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RESEARCH ARTICLE

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Adsorption of diazinon residues from water by *Strychnos potatorum* seed flakes: Equilibrium isotherm and thermodynamic analysis

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Abstract

Objectives: Contamination of pesticide residues due to agricultural activities is a major concern of aquatic pollution and most of the agriculture based rural communities in developing countries are still consuming water from those contaminated water bodies. Therefore, development of readily available user-friendly low cost pesticide contaminant removal methods is still in need for above communities. In this study the suitability of *Strychnos potatorum* seed flakes (SPSF) on effective removal of diazinon residues in water was investigated. **Methods**: SPSF was prepared and characterized using SEM and FTIR analysis. Batch adsorption studies, isotherm studies and thermodynamic studies were carried out to determine the removal efficiency of diazinon by SPSF. **Findings:** The maximum removal of diazinon residues (75.9%) was obtained within 10 min at pH 6. Therefore, SPSF are efficient adsorbent for diazinon removal due to fast and efficient removal of diazinon at ambient conditions. The removal of diazinon residues by SPSF was expressed with Langmuir isotherm model. The maximum adsorption capacity of SPSF, obtained from Langmuir isotherm for diazinon adsorption was found to be 2.5mg/g. Thermodynamic studies revealed that the removal of diazinon by the SPSF was spontaneous and exothermic at relatively low temperatures. **Novelty:** The SPSF adsorbent is a naturally available cost effective and eco-friendly adsorbent which has not been extensively studied for the removal of frequently used pesticides such as diazinon. The findings of the study revealed that the SPSF adsorbent can be considered as a promising remedy for house hold purification of diazinon contaminated waters.

Keywords: Adsorption; diazinon; Strychnos potatorum seed flakes; water

1 Introduction

Usage of pesticides is the most cost effective method of pest control. Organophosphorus pesticides (OPs) are the most widely used pesticides in agriculture [1]. Diazinon is a type

of OPs that is used to control insects in vegetables and fruits[1–3]. With the purpose of increasing the crop yield through effective pest control, usage of OPs including diazinon has increased during the recent past. This has led to increase the risk of environmental contamination of diazinon[1,3]. The residues of this pesticide could be found in water bodies and entering these pollutants into drinking water may cause numerous adverse health effects[4]. The harmful effects depend on exposure time, input concentration, chemical material type and the amount of venom[4,5]. The half-life of diazinon pesticide is ranged between 70 h to 12 weeks in water and 10 to 200 days in soil[1]. Also, this is easily absorbed into skin and it could result severe nervous disorders. Thus, it is essential to remove these contaminants in water sources[3]. Therefore, to achieve this, different strategies such as usage of granular activated carbon[2,3,6], multi-walled carbon nanotubes[7], Fe_3O_4/SiO_2 core/shell nanocrystals[8], magnetic bentonite nanocomposites[9], iron modified montmorillonite[10] and microbial degradation[1,11] have been used in previous studies. Adsorption is an effective method of removing pollutants in terms of low cost, flexibility and simplicity of design, and ease of operation[12].

Strychnos potatorum is a medium sized tree that grows in southern and central parts of India, Sri Lanka, and Burma. This tree is also known as clearing nut tree due to the purification ability of water by their seeds. Furthermore, the seeds of these trees are non-toxic and commonly used in ayurvedic medicine[10,11]. According to the Sanskrit writings from India, the seeds of *Strychnos potatorum* have been used for clarifying the turbid surface water over 4000 years[13]. Therefore, *Strychnos potatorum* seeds (SPS) are effective coagulant aids. This property is due to the presence of polyelectrolytes, carbohydrates, lipids and alkaloids containing –COOH and free –OH surface groups[11,14]. SPS have been used to remove various contaminants in water such as heavy metals, anions and dyes[12,13,15–18]. However, none of the studies has focused on the removal of pesticide in water by SPS. And also, SPS are good adsorbent for domestic water purification due to its readily availability and eco friendliness.

