Original Article

# Removal of Diazinon Pesticide from Aqueous Solutions by Chemical–thermal-activated Watermelon Rind

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## Abstract

**Aim**: The aim of this study was to reduce the amount of diazinon from aqueous media using chemical–thermal-activated watermelon rind. **Materials and Methods:** This experimental study was carried out in a laboratory. First, watermelon rind was activated by chemical–thermal method. Then, the effective parameters of the diazinon adsorption process, including the initial concentration of diazinon (0.17–1 µg/L), pH (3–10), adsorbent amount (0.05–1 g/l), and contact time (30–100 min), were investigated and optimized. The amount of residual diazinon was measured by high-performance liquid chromatography. In this study, Taguchi method was used to determine the sample size and statistical analysis. Furthermore, in this study, to describe the adsorption equilibrium, Freundlich and Langmuir isotherm models were used. **Results:** The results showed that under an optimal pH of 6, the equilibrium time of 30 min, the amount of adsorbent 1 g/L, and the initial concentration 0.17 µg/L, the elimination efficiency of diazinon was 95.1%. Furthermore, the results of isothermic studies have shown that the removal of diazinon follows the Freundlich model ($R^2 = 0.921$). **Conclusion:** Chemical–thermal-activated watermelon rind can effectively be used to remove low concentrations of diazinon from aqueous solutions.

**Keywords:** Adsorption, diazinon, watermelon rind

## Introduction

Today, a wide range of pesticides, including insecticides, fungicides, herbicides, and nematicides, are used in agriculture and horticulture. The extensive presence of these pesticides in the environment is a serious problem.[1,2] In most parts of the world, increasing levels of pesticides in surface and groundwater sources are recognized as a threat to the quality of water resources. Water pollution with insecticides in addition to contaminating the aquatic food chain may eventually find its way into drinking water and destroy the lives of humans and other beings directly and indirectly.[2] Organophosphorus compounds are among the most common pesticides in the world, including Iran.[3,4] These toxins are among the effective factors on the nervous system that inhibit the activity of acetylcholine esterase enzyme activity.[5,6] Diazinon pesticide ($C_{12}H_{21}N_2O_3PS$) is an organophosphorus compound made in 1952 and widely used as insecticide, acaricide, and nematicide.[1,7] This poison is one of the most commonly used organophosphorus insecticides in the world. 6 million pounds of diazinon are used on agricultural land in the United States per year.[8] Diazinon is a nonsystemic insecticide used in agriculture to control the soil, herbs, and fruits of the plants against insects and pests.[5] Diazinon is relatively stable and fluid in the environment. Depending on the soil environment, it can remain in soil for weeks to months. It has a dissolution potential in water and can penetrate into the soil and enter the groundwater.[9,10] Diazinon is present in drinking water and in almost all sea water samples. Diazinon is stable at pH = 7 and does not easily evaporate from soil or water. Therefore, it can remain in the environment for more than 6 months.[1,11] The half-life of diazinon is 80 days in aqueous solutions.[9] Some of the chemical and physical properties of diazinon are shown in Table 1.[12] The World Health Organization has categorized diazinon as 2$^{nd}$ class poisons. The maximum allowed diazinon concentration is set as 0.1 µg/L and total

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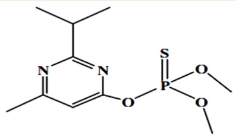
**Table 1: Chemical structure and characteristics of diazinon**

| Structure | Mw (g/mol) | Density at 20°C (g/cm³) | WHO class | Vapor pressure (mmHg at 20°C) | Henry's law constant (atm.m³/mol) | Maximum solubility in water (ppm) |
|---|---|---|---|---|---|---|
|  | 304.3 | 1.11 | II | $1.4\times10^{-6}$ | $1.4\times10^{-6}$ | 40 |

insecticide is considered as 0.5 µg/L by the European Union.[6] Since many water resources in Iran are faced with the problem of excessive concentration of pesticides above the standard level, in addition to adopting strategies to prevent further contamination of these sources, appropriate and reliable solutions should be considered to treat them. So far, many methods have been used for the removal of diazinon such as treatment with photocatalyst, advanced oxidation, biological treatment, membrane filters, and ion exchange.[13-16] Each of these methods has its own advantages and disadvantages. The adsorption method is one of the most effective physical methods for removing pollutants from the environment due to simple design, low cost of the required materials, ease of operation, proper maintenance, lack of need for final treatment, and cost-effectiveness.[17,18]

