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High-performance removal of diazinon pesticide from water using multi-walled carbon nanotubes

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ABSTRACT

Diazinon is one of the most commonly used organophosphorus pesticides in agricultural activities, and its access to groundwater and surface waters damages the animal and plant environments. This study aimed to evaluate the applicability of the adsorption process for the removal of organophosphorus diazinon pesticide from water using multi-wall carbon nanotubes. Specifications of multi-walled carbon nanotubes were determined using FTIR, SEM, TEM and BET technologies. The effects of different parameters of pH (4 and 7), contact time (1–15 min), pesticide concentration (0.3, 0.5 and 1 mg/L) and multi-walled carbon nanotubes' dose (0.1 and 0.3 g/L) were evaluated on the process of removing diazinon by multi-walled carbon nanotubes. Based on the experimental data obtained in this study, maximum removal of diazinon (100%) from water by multi-wall carbon nanotubes are the initial concentration of diazinon (0.3 mg/L), 0.1 g/L concentration of nano-adsorbent at both pH of 4 and 7 at the contact time of 15 min. The results of the study showed that multi-wall carbon nanotubes can be used as effective and high efficient for complete removal of diazinon pesticides from aqueous solutions.

1. Introduction

Today, the pollution of surface water and groundwater resources with pesticides is one of the most important environmental concerns. Pesticides are widely used in agricultural activities [1,2]. Organophosphorus compounds are one of the largest and most diverse pesticide groups, accounting for about 40% of pesticides in the world [3,4]. These pesticides are relatively inexpensive and have high efficacy in the removal of various types of pests [5].

Diazinon is an organophosphorus insecticide widely used in agricultural and animal husbandry activities for pest control. In the World Health Organization classification, diazinon is a medium-risk insecticide (class II), and the acute oral toxicity of this insecticide is 300 mg/kg [6]. The authorized amount of pesticides in water is about 0.5–0.1 μg/L [7].

This pesticide is a serious threat to human health due to its effect on cholinesterase activity and central nervous system disorder. In addition, severe intestinal contractions, chest tightness, blurred vision, headache, diarrhea, hypotension, coma, neurological and psychological complications, peripheral neuropathy, functional decline in psychological tests, and sensitivity to certain chemicals have been reported among the symptoms of poisoning with this pesticide [8–10].

Different methods for the removal of organophosphorus pesticides include photocatalysis, biochemical decomposition, electrochemical decomposition, separation with a variety of membranes, oxidation and adsorption [11]. Among different types of methods, the adsorption process has been considered as a promising method in the process of removing pesticides from aqueous solutions due to its simplicity, eco-friendliness and efficient performance. [12–14]. Various types of adsorbents including silica particles [15], magnetic materials [16],

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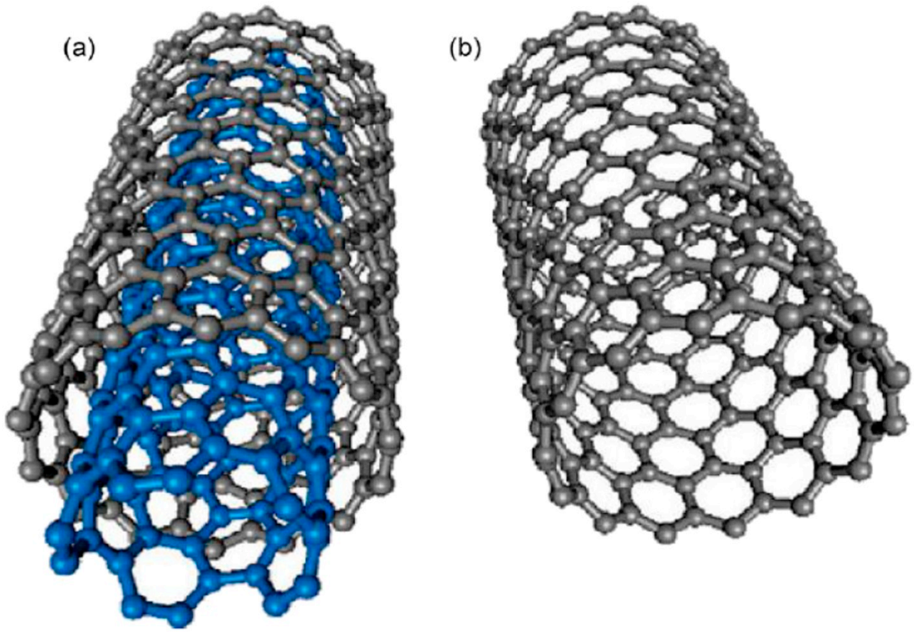


Fig. 1. (a) MWCNT structure, (b) SWCNT structure.

organic porous polymers [17], carbon nanotubes [18], and nano-adsorbents [7] have been reported for the rapid removal of pesticides from aquatic environments.

