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Adsorption of Atrazine from Aqueous Solution Using Desert Date Seed Shell Activated Carbon

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Abstract: *Exposure to atrazine, one of the most commonly used herbicides, has been associated to many health problems including cancer and infertility. During the course of application, off-target movement of these chemicals usually results into crop damaging as well as environmental pollution. Therefore, in this study, an attempt has been made to treat water contaminated with atrazine using activated carbon produced from desert date seed shell. The activated carbon was prepared via chemical activation method using potassium hydroxide. Prior to adsorption experiments carried out at various adsorbent dosages, contact time, initial atrazine concentration and temperature, Fourier transform infrared spectrophotometer was used to characterize the prepared desert date seed shell activated carbon (DDSSAC). In order to investigate the mechanism and kinetics of the adsorption, experimental data were fitted to some adsorption isotherms and kinetic models. The batch adsorption experiments revealed that increasing adsorbent dosage and contact time but decreasing temperature and initial concentration of atrazine increased percentage atrazine removal. The results of the isotherm studies revealed that the equilibrium data fitted to both Freundlich and Langmuir model with R-squared values of 0.9956 and 0.9996 respectively, which implied that Langmuir isotherm had a better fit. This was also found to be an indication that the uptake of atrazine by DDSSAC occurred through monolayer sorption on identical homogenous sites. Also, kinetic modelling results obtained showed that pseudo-second-order model explained the adsorption kinetics of atrazine by DDSSAC best, which meant that chemisorption was the slowest step and, thus, the rate determining step. This study has indicated that desert date seed shell can be used to produce activated carbon that can be applied effectively for adsorption of atrazine from aqueous solution.*

Keywords: *Atrazine, adsorption, kinetics, isotherms, desert date seed shell activated carbon (DDSSAC).*

1. INTRODUCTION

Generally, chemicals such as atrazine (2-chloro-4-ethylamino-6-isoproplamino-1,3,5-trazine) are used in the prevention, destruction or control of pests with the aim of improving crop yield. However, they can pose a threat when they leach into groundwater and enter drinking water supplies. Human exposure to atrazine has been linked to serious health effects including breast and prostate cancer, ovarian cancer, thyroid cancer, birth defects, impaired sexual development, insulin resistance and infertility [1]. As such treatment of herbicide contaminated water is of paramount importance.

Water treatment facilities employ methods such as coagulation/flocculation, ozonation and adsorption to treat various harmful pollutants in water. Activated carbon adsorption has been cited by the United States Environmental Protection Agency (USEPA) as one of the best available environmental pollution control technologies [2]. Activated carbon adsorption involves the use of carbonaceous material with a large internal surface area and high porosity as adsorbents for removal of material of interest called adsorbate from liquid or gas. Production of activated carbon is usually achieved through two major steps, namely carbonation of precursors, which is the method of converting the precursor to carbon by heating at high temperature, and chemical activation. Activated carbon adsorption has been widely used for water/wastewater purification due to its high efficiency and versatility in treating various contaminants [3].

Cost-effectiveness has been identified as one of the major challenges associated with adsorption using activated carbon [4]. Researchers in the recent past have mainly focused on the preparation of the activated carbon from agricultural waste materials as an alternative for the commercial type. Consequently, numerous low-cost alternatives have been proposed including biowaste, agricultural and industrial waste-based precursors, some of which have been fruitfully used for activated carbon preparation [4]. Several agricultural waste products like jackfruit peel [13], banana pitch peel [14], rice husks [23], corn cob [26] and as reviewed by [5], fruit shells, seed husk, saw dust, hyacinth root have been tried as alternative low-cost adsorbents by various researchers in recent past decades. Apart from cost effectiveness, these precursors are easily available, rich in carbon and have low ash content. Also, they are obtained from renewable sources and, therefore, are better than those precursors obtained from coal and petroleum [3].

Presently, there is no known reported scientific use of desert date seed shell. However, it has been suggested that the nutshell is suitable for industrial activated charcoal. The calorific value has been estimated to be 4600 kCal/kg [6]. Therefore, this research work was carried out to investigate the possibility of processing the biowaste into adsorbent for removal of atrazine from aqueous solution. Also investigated the kinetics and mechanism of adsorption of atrazine using the desert date seed shell activated carbon (DDSSAC) produced via chemical activation.

