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Adsorption of herbicide diuron from aqueous solution using cassava peels activated carbon: Equilibrium and kinetics studies

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Abstract

Present study focuses on the development of activated carbon from cassava peels by chemical activation with 10 % orthophosphoric acid (H_3PO_4). The biomass was impregnated for 24 hours and then carbonized at 450 °C for 3 hours. The characterization of the activated carbon obtained showed the microporous structure with an iodine index of 1348.18 mg/g and a specific surface of 671.75 m^2/g. Diuron adsorption tests with this activated carbon show that the equilibrium time is 40 min, the maximum amount of diuron adsorbed is estimated at 809.09 mg/g. Kinetics study showed that the adsorption is best described by the pseudo second order model with correlation coefficients higher than 0.997. The plot of the adsorption isotherms shows that the adsorption process is best described by the Langmuir isotherm model. As for the thermodynamic parameters, they reveal that the adsorption process is spontaneous and exothermic.

1. Introduction

Pesticides are chemicals, which are commonly used in agriculture to protect crops from pest organisms including insects, plants, fungi, rodents, and nematodes. Their use has revolutionized worldwide agricultural practices. They may appear as pollutants in water sources and threat to human health because of their toxicity, carcinogenicity, and mutagenicity [1]. Diuron is a worldwide used herbicide frequently detected in surface water with wide dispersion in watersheds [2]. It is used as pre- and post-emergent preferably in the growth control of a variety of broadleaf weeds, grass weeds and mosses, acting also with effectiveness in the treatment of non-agricultural areas [3].

Treatment of wastewater contaminated by pesticides has been one of the major efforts in recent decades. In general, the conventional treatment procedures are ineffective in recovering the wastewater contaminated by these agricultural residues [4]. Adsorption has been widely investigated as one of the most prominent methods to remove such waste [5, 6]. The main material frequently employed in this technique is activated carbon which has the advantage to possess large surface area, excellent internal and external functions, large adsorption capacities and good adsorption kinetics [7-9]. Recently, growing research interest in the production of carbon-based-activated carbon has been focused on agricultural by-products. Low-cost adsorbents derived from agricultural wastes have demonstrated outstanding capabilities for the removal of pollutants from wastewater [10].

In addition, the challenge of the incessant disposal of cassava peels waste after processing with the subsequent danger of creating breeding grounds for micro-organisms will be addressed with a projection of converting this waste to wealth. Thus, several researches are carried out to convert them into raw material for the production of biosorbents [11], biodegradable plastics [12-13], biopesticides replacing chemical pesticides for the protection of plants [14], biofertilizer [15], and biogas [16].

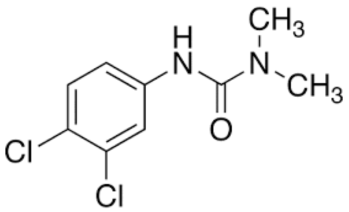
In this work, we attempt to use Cassava peels as low-cost, abundantly available and renewable precursor for production of activated carbon as adsorbent for the removal of diuron from aqueous solutions. The equilibrium and kinetic data of the adsorption process were then studied to understand the adsorption mechanism of diuron onto the prepared activated carbon.

2. Methodology

2.1 Adsorbate

The pesticide used as adsorbate in the experiments is diuron. Some properties and chemical structure of diuron is given in **Table 1.**

Table 1: Some properties and chemical structure of diuron

Common name	Diuron
Chemical structure	
Name	3-(3,4-Dichlorophenyl)-1,1-dimethylurea
Molecular formula	$C_9H_{10}Cl_2N_2O$
Molecular Weight (g/mol)	233.09
Pesticide group	substituted urea herbicide
Activity	herbicide that inhibits photosynthesis, it is used in agriculture for pre-emergent and post-emergent control of broadleaved and grass weeds.
Solubility in water at 20° C (mg /L)	40

2.2 Preparation and Characterization of Adsorbent

Cassava peels used in this study as precursor was sourced locally. These were washed severally with distilled water to remove water-soluble impurities and dried to constant weight in an oven at 70° C for 48 hours then ground. The obtained powder underwent successive sieving in order to retain only those whose diameters are included between 0.2 and 0.5 mm.

