4 h ($240 \ min$) until equilibrium was reached, contents were centrifuged. Supernatant samples saved in amber glass wide-mouth screw cap bottles and zero headspace, stored cool at 0 to 4° C and maintained in this

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condition away from light until analysis. The amount of PCP adsorbed onto DPAC, $q_e(\mu g.g^{-1})$, was calculated by the following equation:

$$qe = \frac{(Co - Ce)V}{m}$$
(1)

Where C_o and C_e are the initial and equilibrium concentrations of PCP solution (ppb), respectively. V is the total volume of the solution (1), and m is the mass of DPAC used (g).

The adsorption equilibrium data were fitted into four different isotherm models to determine the most suitable model to represent the adsorption process. The isotherms used are the Langmuir, the Freundlich, the Temkin, and the Redlich-Peterson isotherm.

6.1. Langmuir isotherm:

The Langmuir isotherm is valid for monolayer adsorption on a homogenous adsorbent surface containing a finite number of identical site and no interaction between adsorbent molecules. The Langmuir expression may be converted into a linear form:

$$\frac{C_e}{q_e} = \frac{a_L}{K_L}C_e + \frac{1}{K_L}$$
(2)

where Ce is the equilibrium concentration $(\mu g/dm^{-3})$; qe is the adsorption capacity at equilibrium $(\mu g/g)$; and K_L is the sorption equilibrium constant (dm^{-3}/g) .The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless equilibrium parameter (R_L), which is defined by:

$$R_{L} = \frac{1}{1 + a_{L}.C_{0}}$$
(3)

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Where R_L is a dimensionless constant separation factor, Since (C_{ref}) is the highest liquid-phase concentration (µg.dm⁻³) encountered (i.e. $C_{ref} = C_0$). and a_L is the Langmuir adsorption constant (dm³.g⁻¹).

6.2. Freundlich Isotherm:

Freundlich isotherm in the other hand assumes heterogeneous surface energies, in which the energy term in Langmuir equation varies as a function of the surface coverage (Weber and Chakravort, 1974). The experimental equilibrium data has been analyzed using the Freundlich isotherm as given by linnera equation (9).

$$\log q_e = \log K_F + (1/n) \log C_e \tag{4}$$

Where (K_F and n) are the Freundlich constants. The K_F value is related to the adsorption capacity; while n value is related to the adsorption intensity. The magnitude of exponent (n) gives an indication of the favorability and capacity of the adsorbent/adsorbate system. Values of (n) greater than 1 represent favorable adsorption according to (Treybal, 1985).

6.3. Temkin Isotherm:

Temkin model considered the effects of indirect adsorbate/adsorbate interactions on adsorption isotherms. The heat of adsorption of all molecules in the layer would decrease linearly with coverage due to adsorbate/adsorbate interactions. The Temkin isotherm has been used in the form as follows (Temkin, 1941):

$$q_e = B_T \ln A_T + B_T \ln(C_e) \tag{5}$$

Where B = RT/b, T is the absolute temperature (K), R is the universal gas constant (8.314 J/gmol. K), A_T is the equilibrium binding constant corresponding to the maximum binding energy (dm³.µg⁻¹), b_T is Temkin Vol.33, No.2, June, 2016 149 isotherm constant (J.g-mol⁻¹) and the constant B_T is related to the heat of adsorption (dimensionless).

6.4. Redlich-Peterson Isotherm:

The Redlich–Peterson isotherm contains three parameters and involves the features of both the Langmuir and the Freundlich isotherms. The linear form is shown in equation (6) from which the constants (K_{RP}, a_{RP} and β) which characterize the isotherm, can be determined as:

$$\log\left(\frac{K_{RP}C_{e}}{q_{e}}-1\right) = \log a_{RP} + \beta \log C_{e}$$
(6)

Where $\beta \leq 1$, K_{RP} is the modified Langmuir constant (dm³.g⁻¹), a_{RP} (dm³.µg⁻¹) and β are constants. Plotting (log [($K_{RP}C_e/q_e$)⁻¹]) against (log C_e) yields a straight line of slope; β and intercept = log a_{RP} . The isotherm parameters are listed with the corresponding values of R² and ARE in Table (3).