Thus the current study was carried out to determine the efficiency of *Strychnos potatorum* seed flakes (SPSF) in removing diazinon residues in water. Adsorption kinetics and isotherm models were used to describe the experimental equilibrium data.

2 Material and methods

2.1 Preparation of adsorbent

SPS were collected from a local Ayurvedic shop in Sri Lanka. Seeds were washed thoroughly with hot distilled water (~50°C) three times to remove dust and other impurities, and dried at 40°C for 2 days in hot air oven. It was then grounded and *Strychnos potatorum* seed flakes (SPSF) were prepared.

2.2 Characterization of the adsorbent

Surface morphology of SPSF and SPS powder was examined by Scanning Electron Microscopy (VEGA3 SEM, TESCAN). Furthermore, Fourier Transform Infrared (FTIR) spectrometry (PerkinElmer Corporation, Norwalk, CT) was used to elucidate the functional groups present in SPSF.

2.3 Preparation of diazinon solutions

Diazinon (98% purity) was purchased from Sigma Aldrich, USA. A Stock solution of diazinon (100 mg/L) was prepared by dissolving diazinon (10.00 mg) in acetonitrile (100.0 ml) and stored at ~10°C in the dark. Stock solution was further diluted with acetonitrile to obtain standard solutions for the instrumental calibration. All the glassware used was washed with deionized water and acetone.

2.4 Determination of diazinon concentrations

High Performance Liquid Chromatography (HPLC) analysis of diazinon was isocratically performed in a reversed-phase Zorbax Eclipse plus C18 Column with a mobile phase consisting of acetonitrile/water (65/35, v/v) at ambient temperature. The injection volume was 10 μL and the flow-rate was 1 ml/min (1260 Infinity, Agilent Technologies, Palo Alto, CA). The wavelength of 245 nm was used as the wavelength of the UV detector[19].

2.5 Batch experiments

Batch adsorption experiments were carried out in 250 mL glass conical flasks by shaking 0.5g of SPSF in a 100mL of diazinon solution (15mg/L) of pH 5.6 (which was prepared using deionized water as the solvent just before the analysis) on a rotary shaker at 200 rpm for 15 min at 25 °C. The effect of contact time, biomass, initial concentration and pH were investigated in this

study. After the adsorption experiment, SPSF free solutions were obtained by centrifugation at 10,000rpm for 10 min (MX-207, Tomy Seiko Co.,Ltd., Tokyo, Japan).

Diazinon concentrations were analyzed using HPLC under above mentioned conditions. The correlation coefficient (R^2) of the calibration curve was between 0.988-1.000. To check the reproducibility, all experiments were carried out in duplicate. The average values were taken for the data analysis.

2.6 Isotherm experiments

Isotherm experiments were carried out using 5g/L of SPSF at pH 5.6,200 rpm and 25°C. Isotherm experiments were carried out in the diazinon concentration range of 5-50mg/L for 30 min.