2. METHODOLOGY

2.1 Precursor Preparation

The desert date seed shell used for this research were obtained during biodiesel production from desert date oil. The shells were gathered after the kernels were removed for oil extraction. They were then washed using tap water to get rid of dirty particles and any fruit remaining on the seed. The seed coat was then sun-dried (Figure 1) and, later, oven-dried for 1 hour. Thereafter, it was ground and sieved using 1.7 mm sieve followed by 2 mm sieve.

Figure 1. Desert dates seed coat sun-dried

2.2 Activation Process

The prepared precursor was impregnated with potassium hydroxide (KOH) solution at a ratio of 1:3 of precursors to activating agent for 1 hour [7]. It was then sun dried and oven dried at a temperature of 105 °C for 3 hours. It was then washed with hydrochloric acid followed by distilled water until a neutral pH was obtained after which it was dried again. The prepared precursor was then heated in an electric kiln up to a temperature of 800 °C while passing a stream of nitrogen gas (N_2) for 3 hours through it. The kiln was then allowed to cool, and the activated carbon was removed and sealed in an air tight container.

2.3 Testing of the Activated Carbon

Methyl orange, a highly soluble organic dye, was used to carry out a preliminary test on the activated carbon in order to know whether the chemical activation was successful. To achieve this, approximately one gram of activated charcoal was placed into the test solution and 60 minutes were allowed for adsorption to take place.

2.4 Characterization of the Activated Carbon

FTIR Analysis: The activated carbon from desert dates was analysed to detect the functional groups present in it using Fourier Transform Infrared Spectrophotometer (Shimadzu FTIR-8400S). The region of the infrared was between 4000-650 cm^{-1}.

Moisture Content Determination: The moisture content of the developed activated carbon was also obtained by weighing 10 grams of the carbon and placing it in an oven at 105 °C for 3 hours. Thereafter, the carbon was cooled in the absence of humidity and reweighed again. The difference between the initial and final weights of the carbon was the water content in the sample.

Pore (Void) Volume Determination: In order to determine the pore volume of the activated carbon, 2.0 g of that sample was immersed in water and boiled for 15 min. After the air in the pores had been displaced, the samples were superficially dried and reweighed. The increase in weight divided by the density of water gave the pore volume.

Bulk Density (Apparent Density) Determination: The bulk density of the sample was determined so as to know the packed density of a bed of granular activated carbon, which was calculated from the relation given in Equation (1).

$$Bulk\ density = \frac{Weight\ of\ activated\ carbon\left({}^{g}\!/_{mL} \right)}{100} \quad (1)$$

2.5 Batch Adsorption Experiment

The known weight of adsorbent was added to 50 mL of the atrazine solution with an initial concentration of 10 mg/L, and the content was shaken thoroughly. The solution was then filtered at particular time intervals and the residual concentration was measured with the aid of visible spectrophotometer (Jenway 6300). The same method was used while varying the initial concentration, the contact time, the mass of activated carbon dose, and temperature of adsorption. The amount of atrazine adsorbed (q_e) and the percent removal ($\%R$) were calculated using Equations (2) and (3), respectively.

$$q_e = V\frac{C_o - C_e}{m} \quad (2)$$

$$\%R = \frac{C_o - C_e}{C_o}100 \quad (3)$$

where q_e is the amount of atrazine adsorbed (mg/g), C_o and C_e are the initial and equilibrium liquid-phase concentrations of the atrazine (mg/L) respectively, V is the volume of the solution (L) and m is the weight of the adsorbent used (g).