The activation process is usually performed within temperature ranges from 400°C to 600°C and from 700°C to 1200°C for chemical and physical activations, respectively. During the pyrolysis process, chemical activation can be carried out at a temperature range of 400°C–700°C using inorganic compounds. On the other hand, the temperature range for physical activation with steam or $CO_2$ is from 700°C–1200°C, which means more power consumption activated carbon by chemical or physical activation. Activated carbon was chemically activated by KOH, $ZnCl_2$, $H_3PO_4$, and $H_2SO_4$.[19]

Today, the use of adsorption to remove some of the major pollutants (pesticides) from water resources is increasing. Taking advantage of agricultural waste such as coconut, walnut and almond shell, sugar beet and sugarcane pulp, and wheat bran are considered by the researchers as inexpensive and natural adsorbents in the removal of pollutants from aqueous solutions.[18,20-22] Watermelon is abundantly produced and harvested in Iran. Thus, using watermelon rind as adsorbent is justifiable due to being cheap, abundantly available, eco-friendly, and easily applicable. Diazinon is also widely used in the fruit gardens of the study area. Therefore, the aim of this study is to investigate the removal of diazinon pesticide from aqueous solutions by chemical–thermal-activated watermelon rind. In this study, the effect of different parameters such as concentration of diazinon, contact time, pH, and adsorption concentration are studied on the diazinon removal.

# Materials and Methods

In this experimental study, the chemical–thermal-activated watermelon rind is used to remove diazinon from aqueous solutions in a laboratory scale. All materials used in this study were purchased from the Merck Co., Germany.

## Adsorbent preparation

Watermelon rind was washed several times with distilled water after separation to remove impurities. The sample was then placed in an oven at 110°C for 6 h. Dried watermelon skin was crushed with household grinder and passed through a standard 30 mesh to provide a uniform powder.

## Thermal–chemical adsorbent activation

For chemical activation of watermelon rind, the prepared powder was washed with deionized water and placed in 0.1 M nitric acid for 1 h. To remove organic and inorganic material in the adsorbent, the sample was immediately placed in methanol for 1 h. The adsorbent was placed in an electric furnace at 300°C for 1 h to be activated thermally and increased contact surface. The activated specimen was crushed in a porcelain pounder and passed through a sieve with a standard mesh of 30–100, to obtain a uniform powder.[20,23]

## Adsorption experiments

All experiments were carried out in a discontinuous reactor (ARLEN). In order to obtain a solution of water with a certain concentration of diazinon, the diazinon with 100% purity was used. The amount of 100 ml of standard diazinon solution (0.17, 0.3, 0.6, and 1 µg/L) was poured into a 250 ml flask and a certain amount of activated adsorbent (0.05, 0.1, 0.4, 0.6, and 1 g) was added to it. The pH of the sample was adjusted by adding drops of hydrochloric acid 0.1 normal or sodium hydroxide 0.1 normal in the amounts of 3, 6, 7, 8, and 10 using the pH meter model (METTLER, Model Mp230).

These experiments were carried out at the stirrer rate of 100 rpm at 30, 60, 90, and 100 min retention times. Then, acetate cellulose paper with pore size of 0.45 µm was used to separate the adsorbent. Diazinon extraction was carried out by solid-phase extraction (SPE) cartridge. The SPE sorbent was conditioned with 3 ml methanol followed by 3 ml water and then loaded with 1ml of extract. Finally, the specimen was kept in −5°C and analyzed by high-performance liquid chromatography (Agilent 1200HPLC) equipped with C18 analytical column (150 mm_ 4.6 mm, 5 mm), used in isocratic mode (1 mL/min) with FID detector for <24 h to read the residual diazinon The mobile phase included methanol and water (10/90 V/V) with a flow rate of 1 mL/min. The retention time for diazinon was 3.16 min. The detection limit for the sample was 0.01 μg/L. The validation study was tested to assess for linearity, recovery, precision, and limits of detection and limits of quantitation. The linearity of the method was studied applying matrix-matched calibrations by analyzing six concentration levels, between 0.1 and 1 μg/l. For the determination of mean recoveries (to estimate the accuracy of the method) and precision (repeatability, expressed as coefficient of variation in %), four spiked blank samples at concentration levels of 0.17, 0.3, 0.6, and 1 μg/l were prepared and then treated according to the procedure earlier described in sample preparation. Finally, the diazinon percentage removal was calculated using the following equation:[20]

$$\text{Removal efficiency}(\%) = (1 - C_t / C_0) \times 100 \quad (1)$$

where $C_0$ and $C_t$ are the initial and final concentrations of diazinon.