3 Results and Discussion

3.1 Characterization of the adsorbent

3.1.1 SEM analysis
SEM images of SPSF and SPSpowder are shown in Figure 1 . As can be seen in the Figure 1(A), the surface of SPSF is uneven, layered and rough with a smaller number of cavities like structures. Nevertheless, in powder form (Figure 1(B)), layered and cavities like structures are not seen, while keeping the rough nature on the surface. Having a layered and cavity like structures would be much more beneficial for an effective adsorption. Therefore, the adsorption studies were performed using SPSF.
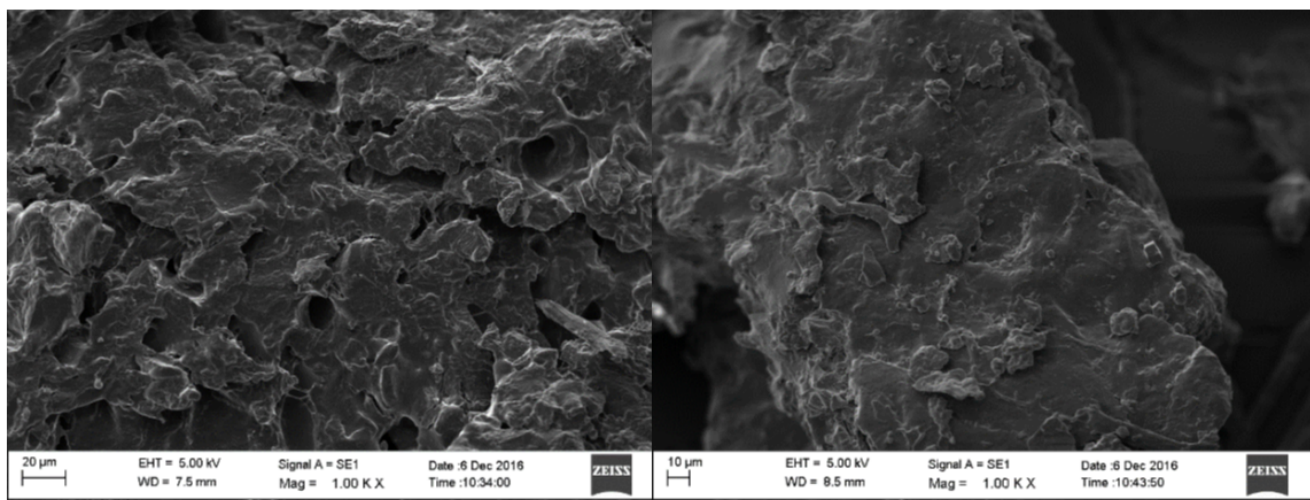

Fig 1. SEM images of (a) *Strychnos potatorum* seed flakes (SPSF) and (b) *Strychnos potatorum* seed powder

3.1.2 FTIR analysis
The FTIR spectrum of SPSF is shown in Figure 2. The strong broad band in the region of 3200-3650 cm^{-1} is due to the intermolecular hydrogen bonded O-H stretch and the peaks at about 1640 cm^{-1} are due to the C=O stretching of amides. Moreover, the peaks between 1032 cm^{-1} and 1006 cm^{-1} may be due to the stretching vibration of C-O group in alcohol and carboxylic acid. And also, peaks between 1640-1550 cm^{-1} are due to N-H bending of amines and amides. In addition, the peak around 2927cm^{-1} is due to C-H stretching of CH_2 moiety.
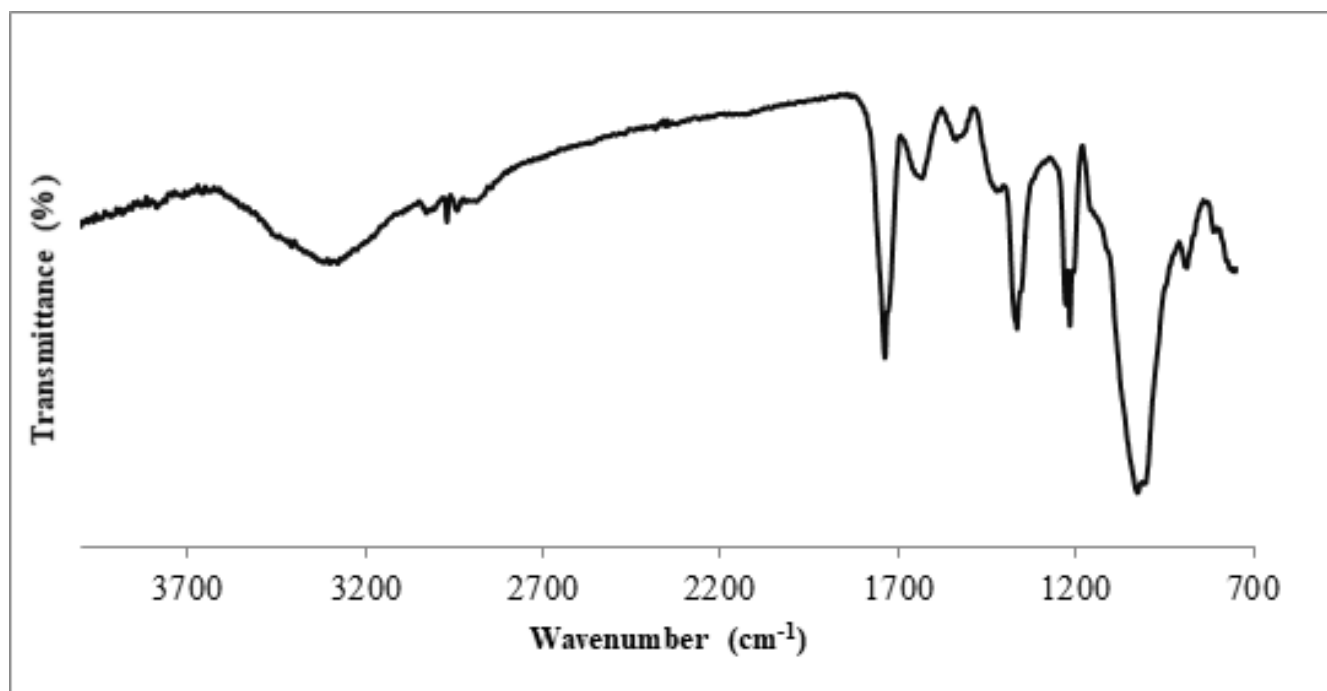
Fig 2. FTIR spectrum of SPSF