2.6 Adsorption Isotherm

The adsorption isotherm can describe the distribution of atrazine between the solid phase and the solution at a certain temperature when the equilibrium is reached. In this work, Freundlich and Langmuir models were applied to fit the generated equilibrium data using linear form of Freundlich model given in Equation (4) by plotting *log* q_e against *log* C_e, and, also, the Langmuir model given in Equation (5) by plotting $1/q_e$ against $1/C_e$ in order to obtain the model constants.

$$\log q_e = \log K_f + \left(\frac{1}{n}\right)\log C_e \tag{4}$$

In Equation (4), K_f and n indicate adsorption capacity and adsorption intensity, respectively. Lower fractional values of n (in which it has values greater than 0 but less than 1[$0 < n < 1$]) indicate that weak adsorptive forces are effective on the surface of the sorbents [8-9] while in Equation (5), q_m is the maximum amount of adsorption (mg/g), and b is the adsorption equilibrium constant (L/mg) [10].

$$\frac{1}{q_e} = \frac{1}{bq_m}\frac{1}{C_e} + \frac{1}{q_m} \tag{5}$$

The essential characteristics of the Langmuir isotherm could be terms of dimensionless constant, separation factor or equilibrium parameter, r, that is defined as follows [11]:

$$r = \frac{1}{1 + bC_o} \tag{6}$$

where C_o is the initial adsorbate concentration (mg/L) and b is the Langmuir constant related to the energy of adsorption (L/mg). The value of r indicates the shape of the adsorption isotherm to know whether adsorption is unfavourable ($r > 1$), linear ($r = 1$), favourable ($0 < r < 1$) or irreversible ($r = 0$).

2.7 Adsorption Kinetics

Study of adsorption kinetics is important because the rate of adsorption, which is one of the criteria for efficiency of adsorbent, and also the mechanism of adsorption can be concluded form the kinetics studies. For this study, experimental data were tested for pseudo first-order, pseudo second-order and Elovich adsorption models using Equations (7), (8) and (9) respectively,

$$\log(q_e - q_t) = \log(q_e) - \left(\frac{K_{ad}}{2.303}\right)t \tag{7}$$

where q_e (mg/g), q_t (mg/g) are adsorption capacity at equilibrium and at time t, respectively, K_{ad} is the rate constant of pseudo first-order adsorption (L/min) [25].

Pseudo second order model is based on the assumption that chemisorption is the rate-determining step and expressed as given in Equation (8),

$$\frac{t}{q_t} = \frac{1}{h} + \frac{1}{q_e}t \tag{8}$$

where h, which is given as $h = kq_e^2$, is the initial sorption rate ($mg.g^{-1}.min^{-1}$) when t approaches zero, k is the pseudo-second-order rate constant [25].

The Elovich adsorption model is given below as

$$q_t = \left(\frac{1}{\beta}\right)\ln(\alpha\beta) + \frac{1}{\beta}\ln(t) \tag{9}$$

where β (g/mg) and α (mg/g/min) are the Elovich constants corresponding to the extent of surface coverage and rate of sorption at zero coverage (initial sorption rate constant) respectively [12].

3. RESULTS AND DISCUSSION

3.1 Test for Activated Carbon

In one of the reported works, adsorption capabilities of some plant-based adsorbents have been tested using methyl red and methyl orange [24]. So, in this work, the preliminary test was carried out on the produced DDSSAC using methyl orange solution. The discolouration of the solution was observed after 1 g of the DDSSAC was added to the solution and left for 1 hour. This was found to be an indication that the activated carbon has adsorptive property and, thereby, adsorbing the methyl orange from solution which made it (the solution) to become colourless later. Though, the adsorption of methyl orange by DDSSAC was not quantified but it (DDSSAC) appeared to perform better than neem tree bark, mango tree bark and locust bean tree bark powder reported by [24].

3.2 Spectral Analysis

Given in Figure 2 is the FT-IR spectrum of desert dates activated carbon, which shows various surface functional groups of the material. The peak obtained at about 2900 cm^{-1} indicated the presence and stretching vibration of carbon-hydrogen single bond (C-H). The broad but very weak peak in region of 2800 and 2700 cm^{-1} was an indication of carboxylic group. Also, weak peaks between 3450 and 3350 were associated with the presence of amine group. The frequencies from 2800 to 2000 cm^{-1} were normally void of other absorptions, and, so, the presence of alkyne or nitrile groups can be easily seen here. As such, the peak around 2400 cm^{-1} was observed to be an indication of one of these functional groups.