The sample was then soaked in orthophosphoric acid (H_3PO_4) 10% for 24 h. After 24 h, the sample was then washed in distilled water until the sample was neutralized, filtrated and dried in the oven at 60 °C for 24 h. The resulting sample was carbonized in a muffle furnace with heating ramps of 10°C per minute up to 450°C for 3 hours. The activated carbon obtained by this treatment were washed with distilled water, filtered and dried at 100 °C for 24 h and stored in a vacuum desiccator until needed.

Characterization of the activated carbon was achieved through various analyses. Iodine and methylene blue indexes were used as indicators for the capacity of the activated carbon to adsorb small (micro-porosity), medium and large organic molecules respectively (mesoporosity/macroporosity). Methylene blue index was calculated using the method of the European Center of Chemical Industry Federations

(CEFIC, 1989) taken from the work of Mamane et al. [17]. The methylene blue index (MBI) is determined by evaluating the amount of methylene blue adsorbed from a solution of initially known concentration per gram of adsorbent (0.1 g of activated carbon), when being in contact for 40 min at room temperature (30 °C). The absorbances were measured by the molecular absorption spectrophotometer (UV-5200). and the MBI was determined as shown in **Eqn. 1**.

$$MBI\ (mg/g) = \frac{V_{MB}.(C_i - C_f) * M}{m_{AC}} \qquad \textbf{Eqn. 1}$$

where V_{MB} (L) is the total volume of methylene blue solution, Ci (mol/L) is the initial concentration of methylene blue solution of, C_f (mol/L) is the final concentration methylene blue solution, m_{AC} (g) is the mass of activated carbon in contact with diuron solution.

Iodine index was carried out according to the AWWA B 600-78 standard taken from the work of Maazou et al. [18]. Iodine index involves titration of a known volume of iodine solution with a 0.1 N solution of sodium thiosulfate before and after adsorption on activated carbon. 20 mL of 0.1 N iodine solution was added to 0.05 g of activated carbon. The mixture was stirred for 30 min at room temperature (30 °C) and centrifuged at 3000 rpm. Then 10 mL of the supernatant was titrated with a 0.1 N sodium thiosulfate solution. **Eqn. 2** shows the calculation for iodine index I_2 (mg/g)

$$I_2 = \frac{25.4(20 - V_{th})}{m_{ac}} \qquad \textbf{Eqn. 2}$$

The specific surface area was calculated from the amount of adsorbed methylene blue at maximum adsorption capacity according to

$$S_L = \frac{Q_m N_A S_{BM}}{M_{BM}} \qquad \textbf{Eqn. 3}$$

S_L : Specific surface area of Langmuir (m^2/g)
Q_m : Maximum adsorption capacity (mg/g)
N_A : Avogadro number ($6.022.10^{23} mol^{-1}$)
S_{BM} : Surface occupied by a molecule of methylene blue ($175.10^{-20} m^2$)
M_{BM}: Molecular Weight of methylene blue = 319.85 g/mol

The determination of the maximum adsorption capacity is done by applying the Langmuir model to the adsorption isotherms of methylene blue on activated carbon

The ash content was determined by the method described by [19]. A sample of 0.5g (m_1) of dried activated carbon is placed in a crucible (m_2). This crucible is introduced into a muffle furnace set at 650°C for 2 hours. At the exit of the furnace, the crucible is cooled to room temperature. The crucible is weighed again (m_3). The ash content is determined as follows:

$$Ash(\%) = \frac{(m_3 - m_2)}{m_1} \times 100 \qquad \textbf{Eqn. 4}$$

2.3 Adsorption kinetic studies

Diuron adsorption kinetics experiments using activated carbon were performed to determine the equilibrium time and the best kinetic model.

For the adsorption kinetic experiments, in 250 mL Erlenmeyer flask, 0.1 g of activated carbon was added to 100 mL of diuron solution with an initial concentration of 100 mg/L. The mixtures were agitated at 120 rpm for the allotted contact time (2–100 min). Activated carbon and solution were separated by centrifuging (SIGMA 2-16P) at 3500 rpm for 10 min. Diuron concentration in the solution was measured using UV-visible spectrophotometer (UV-5200) at 262 nm.

The effect of the initial diuron concentration was investigated by varying the initial concentration from 20 to 150 mg/L. The amount of adsorption at time t, that is q_t (mg/g), was calculated by:

$$q_t = (C_o - C_t) \times \frac{V}{m} \qquad \textbf{Eqn. 5}$$

Where C_t and C_0 (mg/L) are the residual concentration of diuron at time t (min) and the initial concentration of diuron respectively, V (L) is the solution volume, and m (g) is the mass of activated carbon used.