3.2 Effect of contact time

The effect of contact time on the removal of diazinon was investigated and shown in Figure 3 A. The rate of adsorption of diazinon was very high in the beginning while maximum removal was at 10 min. It was assumed that one active site of the adsorbent can adsorb only one diazinon molecule in a monolayer and the number of active sites in an adsorbent is fixed[14]. Therefore, a rapid adsorption was observed at the beginning and adsorption rate decreased with the reduction of available active sites. Since the maximum adsorption was obtained in few minutes, the adsorbent can be considered asan effective adsorbent for diazion removal in aqueous solutions.

3.3 Effect of concentration

The effects of initial diazinon concentration on the adsorption capacity of SPSF and on the percent adsorption of diazinon are shown in Figure 3B. It is clear that, when increasing the diazinon concentration from 5-15 mg/L, the adsorption of diazinon to SPSF was gradually increased. However, with further increasing the diazinon concentration from 15 mg/L to 50 mg/L, there was no significant further increment in the adsorption of diazinon. This may be due to the saturation of adsorbent sites on SPSF. Furthermore, the percent diazinon adsorption has decreased with increasing the initial diazinon concentration.

3.4 Effect of biomass

Biomass is a very important factor that needs to be considered for effective diazinon adsorption. Diazinon removal at different dosage of SPSF(0.1-2 g) was investigated at pH 5.6 while keeping the concentration and volume of the diazinon solution (100 mL) constant. The obtained results are presented in Figure 3C and the percent of diazinon removal had increased with adsorbent dose. This is due to more availability of active adsorption sites with the increase of adsorbent mass. Previous studies also reported similar behavior to this study[3,6,9,10]. However, after the dosage of 0.5g, there was no significant diazinon reduction observed.

3.5 Effect of pH

The pH of the medium is an important parameter in the adsorption process. Therefore, the effect of pH on the diazinon removal was investigated from pH 3 to pH 8. At pH 3 and pH 5, a significant reduction in the diazinon concentration was observed in the control samples which did not containany SPSF. Therefore, the effect of decomposition of diazinon was considered in

calculating the percentages of diazinon removal. Accordingly, previous studies have also reported the acceleration of diazinon decomposition in an acidic solution[6,20]. However, at pH levels of 6, 7 and 8 there is no diazinon reduction in control samples and maximum diazinon adsorption to SPSF was obtained at pH 6. The effect of pH on the diazinon adsorption is shown in Figure 3D.