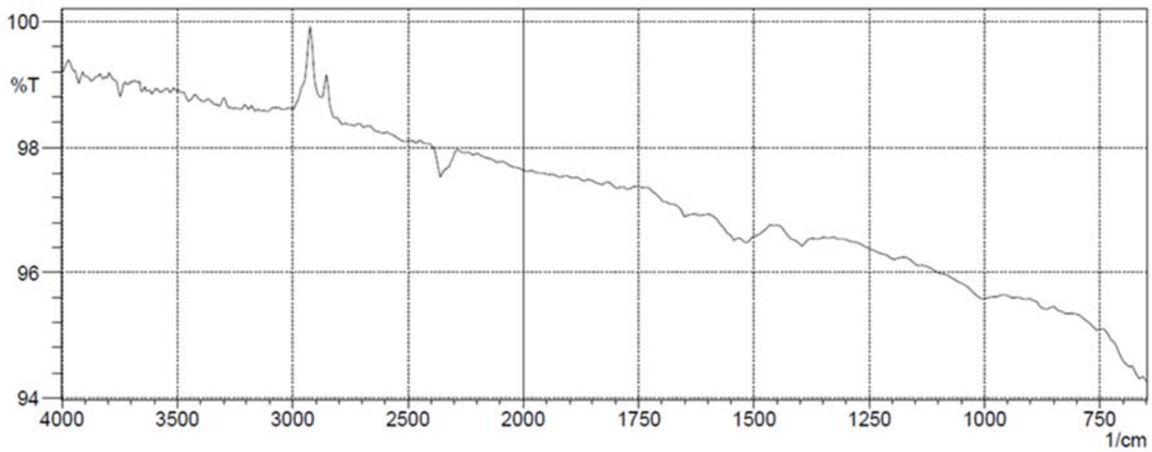

Figure 2. FT-IR spectra of desert dates seed shell activated carbon

3.3 Physical Properties of the Produced Activated Carbon

Some physical properties of the activated carbon produced are given in the Table 1. The value of the moisture content, pore volume and the bulk density of the produced activated carbon revealed that it had good adsorptive properties. It was noticed from the properties of the activated carbon that, though it might not give up to 100% adsorption, it will be good for adsorption of organics to a very large extent [13].

Table 1: Physical properties of the activated carbon

Parameter	Value
Moisture content	3.45%
Density	0.45 g/cm^3
Pore volume	1.23 cm

3.4 Effect of Various Factors on Adsorption of Atrazine

Various factors such as contact time, initial concentration of atrazine solution, mass dosage of activated carbon and initial temperature of the solution were monitored and varied for their effects on the adsorption of atrazine from solution using the produced desert dates seed shell activated carbon.

Effect of Contact Time: The percentage of atrazine adsorbed at various contact time were obtained by keeping DSSAC dosage, temperature and initial atrazine concentration constant at 0.5 g, 25 ^oC and 10 mg/L respectively, and the results were as given in Figure 3(a). As shown in the figure, atrazine removal was found to increase with increase in contact time significantly up till 80 min but, thereafter, no significant increment was observed. Thus, the maximum (equilibrium) time was taken to be 80 min. The later insignificant adsorption rate was due to the fact that the large surface areas of the activated carbon that were available at the beginning for the adsorption of atrazine from the solution had become exhausted after 80 min of adsorption.

Effect of Initial Atrazine Concentration: In order to investigate the effect of initial concentration on adsorption efficiency of DDSSAC, the adsorbent dosage, temperature and contact time were fixed at 0.5 g, 25 ^oC and 120 min respectively. The effect of initial atrazine concentration is graphically represented in Figure 3(b). It was seen from the results that the amount of adsorbate in the solid phase with lower initial concentration of adsorbate was smaller than the amount when higher concentrations were used. For instance, the percentage of atrazine removal was found to be 86.11% for 2 mg/L of initial concentration, where it was obtained to be 65.12% for that of 12 mg/L. It was also seen that the removal of atrazine was dependent on the concentration of atrazine as the increase in initial concentration was observed to decrease the amount of atrazine removed. It was clear from the results obtained that, although the amount of atrazine adsorbed per unit weight of adsorbent, qe, was increasing as the adsorbate initial concentration was increasing, the atrazine removal percentage was decreasing as the atrazine concentration was increasing.