The rate constants of the adsorption were determined from the pseudo-first-order and pseudo-second-order equations. For the pseudo-first-order, the Lagergren expression given below was used [20]:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \qquad \textbf{Eqn. 6}$$

where qe and qt (mg/g) are the amounts of diuron adsorbed at equilibrium and at time t (min), respectively and k_1 is the rate constant of adsorption.

The linear form of the pseudo-second-order reaction [21] can give by:

$$\frac{t}{q_t} = \frac{1}{k_2 {q_e}^2} + \frac{1}{q_e} t \qquad \textbf{Eqn. 7}$$

where the equilibrium adsorption capacity (qe), and the second order constant k_2 (g/mg.min) can be determined experimentally from the slope and intercept of plot t/q_t versus t

2.4 Batch Equilibrium Studies

Batch equilibrium studies were conducted in an incubator with thermostatically-controlled water bath considering the effect of temperature in adsorption in 30, 40, 50 and 60 ± 2°C, shaking of 120 rpm, in duplicate. Adsorption tests were performed in a set of Erlenmeyer flasks (250 mL) where 100 mL of diuron solutions with initial concentrations of 60–200 mg/L were prepared. An amount of 0.1 g of the activated carbon was added into each flask covered with glass stopper and the flasks were then placed in an isothermal water-bath shaker with agitation speed of 120 rpm to reach equilibrium. All samples were filtered prior to analysis in order to minimize interference of the carbon fines with the analysis. The concentrations of diuron in the solution before and after adsorption were determined using a double beam UV-vis spectrophotometer (UV-5200) at 262 nm. The amount of adsorption at equilibrium, qe (mg/g), was calculated by:

$$q_e = (C_o - C_e) \times \frac{V}{m} \qquad \textbf{Eqn. 8}$$

where Co and Ce (mg/L) are the concentrations of diuron at initial and at equilibrium time t respectively. V (L) is the volume of the solution and m (g) is the mass of dry adsorbent used.

The adsorption isotherm model is of fundamental importance to describe the interactive behavior between the adsorbate and the adsorbent. Analysis of isotherm data is significant for predicting the adsorption capacity and describing the surface properties and affinity of the adsorbent. Two commonly used models, the Langmuir and Freundlich isotherms, were selected to analyze the equilibrium experimental data for the adsorption of diuron onto activated carbon.

The linear form of the Langmuir [22] model is:

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{k_L \times q_{max}} \frac{1}{C_e} \qquad \textbf{Eqn. 9}$$

where Ce is the equilibrium concentration of diuron (mg/L), q_e is the amount of diuron adsorbed by a unit mass adsorbent (mg/g), q_{max} represents the maximum amount of diuron adsorbed by the unit mass adsorbent (mg/g), and k_L is Langmuir constant (L/mg).

In order to determine if the adsorption process is favorable or unfavorable, a dimensionless constant separation factor or equilibrium parameter R_L is defined according to the following equation [23]:

$$R_L = \frac{1}{1+k_L C_0} \qquad \textbf{Eqn. 10}$$

where k_L is the Langmuir isotherm constant (L/mg) and C_0 is the initial diuron concentration (mg/L). The R_L value indicates the type of the isotherm: favorable ($0 < R_L < 1$), unfavorable ($R_L > 1$), linear ($R_L = 1$) or irreversible ($R_L = 0$).

The Langmuir constants can be obtained from the plot of 1/qe vs. 1/Ce.

Freundlich isotherm assumes multilayer sorption on a heterogeneous surface. The linear form of Freundlich equation can be expressed as [24]:

$$\mathrm{Ln}q_e = \ln k_F + \left(\frac{1}{\mathrm{n}}\right) \ln C_e \qquad \textbf{Eqn. 11}$$

where q_e and C_e are the same as defined above and k_F and n are Freundlich constants, which indicate the adsorption capacity and adsorption intensity of a given material, respectively.