Comparison of different adsorbents shows that carbon nanotubes have higher potential for the removal of organic compounds and environmental protection than other adsorbents [7,20]. Carbon nanotubes have a very high specific surface area, high permeability, and good thermal and mechanical stability. Due to their high surface area and the layered and hollow structure, they have high adsorption capacity and can be used as organic and non-organic absorbers. [19,21–23]. Carbon nanotubes are divided into two broad categories of single-walled carbon nanotube (SWCNT) and multi walled carbon nanotubes (MWCNT) (Fig. 1).

The purpose of this study was to investigate the effectiveness of multi-walled carbon nanotubes on the removal of diazinon pesticide from aqueous solutions, and the key parameters in evaluating the adsorption process under different laboratory conditions including pH, contact time, initial concentration of diazinon pesticide and adsorbent dose were investigated.

2. Materials and methods

2.1. Chemicals

All the chemicals used in this study were of analytical grade. The used materials including methylene chloride solvent, acetone, hexane, anhydrous sodium sulfate, sodium hydroxide and chloridric acid were prepared from the German company, Merck. The standard diazinon (with a purity of 95% and the chemical formula $C_{12}H_{21}N_2O_3PS$ and the IUPAC name of O, *O*-diethyLO- (2-iso proply-6 methyl) pyrimidin-4-yl) phosphorathioate) used by the American Standard Company was prepared from Modiseh Novin Company and the commercial diazinon (with a purity of 60%) was prepared from Partonar company.

2.2. Adsorbent properties

Multi Walled Carbon Nanotubes (MWCNTS) used in this study (95% purity, specific surface area of 370 m^2/g, outside diameter of 8–15 nm, inside diameter of 3–5 nm, and length of ~50 nm) were purchased as an adsorbent for diazinon from Neutrino Company. In this study, the surface morphology of multi-walled carbon nanotubes was determined by scanning electron microscopy (SEM), Philips XL30 model and the transmission electron microscopy (TEM) JEOL JEM-200CX model, Japan, as well as the specific surface area of multi-walled carbon nanotubes was determined by BET, model (Gemini 2357, Micrometrics Instrument Corporation, USA). The FT-IR experiments were carried out by the American Thermo Nicolet, Avatar 360 -FTIR ATR model, in order to identify the quality of the unknown samples, the type of functional groups and bonds in the multi-walled carbon nanotube molecules, and the FTIR spectrum with a resolution of 4 cm^{-1} in the range of 500–4000 cm^{-1}.

2.3. Adsorption tests

At first, a 1000 mg/L stock solution of diazinon was prepared and the required solutions were prepared in various concentrations by diluting a certain amount of stock solution in deionized water.

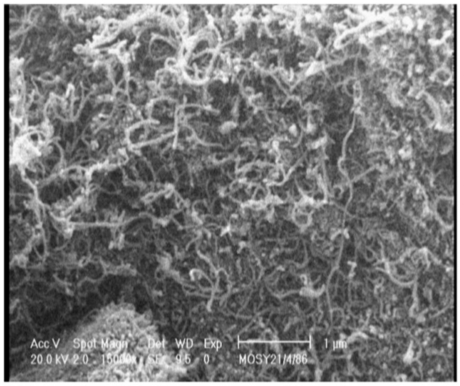
Studies of diazinon adsorption on multi-walled carbon nanotubes were conducted in a batch reactor. Adsorption experiments were carried out inside 100 mL Erlenmeyer flask containing 50 mL of different concentrations of diazinon, with a certain amount of adsorbent at different pH and contact time at room temperature (24 ± 2 °C). In this study, the effect of pH (4 and 7), contact time (1, 5, 8, 10, 12 and 15 min), initial concentration of diazinon (0.3, 0.5 and 1 mg/L) and adsorbent dose (0.1 and 0.3 g/L) on the efficiency of diazinon adsorption was investigated. The pH of the solutions was adjusted using 0.1 M chloridric acid and 0.1 M sodium hydroxide and measured using pH meter (HACH-HQ-USA).

In order to mix properly the adsorbate and adsorbent, samples were placed on the shaker (GFL3018 model) at a speed of 231 rpm for a specified time period. After a certain contact time, the samples were filtered with a filter paper and the remaining concentrations of diazinon in the solution were determined using a TLC scanner 3 apparatus.