The diazinon adsorption to SPSF increased from pH 3 to pH 6 and the maximum adsorption was obtained at pH 6. However, the adsorption Effect of diazinon to SPSF has decreased after pH 6. In contrast, some studies have revealed that the percentage of pesticide removal in the presence different adsorbents is higher at acidic pH values and decreased with further pH increment towards alkaline pH[21]. However, those studies have not considered the possible decomposition of diazinon in acidic conditions and only the overall diazinon removal was taken for the calculation of percentage of diazinon removal. Nevertheless, the study on the investigation of diazinon pesticide removal from contaminated water by adsorption onto NH₄Cl-induced activated carbon[22] has also considered the potential diazinon decomposition and observed a similar trend to this study.
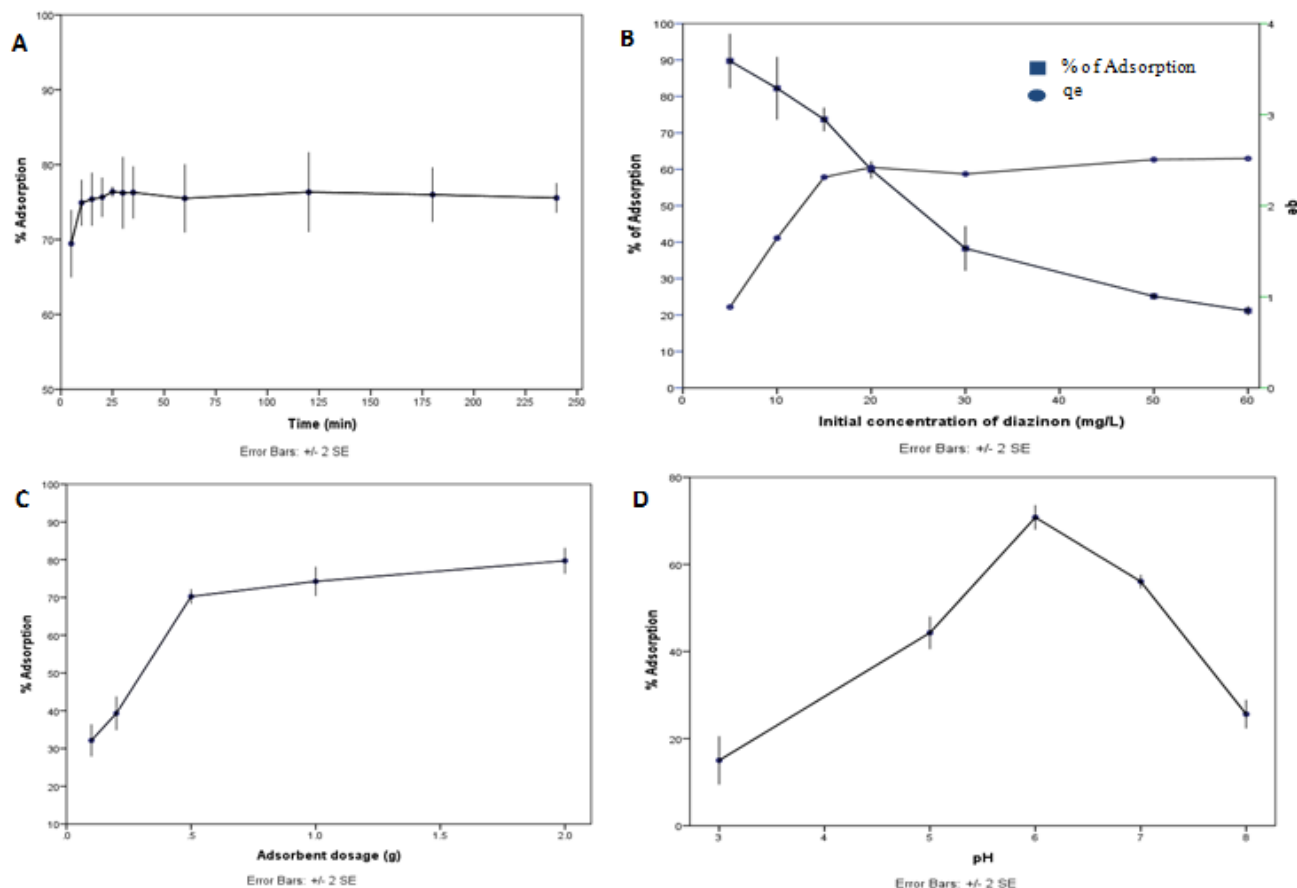
Fig 3. (A) Effect of contact time on % removal of diazinon by the adsorbent SPSF (Initial diazinon concentration = 15 mg/L , temperature = 25 °C, adsorbent dosage = 0.5 g, pH = 5.6) (B) Effects of initial diazinon concentration on the adsorption capacity (q_e) of SPSF and the percent adsorption of diazinon(adsorbent dose 0.5 g, contact time = 15 min, temperature: 25 °C,pH = 5.6) (C) The percent of diazinon removal by the adsorbent at different adsorbent dosages of SPSF (Initial diazinon concentration = 15 mg/L, temperature = 25 °C, contact time = 15 min, pH = 5.6) (D)The percent of diazinon removal by the adsorbent at different pH levels(Initial diazinon concentration = 15 mg/L, temperature = 25 °C, contact time =15 min, adsorbent dose 0.5 g