7 Simulation Results and Correlations:

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Using the estimated parameters of the different models; Langmuir, Freundlich, Temkin and Redlich-Peterson, the theoretical isotherm curves were predicted using known values of C_e . In order to quantitatively compare the applicability of different models, the average relative error (ARE) was calculated using equation (7) (Kapoor, and Yang, 1989):

$$ARE = \frac{100}{N} \left[\sum_{i=1}^{N} \left| \frac{q_{e,cal} - q_{e,exp}}{q_{e,exp}} \right| \right]$$
(7)

Where N is the number of data points, $q_{e,exp}$ and $q_{e,cal}$ (µg.g⁻¹) are the experimental and the calculated values of the equilibrium adsorbate solid concentration in the solid phase, respectively. The values of ARE a measure

of the fitting of the data to an isotherm equation, small values of ARE would indicate a perfect fit.

RESULTS AND DISSCUSSIONS

1 Characterization of prepared activated carbon:

1.1 Surface Analysis:

The BET surface area of the prepared activated carbon was found to be 825 m_2/g . The density and surface properties of date-pits activated carbon (DPAC) are shown in Table (1).

Solid-phase density (ρ_s) (g/cm ³)	(1.04)±0.04
Particle density (ρ_p) (g/cm ³)	$(0.50) \pm 0.04$
Porosity (ε_p)	0.52±0.01
Specific Surface area S_{BET} (m ² /g)	825±2
V_{total} (cm ³ /g)	0.816
Particle diameter (d _p)	180-250µm

Table (1): Physical properties of date-pits activated carbon.

1.2 FTIR Analysis:

FTIR spectra are a useful tool to identify functional groups in a molecule, as each specific chemical bond often has a unique energy adsorption band, and can obtain structural and bond information on a complex to study the strength and the fraction of hydrogen bonding and miscibility (Zhou *et al.*, 2004). The spectrum Figure (1) shows a strong and broad adsorption peak appeared at at 3433cm⁻¹ indicated the presence of the O–H group of phenol. The methylene group (aliphatic) is detected by C–H stretching at a wave number of 2919 and 2852 cm⁻¹. The aldehyde group of –O–CH₃ is found around 2853 cm⁻¹. IR band around 1445 cm⁻¹ indicate to stretching C=O of

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carboxyl or carbonyl groups, a strong band at about 1079 cm⁻¹ corresponding to C–O stretching in acids, alcohols, phenols, ethers and esters and (Lua, and Yang, 2005) and (Mahmoudi et al., 2014).



Figure (1): FTIR for Date pits activated carbon (DPAC)

2. Effect of Contact Time:

The adsorption capacity and required contact time are two of the most important parameters to understand in an adsorption process (Ho and McKay, 1998). It is noted from Figure (2) that the removal of PCP species is rapid in the initial stages of contact time and gradually decreases with lapse of time until saturation and attains equilibrium in 120 minutes for initial concentrations 500 and 1000 (μ g.dm⁻³) of PCP solutions at temperature 25°C, however, equilibrium experiments were run for 240 minutes to insure that the adsorption process achieved the state of equilibrium.

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Figure (2): Equilibrium Time for the Adsorption of PCP onto DPAC at Different Initial Concentrations at 25±°C

3. Effect of pH:

The effect of pH on the adsorption of DPAC for pH ranging between 2 and 11 is illustrated in Figure (3). The uptake amounts of PCP adsorbed qe (μ g/g), at acidic pH remained constant or decreased slightly with the pH, however at (pH=9) a sharp decrease in adsorption capacity is observed. These results are in accordance with results obtained by Ali *et al.*, (2008) for adsorption of para-chlorophenol onto (Date pits-clay) carbon composite from aqueous media; also with Abdel-Ghani *et al.*,(2015), for adsorption of pentachlorophenol onto activated carbon prepared from corn wastes from aqueous media. According to Abdel-Ghani *et al.*, (2015), PCP exists entirely in the anionic form at neutral and basic pH. Therefore, electrostatic repulsion between the negatively charged adsorbent surface and the anionic PCP will take place leading to lower adsorption.



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Figure (3): Effect of pH on the adsorption capacity of PCP onto date -pit activated carbon at Temp.=25 °C.

4. Equilibrium modeling:

The adsorption data have been subjected to different four adsorption isotherms, namely Langmuir, Freundlich, Temkin and Redlich-Peterson models at different temperature 25, 35, 45 and 55°C. Figures 4.5,6 and 7 show the linearization plots of the applied isotherm models at different temperatures. The results suggest the applicability of the Langmuir isotherm and demonstrate monolayer coverage of the adsorbate on the outer surface of the adsorbent. The isotherm models parameters obtained for adsorption of PCP on the DPAC at different temperatures are listed in Table 2. The adsorption process was favorable as Langmuir separation factor, R_L was $0 < R_L < 1$ and supported by *n* value of Freundlich which were more than one. A value for *n* over one indicates a Langmuir-type isotherm because it becomes more and more difficult to adsorb additional adsorbate molecules at higher and higher adsorbate concentrations (Konstantinos *et al.*, 2007) 154 Vol.33, No.2, June, 2016





Figure (4): Langmuir plot for the adsorption of PCP onto DPAC at different temperatures.