In this study, Freundlich and Langmuir isotherm models were used to describe the equilibrium state in adsorption between solid and liquid phase. The linear equation of the Langmuir model is based on Eq. (2).

$$\frac{1}{q_e} = \frac{1}{q_m k_L C_e} + \frac{1}{q_m} \quad (2)$$

The linear form of the Freundlich relation is written Eq. (3)

$$\text{Log } q_m = \text{Log } k_f + \frac{1}{n} \text{Log } C_e \quad (3)$$

where $q_e$ is the amount of the absorbed material per unit mass of the adsorbent substance in mg/g, $C_e$ is the equilibrium concentration of the absorbed material in the solution after adsorption in mg/l, and $q_m$ and $K_L$ are Langmuir constants obtained by plotting $1/q_e$ against $1/C_e$. $k_f$ and n are Freundlich constants which are dependent on the capacity and adsorption intensity obtained by plotting the Log $q_e$ against Log $C_e$.[20,24,25]

Absorbent characterization was evaluated using BET test (temperature: 298 K, pressure: 0.88 atm). Furthermore, the absorbent morphology obtained by SEM images was investigated.

In this study, based on the Taguchi statistical method and the number of factors, 25 specimens were prepared for each concentration of pollutants that considering 4 concentrations and two repetitions of the test to ensure the accuracy of the data for each concentration, 50 samples were prepared and 200 were conducted. The results of these experiments were presented using Mini Tab and ANOVA and the Excel software was presented in the form of diagrams.[10]

# Results

## Adsorbent properties

Figure 1 shows the SEM image of the watermelon rind before and after the activation.

The results of the BET test are presented in Table 2.

## Effect of adsorbent parameters

In this study, the effect of contact time, initial concentration of diazinon, pH, and the amount of adsorbent was investigated on the diazinon removal process from aqueous solutions. Removal efficiency of diazinon at different conditions is shown in Table 3. The results of this study on the effect of pH of aqueous solutions on the diazinon removal efficiency with synthesized adsorbent, the effect of synthesized adsorbent on the removal efficiency, the effect of contact time on the removal efficiency, and the effect of the initial concentration of diazinon on the removal efficiency are shown in Figures 2-5, respectively. Furthermore, the mean ± standard deviation of removal efficiency of diazinon at different concentration is shown in Table 4.

## Adsorption isotherms

In this study, Langmuir and Freundlich adsorption isotherms were studied and their results are shown in Figure 6. Comparison of the $R^2$ values showed that the diazinon adsorption process follows the Freundlich model because of higher $R^2$ ($R^2 = 0.9261$).

The obtained value of 1/n Freundlich models between $0 < 1/n < 1$, which represents the adsorption, is desirable [Table 5].

# Discussion

In this study, the use of watermelon rind activation by chemical–thermal as an adsorbent was investigated for the removal of diazinon from aqueous solutions. The results of the SEM images [Figure 1] and the BET test [Table 2] showed that the chemical–thermal watermelon rind activation was done well. The raw watermelon rind has cavities of varying sizes, but its inner and outer surfaces almost smooth and even. However, the activated watermelon rind surface has fairly fine cavities. The adsorbent surface and volume in this case are increased due to the high number of cavities. The BET test showed that chemical–thermal watermelon rind activation increased the special surface 9 times higher and also increased the volume of total cavities in unit weight. In the study by Ahmad *et al*., BET surface area of watermelon rind-activated carbon was 776.65 $m^2/g$.[26] Polowczyk *et al*. reported that the highest adsorption capacities were obtained for adsorbents activated with chemical method.[27] Memon *et al*. reported that adsorbent is activated with chemical and thermal method.[28]

The pH of the solution is very effective in achieving the maximum removal rate. The pH of the solution can play the primary role in the adsorption and photocatalytic oxidation of pollutants. The catalyst surface will be charged negatively when pH > pHpzc, positively when pH < pHpzc,