Effect of Adsorbent Dosage: The effect of mass dosage of DDSSAC was studied in the range of 0.2-1.2 g with all other factors kept constant (contact time = 120 min, temperature = 25 ^oC, initial atrazine concentration = 10 mg/L), and the results obtained were as shown in Figure 3(c). According to the results, significant increase in uptake of atrazine was observed to occur when the dose was increased from 0.2 to 0.6 g. Any further addition of the adsorbent beyond this point was found not to cause any significant change in the adsorption. This phenomenon was observed to be due to overlapping of adsorption sites as a result of overcrowding of adsorbent particles [14]. It was apparent that by increasing the adsorbent dose, the amount of atrazine adsorbed increased, but adsorption density, the amount adsorbed per unit mass decreased. It was readily understood that the number of average adsorption sites increased by increasing the adsorbent dose and, this, therefore, resulted in increase in the amount of adsorbed atrazine [15]. The increase in adsorption density with an increase in the adsorbent dose was mainly due to adsorption sites remaining unsaturated during the adsorption. The decrease in adsorption density with an increase in the adsorbent dose was mainly because of the adsorption sites remaining unsaturated during the adsorption process whereas the number of sites available for adsorption site increased by increasing the adsorbent dose [16-17].

Effect of Temperature: The effect of temperature on activated carbon adsorption of atrazine was studied from 20 – 70 °C with all other factors being kept constant (DDSSAC dosage = 0.5 g, contact time = 120 min, initial atrazine concentration = 10 mg/L). The results are graphically shown in Figure 3(d). From the results, it was observed that there was a decrease in the amount of atrazine removed as the temperature was increased from 20 to 70 °C. This was found to be as a result of the fact that as the temperature was increased, the solubility of atrazine in solution also increased resulting in stronger interaction forces between atrazine and solute than those between atrazine and desert dates activated carbon [17-19]. Furthermore, a decrease in the adsorption of atrazine with increasing temperature was related to the increasing Brownian movement of molecules in solution. Also, it was observed that high temperature might lead to the breaking of existing intermolecular hydrogen bonding between atrazine and desert dates activated carbon, which was found to be an important contribution to the adsorption process [20-21].