3.6 Sorption isotherms of diazinon on *Strychnos potatorum* seed flakes

The diazinon adsorption capacity of SPSF at different diazinon concentrations (5-50 mg/L) were tested in order to evaluate the diazinon removal mechanisms by Langmuir and Freundlich isotherm models while using fixed amount of adsorbent (0.5 g)at pH 5.6 by shaking at 200 rpm for 15 min.

The amount of diazinon retained in the adsorbent phase was calculated using the following equation (1); where q_e is the amount of absorbate adsorbed per gram of the adsorbent at equilibrium (mg/g), C_o is the initial concentration of adsorbate (mg/L) C_e is the equilibrium concentration of adsorbate (mg/L), V is the volume of the diazinon solution (L) and M is the adsorbent dosage (g).

$$q_e = (C_o - C_e) V M^l \qquad (1)$$

The linear form of the Langmuir isotherm model is expressed with equation (2); where, q_e is the amount of adsorbate adsorbed per gram of the adsorbent at equilibrium (mg/g), q_{max} is the maximum monolayer coverage capacity (mg/g), K_L is the Langmuir isotherm constant (L/mg) and C_e is the equilibrium concentration of adsorbate (mg/L).

$$\frac{C_e}{q_e} = \frac{1}{q_{max} K_L} + \frac{C_e}{q_{max}} \qquad (2)$$

Langmuir model was proposed by Irving Langmuir in 1916, describing homogeneous and independent monolayer adsorption[23]. According to Saadi et al. (2015), Freundlich adsorption isotherm is an empirical model and it can be applied to multilayer adsorption in a heterogeneous surface.

The linear form of the Freundlich isotherm model is expressed with equation (3); where q_e and C_e are the amount of adsorbate adsorbed per gram of the adsorbent at equilibrium (mg/g) and the equilibrium concentration of adsorbate (mg/L), respectively.

$$log q_e = \frac{1}{n} log C_e + log K_f \qquad (3)$$

Freundlich isotherm parameters of n and K_f (mg/g) are related to intensity of adsorption and adsorption capacity, respectively[24].

The obtained results were well fitted to the Langmuir isotherm model with R^2=0.999 (Table 1). Langmuir isotherm model describes adsorption occurring on homogeneous surface by monolayer adsorption without any kind of interaction with adsorbate[14]. This isotherm model provides details about maximum adsorption capacity, q_{max} and in this study it was found to be 2.556 mg/g. Maximum adsorption capacity (q_0) values reported for the adsorptions of diazinon onto different adsorbents recorded in the literature are given are Table 2.