**Table 2: Results of Brunauer-Emmett-Teller experiment**

| Type of adsorbents | Specific surface area ($g/m^2$) |
|---|---|
| Raw watermelon rind | 20.11 |
| Activated watermelon rind | 180.8 |

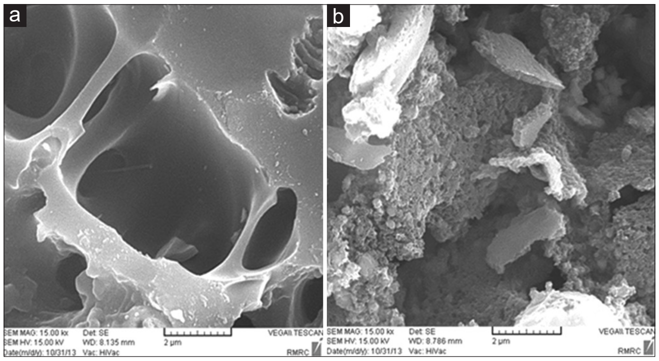



**Figure 1:** SEM images of the watermelon with 15,000 times magnification (a) before activation, (b) after chemical–thermal activation

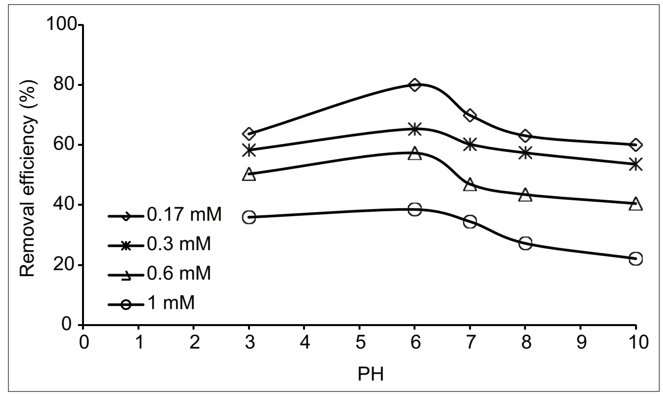



**Figure 2:** Effect of pH on diazinon removal efficiency

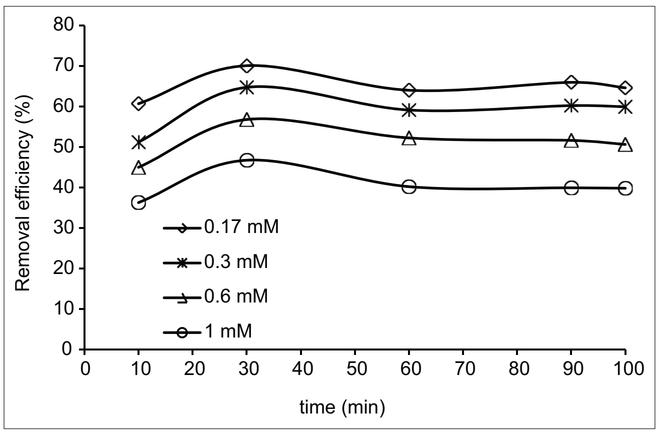



**Figure 3:** Effect of contact time on diazinon removal efficiency

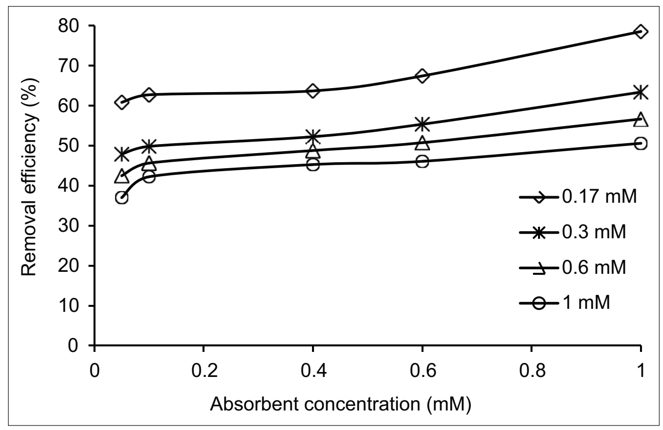



**Figure 4:** Effect of adsorbent dose on diazinon removal efficiency

and neutrally when pH ≈ pHpzc.[20] The effect of pH on the adsorption performance can, therefore, be explained in terms of electrostatic interaction between the catalyst surface and the target substrate. Diazinon is negatively charged above pH 2.6, as catalysts are positively charged below pH 7. Optimal conditions were found at which the positively charged activated carbon and negatively charged insecticide molecules should readily attract each other.[29] According to Figure 2, the highest amount of diazinon adsorption has occurred for all concentrations at pH = 6. The percentage of diazinon removal has had an increasing trend within the pH of 3–6 and after that, the removal efficiency decreased with increasing pH. The results show that the adsorption process has the highest productivity in the near-neutral pH. Memon *et al*. studied the removal of methyl parathion with an absorbent made of watermelon rind and obtained the highest adsorption at pH = 6 and near neutral.[28] Furthermore, similar results were obtained by Farmany *et al*.[18] In Samadi *et al*., the highest removal rate of diazinon was obtained at pH = 9.[30] Wang and Shin reported that the highest diazinon removal occurs at pH = 3.[1] Memon *et al*. reported that removal of methyl parathion pesticide is higher at acidic pH.[28] The reason for these differences is the toxin removal process.

In the study of the effect of contact time on the diazinon removal efficiency, the maximum removal rate was observed in the first 30 min of contact and after that, with a longer contact time, no significant effect was observed on the removal efficiency [Figure 3]. Therefore, this time can be considered as the equilibrium retention time. In fact, the adsorption efficiency increases slowly after 30 min due to the filling of adsorbent sites at the adsorbent surface and inside its pores as well as the reduction of the adsorbent specific surface. In Moussavi *et al*. (2013), it was observed that until 30 min, the amount of diazinon adsorption was increased and then reached a state of equilibrium.