The thermodynamic parameters such as free energy change (ΔG°), enthalpy change (ΔH°) and entropy change (ΔS°) have a significant role to define the feasibility, spontaneity and heat change for the adsorption process and can be estimated by the following equations:

The free energy of adsorption (ΔG°) can be related with the equilibrium constant K_d (L/mol), the values of enthalpy change (ΔH°) and entropy change (ΔS°), for the adsorption process were calculated, using the following Eqns. 12 – 14

$$K_d = \frac{q_e}{C_e} \qquad \textbf{Eqn. 12}$$

$$\Delta G^O = -RTLnK_d \qquad \textbf{Eqn. 13}$$

$$LnK_d = \frac{\Delta S^O}{R} - \frac{\Delta H^O}{RT} \qquad \textbf{Eqn. 14}$$

Plot of LnK_d as a function of 1/T yields to a straight line. The ΔH° and ΔS° parameters were calculated from the slope and intercept of the plot, respectively.

3. Results and Discussion

3.1 Characterization of adsorbent material

Table 2 shows the physicochemical characteristics of cassava peels activated carbons. According to [17], the maximum tolerated ash content value for activated carbon is 15%. In the case of this study value obtained 4%. is well below this value. Low value of MBI (331 mg/g) compared to iodine index

(1348.18 mg/g) demonstrates that this activated carbon is well indicated for adsorption of small particles and could then be considered as microporous. According to **Table 2**, the total specific area of the cassava peels activated carbons. was 621.75 m^2/g, whose value is consistent with the limit recommended for commercial activated carbons according to [3].

Table 2. Physical and chemical characteristics of cassava peels activated carbon

Caractéristiques	**Description**
Ash content (%)	4%
Iodine index (mg/g)	1348.18
Methylene blue index (mg/g)	331.16
Surface spécifique m^2/g	621.75

3.2. Effect of initial concentration and agitation time on diuron adsorption

The Equilibrium contact time is very important in order to determine the adsorption capacity of the adsorbents. **Figure 1** describes the effect of contact time and initial concentration on the removal of diuron by cassava peels activated carbon.

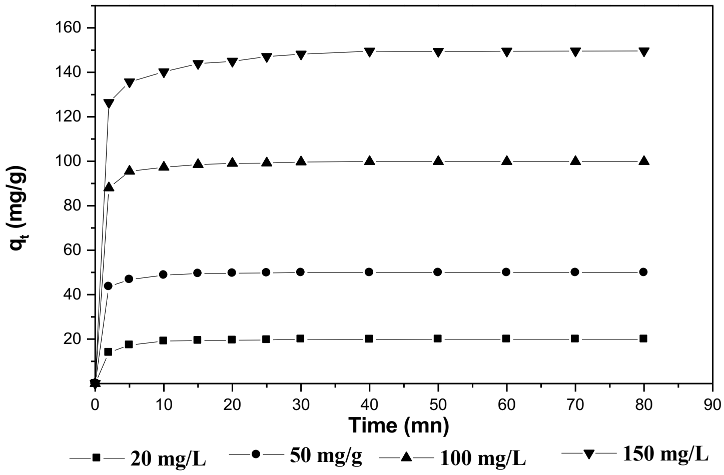


Figure 1. Effect of diuron initial concentration and agitation time on adsorption uptake at (30 ◦C)

The adsorption study was conducted at different predetermined contact time and initial concentrations four initial concentrations 20, 50, 100 and 150, mg/L were selected for this study while adsorbent dose remained constant at a temperature of 30°C. The plots showed that the adsorption of diuron increase with time till it reached a constant value beyond which no more diuron was further removed from the solution. The results revealed that the diuron adsorption was fast at the initial stages of the contact period, and slowed down near equilibrium. The high adsorption rate at the beginning of adsorption was due to the adsorption of diuron by the exterior surface of the adsorbent. When saturation was reached at the exterior surface, the diuron molecules entered in the pores of adsorbent and were adsorbed by the interior surface of the particles [5]. Similar trend was observed in the adsorption of TCP on coconut husk-based activated carbon [25].

Figure 1 show that the time for the diuron solution with initial concentration of 20 to 50 mg/L to reach equilibrium was 30 min while the diuron solutions with initial concentration of 100 to 150 mg/L required longer time to reach equilibrium (40 min). This was due to the fact that at low concentration, the ratio of available surface to the initial diuron concentration was larger which had eased the adsorption process; therefore, it was easier for the process to attain equilibrium**.** However, in the case of higher concentrations, this ratio was relatively lower, thus more time to was need to achieve equilibrium. This indicates that initial concentrations played an important role in the adsorption of diuron on activated carbon.