Table 1. sotherm parameters ofdiazinon adsorption on SPSF

Isotherm parameters	Langmuir constants	q_o(mg/g)	2.556
		K_L(L/mg)	1.178
		R^2	0.999
	Freundlich constants	K_f(mg/g)	1.330
		n	4.950
		R^2	0.760

Table 2. Langmuir constants (q_o) for adsorption of diazinon onto various types of adsorbents reported in the literature

Adsorbent	(q0) Maximum adsorption capacity (mg/g)	References
Rhizopusarrhizus activated sludge	0.5	(John & Bell, 1987)
sodium hydroxide coconut shell biochar	0.63	(Baharum et al., 2020)
activated coconut shell biochar	1.73	(Baharum et al., 2020)
Activated Bentonite	5.56	(Ouznadji, Sahmoune, & Mezenner, 2016)
SPSF	2.5	Present study

3.7 Separation factor

The separation factor was calculated using the Langmuir isotherm model and that is useful for predicting the affinity between adsorbent and adsorbate[25]. The equation is expressed as follows;

$$R_L = \frac{1}{(1 + K_L C_0)} \qquad (4)$$

Where R_L is the separation factor, K_L is the constant in Langmuir equation and C_o is the initial concentration of adsorbate. Separation factor (R_L) points out the nature of the sorption process and shape of the isotherm: $R_L = 0$, irreversible; $0 < R_L < 1$, favorable; $R_L = 1$, linear; and $R_L > 1$, unfavorable(14). The R_L values were determined using the above equation (Equation (4)) and it was found to be 0.014-0.145 (Table 2). These R_L values thus obtained were in the range of 0-1 and that showed a favorable adsorption onto the adsorbent [26].

3.8 Adsorption Thermodynamics

To evaluate the thermodynamic feasibility and to assess the spontaneous nature of the adsorption process, adsorption thermodynamic parameters were calculated. Those thermodynamic parameters are free energy change (ΔG^o), enthalpy change (ΔH^o) and entropy change (ΔS^o).

The Gibbs free energy change (ΔG^o) value was obtained using equation (5).

$$\triangle G^O = -RT \ln K_C^0 \tag{5}$$

$$\ln(K_C) = \frac{\triangle S^O}{R} - \frac{\triangle H^O}{RT} \tag{6}$$

where, K_c° is the equilibrium constant and it is the ratio of the amount of diazinon adsorbed on the SPSF at equilibrium (q_e, mg/g) to the remaining diazinon concentration in the solution at equilibrium (C_e, mg/L), R is the universal gas constant and T is the absolute temperature in Kelvin (K)(14). The Gibbs free energy change (ΔG°) value is negative for the diazinon concentration range of 5-20 mg/L (-5.545kJmol^{-1} to -0.991 kJmol^{-1}) indicating a spontaneous nature of adsorption. Generally, ΔG° values between 0 to -20 kJmol^{-1} and -80 to -400 kJmol^{-1} indicate a physisorption and chemisorption, respectively [27]. Therefore, according to the results the adsorption can be considered as a physisorption. However, when increasing the diazinon concentration from 30 to 60 mg/L, the ΔG^o value was positive and that indicated a non-spontaneous nature of adsorption (Table 3). To be an effective adsorbent, the adsorption process should be spontaneous. In agricultural practices pesticides are applied in relatively small quantities and the contamination of such pesticides to water bodies may be in residual amounts. Therefore, SPSF can be considered as a potentially effective adsorbent for the removal of diazinon residues in water bodies due to the spontaneous nature and better adsorption capacities shown at low diazinon concentrations. The functional groups revealed by the IR study of SPSF may accountable for this performance. Hydrogen bonding and certain dipole – dipole interactions can be considered as possible interactions between the SPSF and diazinon.