[17] This did not match with the results of previous studies. Hence, in the study by Bazrafshan *et al*., the maximum diazinon adsorption was 60 min which is more than the present study. The reason for this could be the difference in the process used to remove the diazinon toxin.[31]

Investigating the effect of adsorbent dose on removal efficiency in adsorption processes is important due to economic issues. In this study, 1 g/l adsorbent content has the highest removal efficiency. In fact, by increasing the amount of adsorbent, the number of active sites is increased and the diazinon molecules will have a greater chance of being trapped in these sites. Pirsaheb and Dargahi studied diazinon removal from aquatic media using granular-activated carbon and concluded that increasing the concentration of adsorbent increases the diazinon removal efficiency.[30] Similar results have also been reported by Akhtar *et al*.[32]

**Table 3: Removal efficiency of diazinon at different condition**

| Experiment number | Adsorption capacity (mg/g) | | | | Parameter | | |
|---|---|---|---|---|---|---|---|
| | 1 (μg/l) | 60 (μg/l) | 30 (μg/l) | 0.17 (μg/l) | Adsorbent dose (g/l) | Contact time (min) | pH |
| 1 | 0.06 | 0.031 | 0.059 | 0.038 | 0.05 | 10 | 3 |
| 2 | 0.055 | 0.018 | 0.056 | 0.03 | 0.1 | 30 | 3 |
| 3 | 0.058 | 0.025 | 0.042 | 0.024 | 0.4 | 60 | 3 |
| 4 | 0.05 | 0.02 | 0.048 | 0.027 | 0.6 | 90 | 3 |
| 5 | 0.053 | 0.028 | 0.051 | 0.028 | 1 | 100 | 3 |
| 6 | 0.052 | 0.026 | 0.039 | 0.04 | 0.1 | 10 | 6 |
| 7 | 0.049 | 0.028 | 0.044 | 0.039 | 0.4 | 30 | 6 |
| 8 | 0.046 | 0.019 | 0.03 | 0.036 | 0.6 | 60 | 6 |
| 9 | 0.04 | 0.012. 0.0 | 0.036 | 0.031 | 1 | 90 | 6 |
| 10 | 0.056 | 0.023 | 0.041 | 0.043 | 0.05 | 100 | 6 |
| 11 | 0.072 | 0.041 | 0.065 | 0.048 | 0.4 | 10 | 7 |
| 12 | 0.066 | 0.034 | 0.058 | 0.047 | 0.6 | 30 | 7 |
| 13 | 0.063 | 0.033 | 0.053 | 0.045 | 1 | 60 | 7 |
| 14 | 0.073 | 0.033 | 0.072 | 0.046 | 0.05 | 90 | 7 |
| 15 | 0.059 | 0.022 | 0.062 | 0.042 | 0.1 | 100 | 7 |
| 16 | 0.081 | 0.056 | 0.072 | 0.05 | 0.6 | 10 | 8 |
| 17 | 0.077 | 0.038 | 0.066 | 0.048 | 1 | 30 | 8 |
| 18 | 0.084 | 0.048 | 0.078 | 0.056 | 0.05 | 60 | 8 |
| 19 | 0.072 | 0.032 | 0.062 | 0.039 | 0.1 | 90 | 8 |
| 20 | 0.075 | 0.053 | 0.069 | 0.053 | 0.4 | 100 | 8 |
| 21 | 0.093 | 0.085 | 0.09 | 0.058 | 1 | 10 | 10 |
| 22 | 0.096 | 0.09 | 0.093 | 0.068 | 0.05 | 30 | 10 |
| 23 | 0.086 | 0.061 | 0.078 | 0.049 | 0.1 | 60 | 10 |
| 24 | 0.084 | 0.058 | 0.088 | 0.054 | 0.4 | 90 | 10 |
| 25 | 0.09 | 0.078 | 0.082 | 0.06 | 0.6 | 100 | 10 |