3.3. Adsorption kinetics

The kinetics of the adsorption process is very important in order to assess the rate and fix the equilibrium time of adsorption based on physical and chemical properties of adsorbent and adsorbate. In order to evaluate the adsorption kinetics of diuron, the two models of Lagergren pseudo-first-order given in **Eqn. 6** and Ho pseudo-second-order represented by **Eqn. 7** were used. The values of qe and k_1 can be determined from the slope and intercept of the plot obtained by plotting log (qe – qt) versus t (Figure not shown). The same way, values for k_2 and qe were calculated from the slope and intersection of the straight line from the graphic t/qe versus t (**Figure 2**).

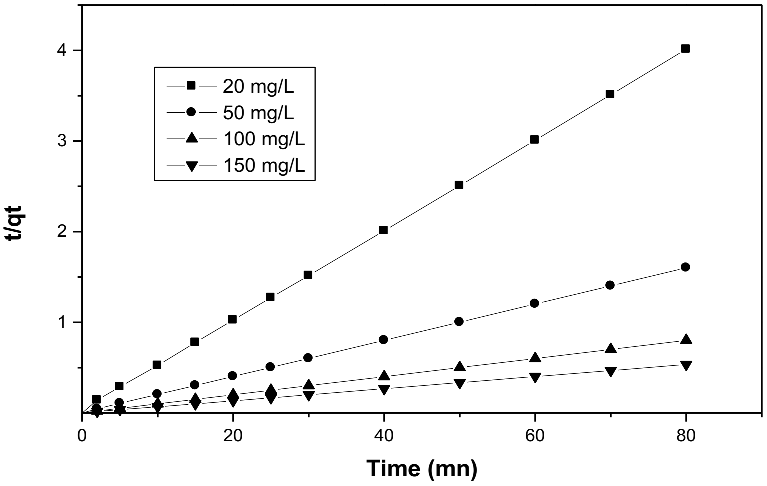


Figure 2. Pseudo-second-order kinetics for adsorption of diuron at various initial concentration onto activated carbon

Values for all constants under different initial concentration of diuron for cassava peels activated carbon can be found in **Table 3**.

Table 3. Kinetic constants for pseudo-first and pseudo-second order models of diuron on activated carbon

		Pseudo-first order			Pseudo-second order		
Initial concentration (mg/L)	$qe_{,exp}$ (mg/g)	$qe_{,th}$ (mg/g)	k_1 (1/min)	R^2	$qe_{,th}$ (mg/g)	k_2 (mg/g.min)	R^2
20	19.94	7.98	0.152	0.956	20.1	0.092	0.999
50	49.89	14.521	0.206	0.965	50.05	0.108	0.999
100	99.82	39.07	0.186	0.947	99.88	0.100	0.998
150	148.93	46.15	0.216	0.943	150.15	0.040	0.997

Based on the pseudo-first-order, the R^2 values obtained were relatively low and the experimental qe values did not agree with the calculated values obtained from the linear plot. This indicate that the adsorption of diuron onto the activated carbon did not fit into the pseudo-first-order equation. But, the plot presents an ideal fit to the pseudo-second-order model (**Figure 2**), with extremely high R^2 almost equal to unity for all initial concentration (**Table 3**). A good agreement can further be supported by the similar values of the calculated and experimental values of qe. The best fit to the pseudo-second-order kinetics indicated that the adsorption mechanism might depend on both the adsorbate and the adsorbent **[26]**, and the ratelimiting step may be chemisorption involving valence forces through the sharing or exchange of electrons **[27]**. Moreover, the pseudo-second-order kinetic model covers all processes of adsorption, such as external liquid film diffusion, internal particle diffusion and surface adsorption, among others **[28]**, which can reflect the adsorption mechanism of diuron onto activated carbon more accurately and comprehensively

3.4. Adsorption isotherm

The results behavior of diuron adsorption isotherms evaluated in the temperatures (30, 40, 50 and 60) °C are displayed in **Figures 3** and **4**. Whereas in **Table 4**, are the values of the parameters of both models evaluated (Langmuir and Freundlich) in different temperatures, calculated by **Eqns. 9** and **11**. According to **Table 4**, the correlation coefficients (R^2) indicate that both Langmuir and Freundlich equations were satisfactory, but the Langmuir isotherm fits the equilibrium data better at different temperatures which confirms assumption that maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface, with no lateral interaction between the sorbed molecules **[29]**. With the increase of temperature from 30 °C to 60 °C, the maximum capacity at equilibrium of activated carbon decreased from 809.09 mg/g to 714.28 mg/g; which implies that the adsorption process is exothermic and favored at lower temperatures. The values of R_L were in the range of 0.213 – 0,333 indicating that the adsorption was favorable. The values of 1/n ($0.1 < 1/n < 1$) indicated favorable adsorption at experimental conditions and implies that adsorption process is chemisorption **[30]**.