Table 3. Separation factor (R_L) and Gibbs free energy (ΔG°) values at different diazinon concentration levels at 25 $^\circ$C

Initial diazinon concentration (mg/L)	RL	Gibbs free energy (ΔG°) (kJ)
5	0.145	-5.545
10	0.078	-3.868
15	0.054	-2.558
20	0.041	-0.991
30	0.028	1.190
50	0.017	2.704
60	0.014	3.260

Table 4. Dependence of Gibbs free energy (ΔG°) values on temperature for the of diazinon adsorption by the SPSF

Temperature	Gibbs free energy (ΔGo) (kJ)
20 $^\circ$C	-2.295
25 $^\circ$C	-2.008
30 $^\circ$C	-0.838
40 $^\circ$C	2.973

As seen in Table 4, the values of ΔG^o decreased with the increase in temperature and ended up with a positive value at 40 $^\circ$C. This indicated that adsorption was more favorable at relatively lower temperatures. Based on Equation (6), the calculated ΔH^o and ΔS^o values are -84.192kJmol^{-1} and -277.222JK^{-1} mol^{-1} respectively. The negative values of ΔH^o and ΔS^o indicated that the adsorption reaction was exothermic and enthalpy driven [28].

4 Conclusion

The adsorption of organophosphate pesticide, diazinon on *Strychnos potatorum* seed flakes was examined under different operational variables. It was found that the adsorption process greatly depends on the adsorbent dosage, contact time, pH, and initial diazinon concentration. The optimum adsorption for 100mL of diazinon solution (15mg/L) using 0.5g of SPSF could be obtained in 10min at pH 6. The experimental data was best described by Langmuir isotherm model. According to the FTIR analysis there are various functional groups that are potentially responsible for the adsorption. Since the maximum adsorption was obtained within few minutes, this readily available eco-friendly, material can be considered as an effective adsorbent for the removal of diazinon residues at ambient temperature and pH. Also, this effective natural adsorbent may be further modified to remove various contaminants in water in large scale.

Acknowledgment

Conflict of Interest

The authors declare no conflict of interest.
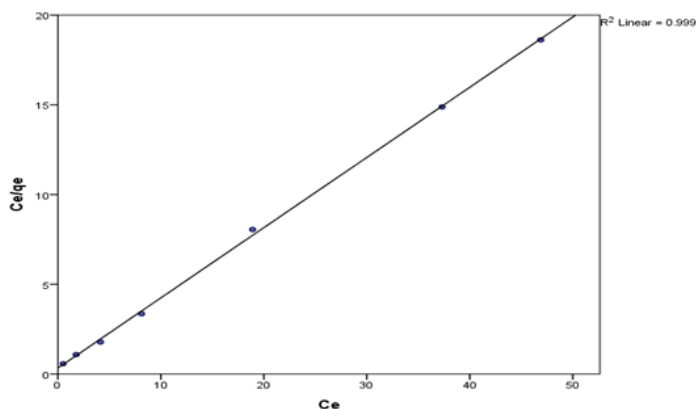
Supplementary material

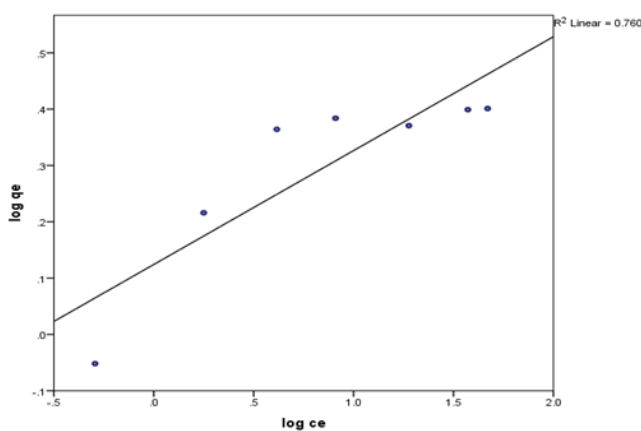**Fig 4.** SM1 Langmuir adsorption isotherm for the diazinon adsorption on to SPSF

Fig 5. SM2 Freundlich adsorption isotherm for the diazinon adsorptionon to SPSF

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