**Table 4: The mean±standard deviation of removal efficiency of diazinon at different concentration**

| Diazinon of concentration (μg/L) | Mean±SD (%) | pH | Time (min) | Adsorbent dose (g/L) |
|---|---|---|---|---|
| 0.17 | 95.1±1.4 | 6 | 30 | 0.1 |
| 0.3 | 91±2.7 | 6 | 30 | 0.1 |
| 0.6 | 88±1.2 | 6 | 30 | 0.1 |
| 1 | 82.3±2 | 6 | 30 | 0.1 |

SD: Standard deviation

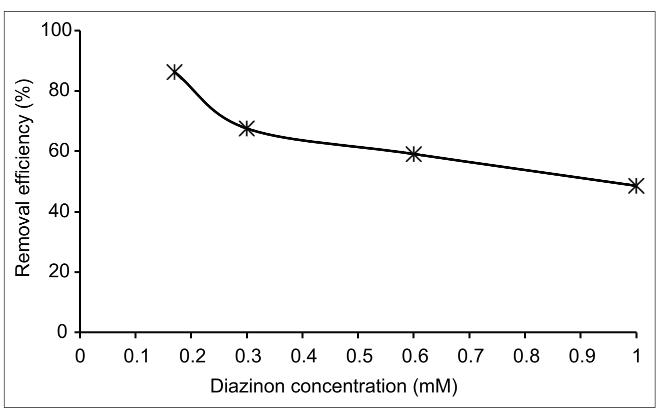



**Figure 5:** Effect of diazinon initial concentration on removal efficiency

In this study, the amount of removal was reduced by increasing the concentration of diazinon. In fact, by filling adsorbent cavities and decreasing the surface area, the adsorbent capability to absorb the toxin decreases until reaching a relative equilibrium and no adsorption occurs after that. The high rate of adsorption of diazinon in the first phase of the process and low concentrations may be due to the presence of active adsorption sites that quickly absorb toxin molecules. However, the number of these adsorption sites gradually decreases with increasing the process time and the increase in the number of toxin molecules adsorbed onto the adsorbent will increase, so that the rate of adsorption decreases significantly and leads to the formation of the second phase of adsorption.[33,34] Active adsorption sites are located in both surface and deep areas of the absorbent. Therefore, at the start of the adsorption reaction, all sites are ready to absorb, but surface sites are easily exposed to diazinon molecules and have a greater chance of exposure to diazinon molecules. Therefore, this increases the rate of adsorption, but gradually with the saturation of the surface and external sites, adsorption continues through deep and inward areas which will slow down the rate of

**Table 5: Results for Langmuir and Freundlich isotherms parameters**

| Parameters of Langmuir and Freundlich isotherms | | | | |
|---|---|---|---|---|
| Langmuir isotherm model | $\frac{1}{q_e} = \frac{1}{q_m k_L C_e} + \frac{1}{q_m}$ | $q_m$ (mg/g): 68.15 | $K_L$: 0.15 | $R^2$: 0.9261 |
| Freundlich isotherm model | $\text{Log } q_e = \text{Log } K + \frac{1}{n}\text{Log } C_e$ | K: 8.6 | $1/n$: 0.4844 | $R^2$: 0.8266 |