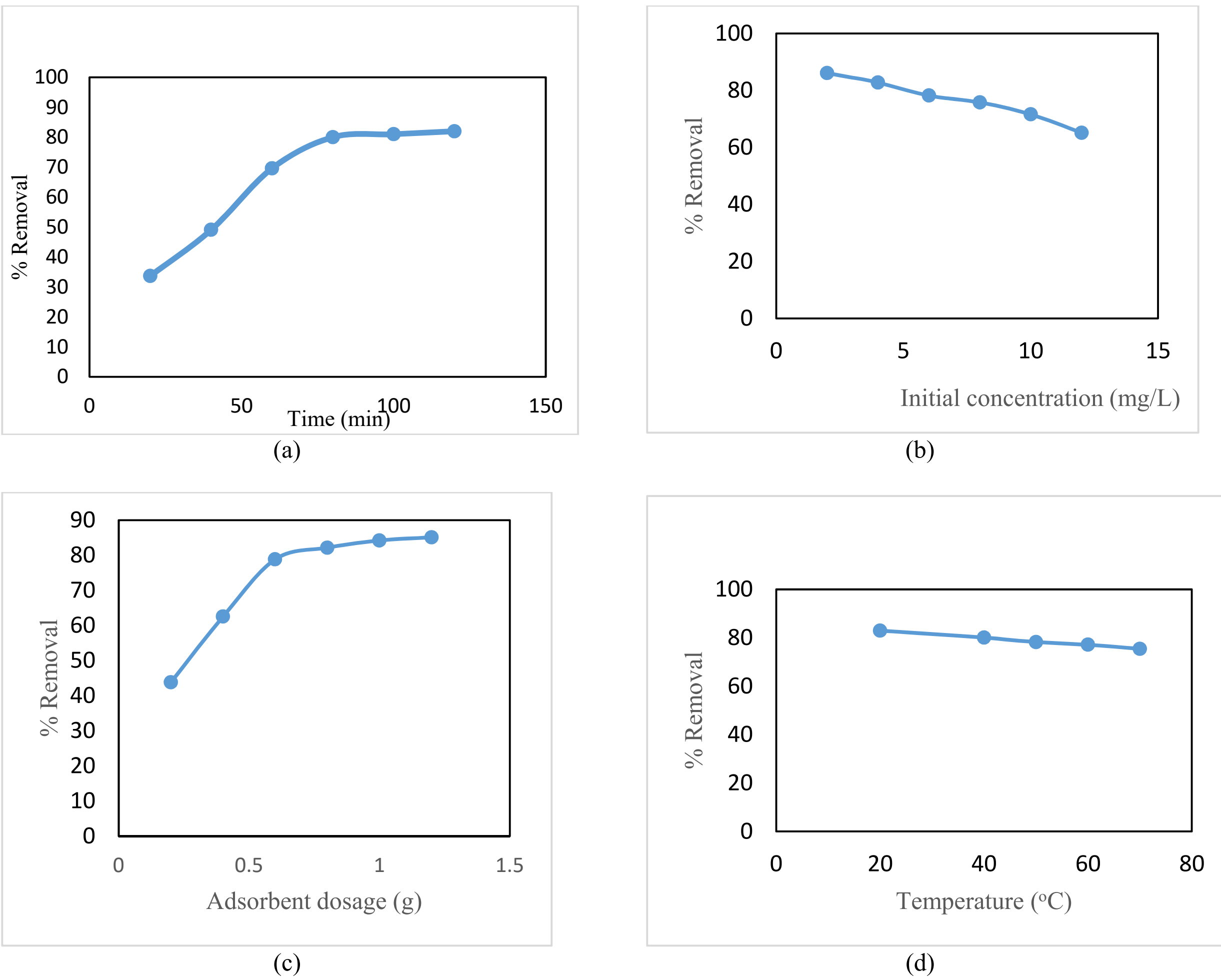

Figure 3. Effects of contact time (a), initial concentration (b), adsorbent dosage (c) and temperature on adsorption of atrazine onto DDSSAC

3.5 Adsorption Isotherms

Various adsorption isotherm models were tried to obtain the best fit to atrazine adsorption by DDSSAC. The plot for the Freundlich adsorption isotherm is given in Figure 4(a). The Freundlich constants K_f and n were found to be 0.0982 and 1.1644 respectively. The magnitudes of K and n show how easy the separation of atrazine from the aqueous solution is achieved and indicate favourable adsorption. The intercept K_f value is an indication of the adsorption capacity of the adsorbent while the slope $1/n$ indicates the effect of concentration on the adsorption capacity and represents adsorption intensity. The Freundlich isotherm was found to fit well with a square of correlation coefficient of 0.9956. In addition, the value of n being greater than unity was observed to be an indication that the adsorption of atrazine by DDSSAC was a physical type, according to Aljeboree *et al.* [20] and Kumar *et al.* [22].

The plot for the Langmuir adsorption isotherm is given in Figure 4(b). It is seen from the figure that the data were able to be fitted into the Langmuir equation well

(R^2=0.9996). The values of q_m and b were determined from the plot and were found to be 3.235 mg/g and 0.0282 L/mg respectively. The value of the separation factor or the equilibrium parameter, r, was obtained as 0.780. This value of r ($0 < r < 1$) was an indication that the adsorption was favourable.