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Figure (6): Tekmin plot for the adsorption of PCP onto date-pits activated carbon.



Figure (7): Redlich- Petroson plot for the adsorption of PCP onto date-pits activated carbon.

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 Table 2: Estimated Langmuir, Freundlich and Temkin isotherm models

 parameters for adsorption of PCP onto DPAC at different

 temperatures.

Isotherm Model	Parameters	Temp. (°C)			
Langmuir		25 °C	35 °C	45 °C	55 °C
	K_L	2.7548	1.0025	0.7912	0.6788
	$a_{ m L}$	0.0055	0.00260	0.00221	0.00203
	R _L	0.1337	0.2174	0.2383	0.2491
Frendulich	K _f	16.48	4.607	3.490	2.919
	n	2.163	1.690	1.631	1.598
Temkin	b _T	32.685	34.544	34.544	41.161
	A _T	0.1379	0.0382	0.0319	0.0287
Redlich-Peterson	K _{RP}	8.985	1.209	0.899	0.745
	$a_{\rm RP}$	0.03617	0.00466	0.00380	0.00264
	β	0.9709	0.9691	0.9602	0.9912

5 Simulation and Correlation

Figure 4 show the graphical comparison between the experimental and predicted data for PCP onto DPAC using the four applied equilibrium models equations (Langmuir, the Freundlich, the Temkin, and the Redlich-Peterson isotherm) and the estimated values of their parameters. So it could be concluded that the Langmuir model be best to fit the experimental data according to graphical representations and error function values.

The applicability of the isotherm models to fit the adsorption data was compared by judging the correlation coefficients, R^2 and average relative error values. The closer the R^2 value to unity and the smaller ARE value, the better the fit. Based on the tabulated values of correlation coefficient (R^2) and Average relative error (ARE), it can be concluded that the adsorption of PCP on DPAC was demonstrated well by Langmuir isotherm model Vol.33, No.2, June, 2016 157 compared to other models. The correlation coefficient, R^2 for this model is 0.987 and ARE value is 21.424.

 Table (3): Simulation and correlation values for the Adsorption of PCP onto

 DPAC using Different Models.

Isotherm modelT = 25 °C	ARE	\mathbf{R}^2
Langmuir	21.424	0.9876
Freundlich	84.194	0.9595
Temkin	240.21	0.8860
Redlich-Peterson	39.307	0.8080



Figure (8): Adsorption isotherm of PCP onto date -pit activated carbon at $25\pm1^{\circ}$ C.

6 Thermodynamics study:

Results in Table (2) reveled that the value of K_L decrease with increasing temperature suggesting that the adsorption of PCP on DPAC is exothermic in nature. The thermodynamic parameters that must be considered to determine the process are changes in standard enthalpy (ΔH°), standard entropy (ΔS°) 158 Vol.33, No.2, June, 2016 and standard Gibbs free energy (ΔG°) due to transfer of unit mole of solute from solution onto the solid–liquid interface. The Gibbs free energy change of adsorption is defined as:

$$\Delta G^{o} = -RT \ln K_{L} \tag{8}$$

Where (K_L) is Langmuir equilibrium constant, (R) is the universal gas constant (8.314 J.g-mol⁻¹.K⁻¹) and (T) is the absolute temperature (K). The values of (Δ H^o) and (Δ S^o) was computed using Van't Hoff equation (Cheung et al., 2009) :

$$\ln K_{L} = \frac{\Delta S^{o}}{R} - \frac{\Delta H^{o}}{RT}$$
(9)

A plot of (ln K_L) versus (1/T) (Figure 5) should produce straight line with slope equals to $-\Delta H^{\circ}/RT$ and intercept equals to $\Delta S^{\circ}/R$ The calculated values of (ΔH°) and (ΔS°) and (ΔG°) are listed in Table (3).



Figure (9): Plot of K_L against 1/T for adsorption of PCP onto DPAC

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Temp. (°C)	ΔG^{O} KJ.mol ⁻¹	$\Delta H^{O} k$ $J.mol^{l}$	ΔS^{O} J.mol ⁻¹ .K ⁻¹	
25	-2.510		-115.947	
35	-0.0064	26 500		
45	0.6192	-30300		
55	1.0565			

Table (3): Thermodynamic parameters of PCP adsorption onto DPAC

The standard enthalpy (ΔH°) and entropy (ΔS°) changes of adsorption determined from equation (9) were found to be -36.500 (kJ.mol⁻¹) and - 115.947 (J. mol⁻¹.K⁻¹), respectively. The magnitude of (ΔH°) for physical adsorption ranges from 4 to 40 (kJ.g-mol⁻¹), compared to that of chemical adsorption ranging from 40 to 800 (kJ.g-mol⁻¹) (Crini and Badot, 2008). The negative value of (ΔH°) for PCP onto DPAC system indicates the exothermic nature of the process.