**Table 6: Comparison of adsorption capacities of diazinon with other adsorbents**

| Adsorbent | pH | Equilibrium time (min) | Maximum sorption capacity (mg/g) | Efficiency (%) | References |
|---|---|---|---|---|---|
| Bentonite | 5.2 | 60 | 5.56 | - | [38] |
| Granular-activated carbon | 6 | 50 | - | 88 | [39] |
| $NH_4Cl$-induced activated carbon | 7 | 30 | 250 | 97.5 | [17] |
| Activated Watermelon Rind | 6 | 30 | 68.15 | 95.1 | This study |

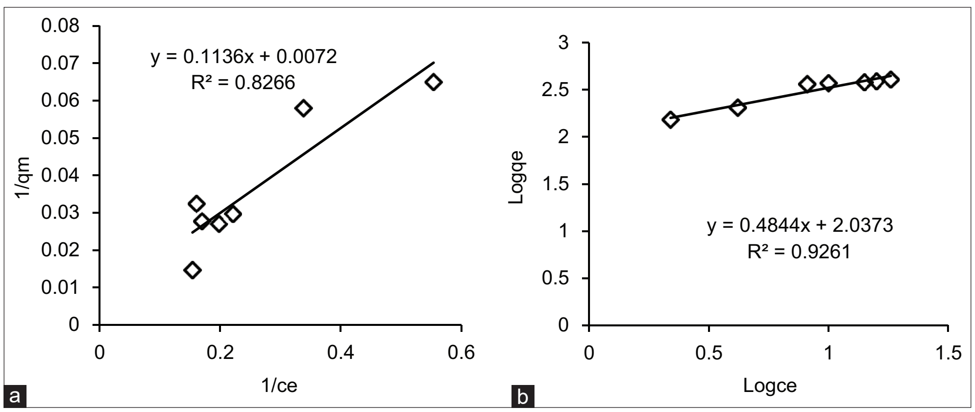



**Figure 6:** Langmuir (a) and Freundlich (b) isotherm models on diazinon removal efficiency

adsorption. Figure 4 shows that by increasing the concentration of diazinon, the amount of adsorption decreased to <1 µg/L. The results presented by Chaudhary *et al*. also confirm the issue that the removal efficiency decreases with increasing concentrations.[35] Akhtar *et al*. (2009) studied triazophos removal and observed that the removal efficiency decreased with increasing poison concentration.[32] Similar results were also observed in Memon *et al*. in endosulfan toxin removal.[36]

In this study, Langmuir and Freundlich adsorption isotherms were studied and comparison of the $R^2$ values showed that the diazinon adsorption process follows the Freundlich model because of higher $R^2$ ($R^2 = 0.9261$) [Figure 6]. Therefore, it can be said that the adsorbent surface has a heterogeneous state. However, in the study by Ouznadji *et al*., the equilibrium adsorption was best described by the Langmuir isotherm model.[37]

In the present study, activated watermelon rind has been compared with other adsorbents based on their maximum adsorption capacity for diazinon and shown in Table 6.

## Conclusion

In this study, the use of watermelon rind activation by chemical–thermal as an adsorbent was investigated for the removal of diazinon from aqueous solutions. Diazinon is one of the most commonly used organophosphorus pesticides in the world. This experimental study was carried out in a laboratory. The results of BET analysis and SEM images showed that the adsorbent chemical–thermal activation was performed in a good manner. The results of experiments on adsorption of diazinon from aqueous solution using chemical–thermal-activated watermelon rind showed that the best conditions for removing diazinon include concentration of 0.17 µg/L, 1 g/l adsorbent, 30 min equilibrium time, and pH = 6. The maximum removal efficiency in these conditions is 95.1%. The adsorption process is subject to Freundlich isotherm ($R^2 = 0.921$). Regarding the results of this study, it can be said that diazinon removal using chemical–thermal-activated watermelon rind can be converted into an efficient and reliable approach due to abundant watermelon rind availability in the country as an agricultural waste, simple system, low cost, and relatively desirable removal efficiency.

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Shahrekord University of Medical Sciences, Shahrekord, Iran.

## Conflicts of interest

There are no conflicts of interest.

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