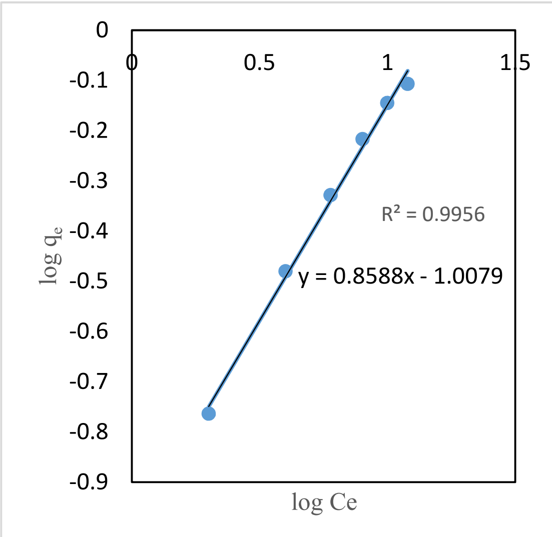

Figure 4(a). Freundlich adsorption isotherm

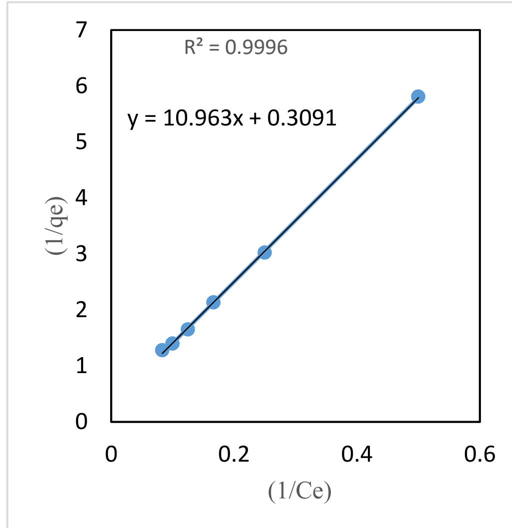

Figure 4 (b). Langmuir adsorption isotherm

3.6 Kinetics Study

Figure 5(a) shows the variation of the amount of atrazine adsorbed (q_e) as a function of time. The rate of adsorption of atrazine was observed to be high at initial early time but, later, almost became constant. This was found to be due to the availability of larger area for adsorption at the early time of the process.

The pseudo first-order plot obtained is given in Figure 5(b). The straight line plot of $\log(q_e - q_t)$ against q_t was used to estimate the rate constant, k_{ad} to be 0.01335 min^{-1} and the theoretical q_e as 0.9677 mg/g, which was obtained to be close to the practical value given as 1.0954 mg/g. With the square of correlation coefficient of 0.9484, it was seen that adsorption of atrazine using desert dates activated carbon could follow the pseudo first-order kinetics well.

Given in Figure 5(c) is the linearized plot of the pseudo second-order kinetics. It was discovered that this model gave a better straight line with square of correlation coefficient of R^2 = 0.9663, compared to that of the pseudo first-order model. The pseudo second-order rate constant, k, was estimated to be 0.0161 $g.mg^{-1}.min^{-1}$ and h, which is the initial sorption rate was obtained to be is 0.0238 $mg.g^{-1}.min^{-1}$. The theoretical value of q_e (1.2178 mg/g) was also obtained to be very close to the experimental value of 1.0954 mg/g. These results indicate that the adsorption of atrazine by desert dates activated carbon follows the pseudo second-order kinetic model, which relies on the assumption that chemisorption (chemical adsorption) may be the rate determining step. According to Wong et al. [23], this implies that the atrazine molecules were sticking to the surface of the adsorbent by forming a chemical (usually covalent) bond and tending to find sites that maximize their coordination number with the surface.

The Elovich equation was used for general application to chemisorption. The equation has been applied satisfactorily to some chemisorption processes and has been found to cover a wide range of slow adsorption rates. The graph is shown in Figure 5(d). The values of the constants were determined from the linear plot of q_t versus ln(t). The value of the extent of surface coverage, β was obtained to be 3.3715 g/mg while the rate of sorption at zero coverage, α was 0.0457$mg.g^{-1}.min^{-1}$. The square of correlation coefficient was found to be 0.9565, which was observed to be higher than that of the pseudo first-order kinetics but lower than that obtained for the pseudo second-order kinetics, implying that the adsorption process follows the pseudo second-order kinetics better than the Elovich model.