The negative value of ΔS° , which indicate that there is a decrease in state of disorderness in the molecules during process, which in turn is due to the binding of molecules with adsorbent surface (Gupta et al., 2006).

CONCLUSIONS

This work investigated the equilibrium adsorption of pentachlorophenol (PCP) onto prepared date-pit activated carbon (DPAC) at different temperatures. Langmuir isotherm was found to have the best fit to the experimental data over the whole concentration range with the lower average relative error (ARE) and higher correlation coefficient (\mathbb{R}^2) values, 21.424 and 0.9876 respectively. Thermodynamic parameters indicate that the adsorption of PCP on DPAC is exothermic process. The adsorption capacity Vol.33, No.2, June, 2016

of PCP remained constant or decreased slightly at pH (2-9), followed by a sharp decrease by further increasing in pH.

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إزالة ماحة حماسي كلورفينول من المحاليل المائية بالامتزاز علي الفحو المنشط المحضر من نوى التمر

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المستخلص

تعرف مركبات الهاليدات العضوية القابلة للامتزاز بأنها مواد سامة نتتج كنواتج ثانوية لتعقيم مياه الشرب باستخدام الكلور، ويدرس هذا البحث التخلص من مركب خماسي كلوروفينول من المحاليل المائية كنموذج لهذه المركبات، باستخدام فحم منشط محضر من مخلف زراعي وهو نوى التمر. وقد تم فحص الفحم المنشط المحضر باستخدام التحليل الكيميائي للعناصر، وتقنية تحت الحمراء(FTIR). تم فحص الفحم الفحم المنشط المحضر باستخدام التحليل الكيميائي للعناصر، وتقنية تحت الحمراء(FTIR). وتم دراسة كفاءة الامتزاز عند تغيير الأس الهيدروجيني (2–11) عند درجة حرارة ٢٥ درجة مئوية . وتم دراسة كفاءة الامتزاز عند تغيير الأس الهيدروجيني (2–11) عند درجة مدارة ٢٥ درجة مئوية . كما تم دراسة الايزويثيرم عند درجات حرارة مختافة ٢٥، ٤٥، ٥٥، ٥٥ درجة مئوية. تم دراسة النمذجة لنتائج الأيزويثيرم باستخدام نماذج لانجمير وتمكين وفريندوليش وريدريك بيترسون مع حساب متوسط الخطأ النسبي، ووجد أن نموذج لانجمير هو الأكثر تطابقاً مع النتائج المعملية مع أعلي معامل ارتباط وأصغر متوسط خطأ نسبي، وعليه فإن النتائج أثبتت قدرة الفحم المنشط المحضر من نوى التمر علي التمار عائم معامل ارتباط الخطأ النسبي، ووجد أن نموذج لانجمير هو الأكثر تطابقاً مع النتائج المعملية مع أعلي معامل ارتباط المخار النحائية المن المركبات من موليه فإن النتائج أثبتت قدرة الفحم المنشط المحضر من نوى التمر علي الخطأ النسبي، ووجد أن نموذج لانجمير هو الأكثر تطابقاً مع النتائج المعملية مع أعلي معامل ارتباط المخطأ النسبي وحد أن نموذج لانجمير هو الأكثر تطابقاً مع النتائج المعملية مع أعلي معامل ارتباط المخلي الخطأ النسبي وعليه فإن النتائج أثبتت قدرة الفحم المنشط المحضر من نوى التمر علي وأصغر مات مالي المائية بكفاءة ويمكن استخدامها بدلا من المواد الأكثر تطابقاً مع المائية المعملية مع أعلي معامل ارتباط وأصغر منوالي مالي المائية بكفاءة ويمكن المنظر المحضر المعملية مع أعلي معامل ارتباط وأصغر ماتوسط خطأ نسبي وعليه فإن النتائج أثبتت قدرة الفحم المنشط المحضر مان نوى التمر علي وأصغر ماتوسط خطأ نسبي وعليه فإن الندائية بكفاءة ويمكن استخدامها بدلا من المواد الأكثر المائية المائية المائي المائية المائيم المولية المائية المائي المائية ال

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