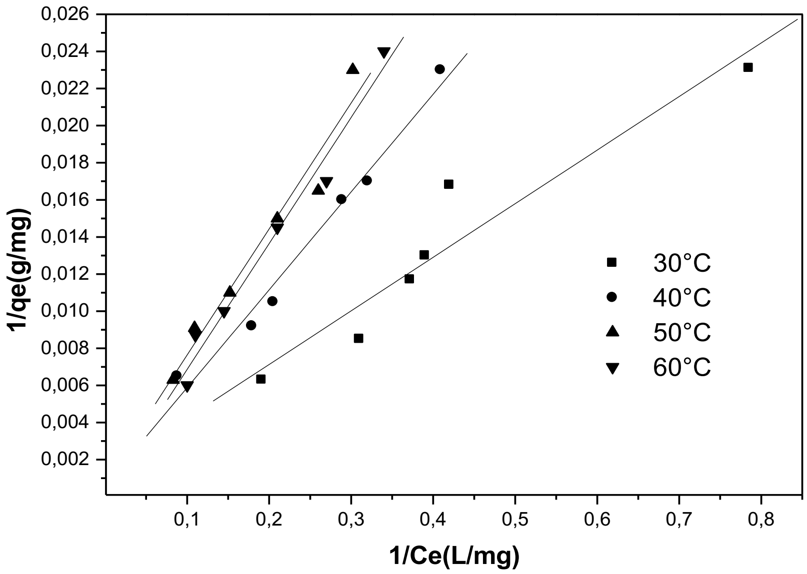


Figure 3. Langmuir isotherm plots for adsorption of diuron onto activated carbon at different temperatures.

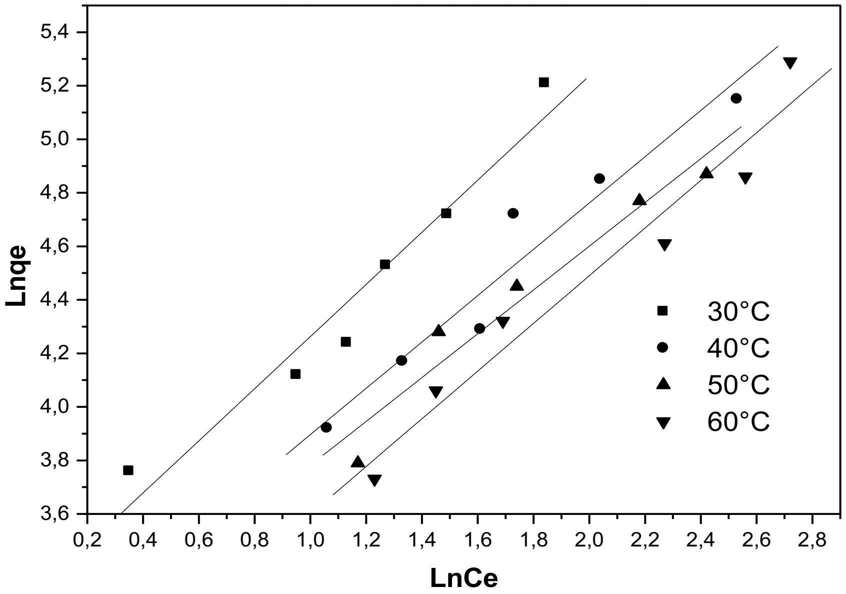


Figure 4. Freundlich isotherm plots for adsorption of diuron onto activated carbon at different temperatures.