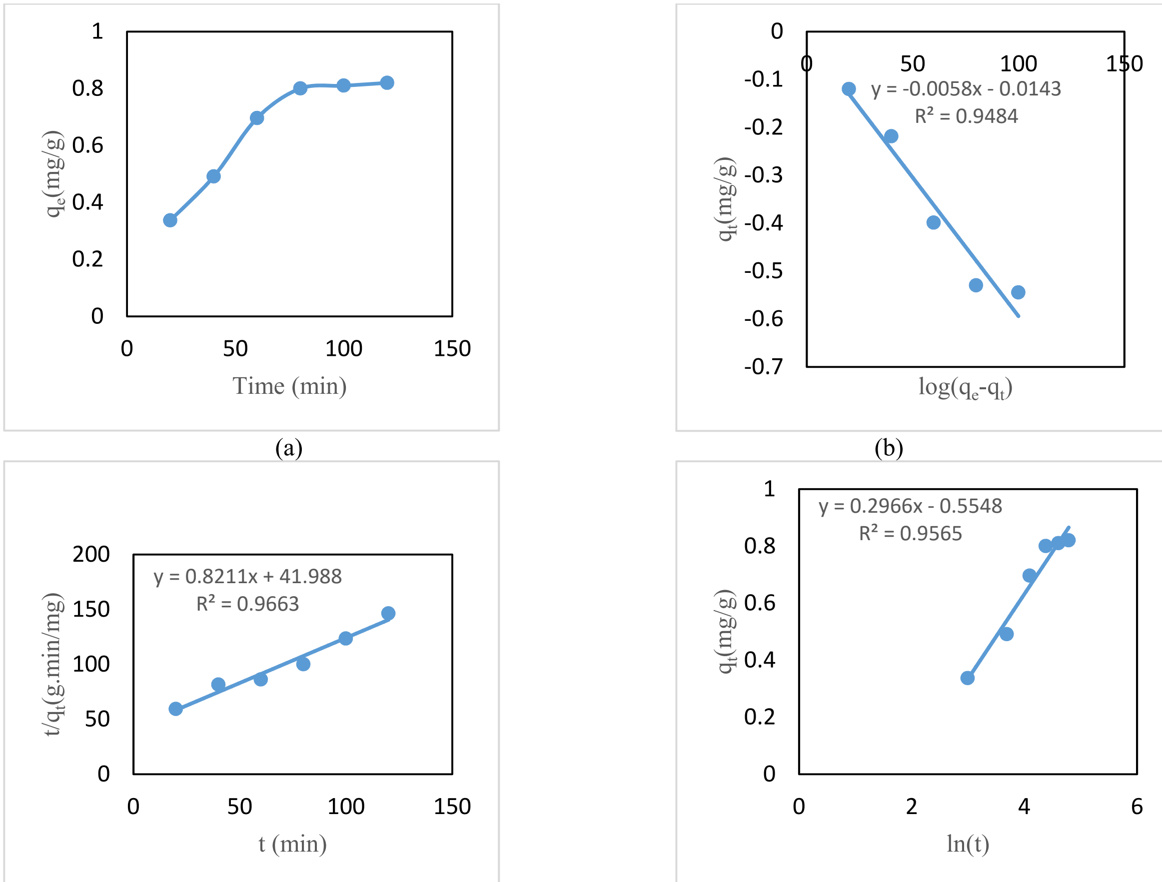

Figure 5: (a) Kinetics curve; (b) Pseudo first-order plot; (c) Pseudo second-order plot and (d) Elovich plot

Table 2: Summary of isotherm studies

Freundlich Isotherm			Langmuir Isotherm			
K_f	N	R^2	q_m (mg/g)	b (L/mg)	r	R^2
0.0982	1.1644	0.9956	3.2352	0.0282	0.7801	0.9996

Table 3: Summary of kinetics studies

Kinetic Model	Parameter	
Lagergren pseudo first-order rate equation	q_e (mg/g)	1.8447
	K_{ad} (min^{-1})	0.0134
	R^2	0.9484
Pseudo second order rate equation	k (g/mg.min)	0.0161
	q_e (mg/g)	1.2179
	h (mg/min)	0.0238
	R^2	0.9663
Elovich equation	A	0.0457
	B	3.3715
	R^2	0.9565

CONCLUSIONS

In this work, desert date shells, a waste, has been successfully utilized as a precursor for the preparation of activated carbon used for the removal of hazardous water contaminant, atrazine from water. Adsorption of atrazine on DDSSAC was found to be affected by initial contact time, initial atrazine concentration, adsorbent dosage and temperature. The adsorption was found to follow the pseudo second-order kinetics model and the Langmuir adsorption isotherm model with R^2-value 0.9663 and 0.9996 respectively. DDSSAC is a promising biosorbents for adsorptive removal of atrazine from aqueous solution with 80% efficiency.

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