Table 4. Parameters of adsorption for isotherms of Langmuir and Freundlich

T (°C)	Langmuir isotherm				Freundlich isotherm		
	qm (mg/g)	K_L (L/mg)	R^2	R_L	K_F (mg/g)(mg/L)$^{1/n}$	1/n	R^2
30	809.09	0.037	0.956	0.213	28.76	0.897	0.940
40	758.28	0.025	0.989	0.286	21.18	0.885	0,970
50	746.23	0.022	0.979	0.312	19.11	0.869	0.971
60	714.28	0.020	0.989	0.333	17.24	0.796	0.975

3.5. Adsorption thermodynamics

Van't Hoff plot of LnK_d as a function of 1/T (**Figure 5**) yields to a straight line. The standard enthalpy change (ΔH°) and standard entropy change (ΔS°) parameters were calculated from the slope and intercept of the plot, respectively.

The values of thermodynamic parameters are listed in **Table 5**. The negative values of Gibbs free energy change (ΔG°) indicate that process is thermodynamically feasible and spontaneous. The negative value of ΔH° confirms that process is exothermic. The positive value of ΔSº indicates that an increase in the degree of disorder in the liquid-solid interface was necessary **[31]**, in relation to molecules of diuron adsorbed in aqueous solution, related with the best diffusion of these molecules to the interior of the micropores of the activated carbon. All thermodynamic parameters were very satisfactory to the adsorption process of diuron since they showed great adjustment (R^2) of 0.997. The results obtained agreed well with those obtained by several authors **[32-34]**.

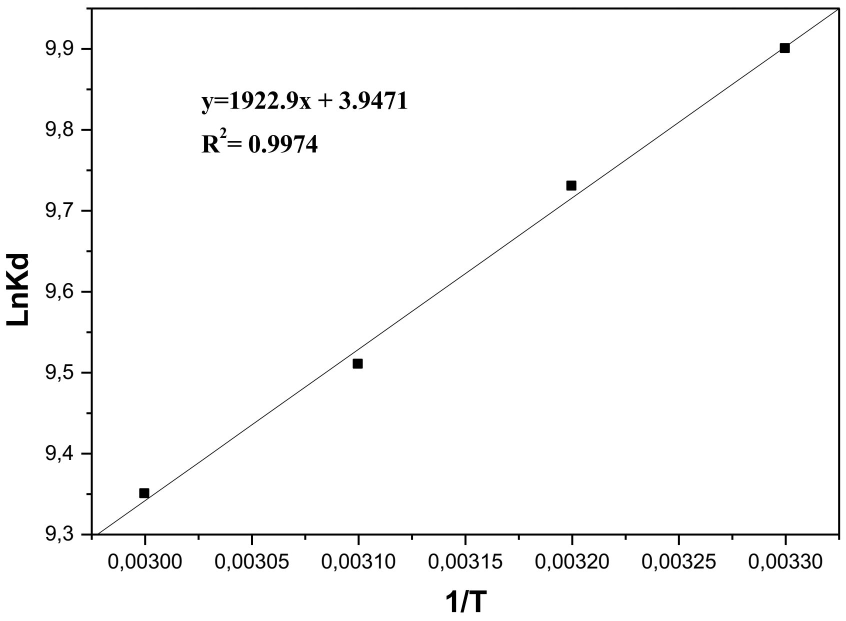


Figure 5. Plot of $\ln K_d$ versus 1/T

Table 5. Thermodynamic parameters of adsorption of diuron on activated carbon.

Température(°C)	ΔG° (kJ /mol)	ΔH° (kJ/mol)	ΔS° (J/K.mol)	R^2
30	-24.85	-15.98	32.80	0.997
40	-25.17			
50	-25.49			
60	-25,806			

Conclusion

In this work, activated carbon prepared from Cassava peels using phosphoric acid as activation agent has been applied to remove diuron from aqueous solutions. The surface area of the adsorbent was found to be 621.75 m^2/g. Equilibrium data were fitted to the Langmuir and Freundlich isotherms, and the equilibrium data were best described by the Langmuir isotherm model, with the maximum monolayer adsorption capacity of 809.09 mg/g at 30°C. Kinetic data were tested using the pseudo-first order and pseudo-second order kinetic models equation. The kinetics of the adsorption process was found to follow the pseudo-second-order kinetic model. Thermodynamic parameters suggested that the adsorption of diuron on the Cassava peels activated carbon was feasible, spontaneous and exothermic in nature.

Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest. *Compliance with Ethical Standards:* This article does not contain any studies involving human or animal subjects.

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