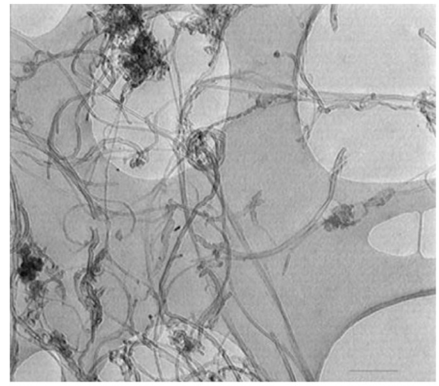
The efficiency of diazinon removal by multi-walled carbon nanotubes was calculated using Eq. (1): [24–26].

$$RE = \frac{(C_i - C_t)}{C_i} \times 100 \quad (1)$$

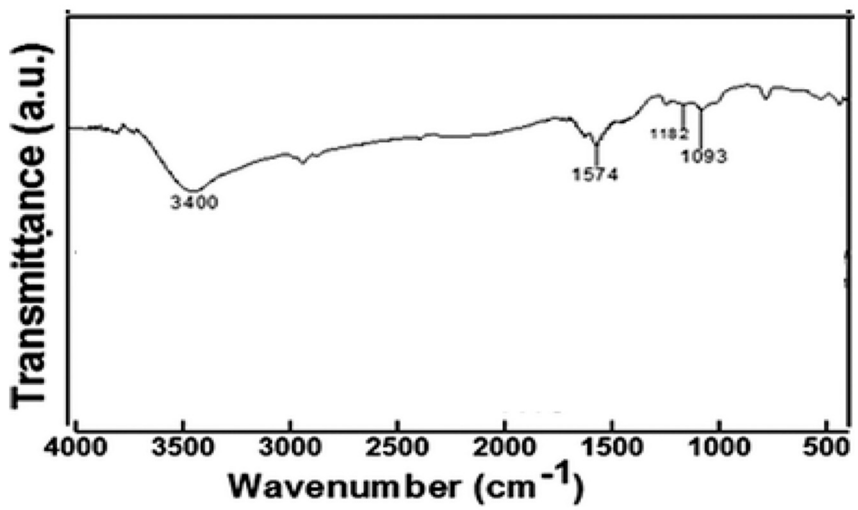
where,



(a)



(b)



(c)

Fig. 2. SEM and TEM images (a, b), FTIR (c) of CNTs.

C_i is the initial diazinon concentration, C_t the diazinon concentrations (mg/L) at time t.

3. Results and discussion

3.1. Adsorbent properties

The size, morphology and structures of multi-walled carbon nanotubes were determined by SEM and TEM and the results are shown in Fig. 2 (a–b). SEM and TEM images show porous tubular structures of multi-walled carbon nanotubes, and the existence of these porous structures on the absorbent increases the adsorption capacity for the removal of diazinon pesticide. The specific surface area (S_{BET}) and the inside diameter of the multi-walled carbon nanotubes were 370 m^2/g and 3–5 nm, respectively. The results show that multi-walled carbon nanotubes have a high surface area that helps absorb better the diazinon pesticide [11].

Infrared spectroscopy analysis (FTIR) was used to study the structure and measure the chemical species. Fig. 2c shows the analysis of the FTIR spectrum. In the multi-walled carbon nanotube spectrum, adsorption bands of 3400 cm^{-1} and 1574 cm^{-1} are related to OH– and N—H functional groups, respectively. The absorbance band observed in 1093 cm^{-1} can be correlated with C—O flexural vibrations in the structure of carboxylate and ether groups and 1182 cm^{-1} adsorption band can be related to C—N functional groups on multi- walled carbon nanotubes.

3.2. pH and contact time effects

pH is one of the factors affecting the quality of groundwater, so that in 95% of groundwater, pH is in the range of 5.5–9 and in 80% of the water, it is in the range of 6.5–8.5. Also, pH in the adsorption process plays an important role through the interaction between adsorbent and adsorbate molecules [7,11]. In this study, the effect at both pH = 4 and 7 on the efficiency of diazinon removal by multi-walled carbon nanotubes was investigated. Hydrolysis of diazinon in aqueous solutions depends on the temperature of the aqueous environments, so that the pesticide has a small thermal stability and is rapidly degraded by increasing the environmental temperature [7]. Therefore, in this study, the temperature during all adsorption processes was considered 24 ± 2 °C. Fig. 3. shows the effect of the solution pH at different contact times on the diazinon adsorption process by multi-walled carbon nanotubes. The results showed that at studied times, the efficiency of removal of diazinon at pH = 4 was greater than pH = 7. The highest efficiency of diazinon pesticide removal was observed by multi-walled nanotubes at pH = 4 (100%) and the lowest removal efficiency (1%) at pH = 7.

Increasing the efficiency of removal of diazinon in acidic pH can be due to the protonation of the hydroxyl group on the adsorbent and the

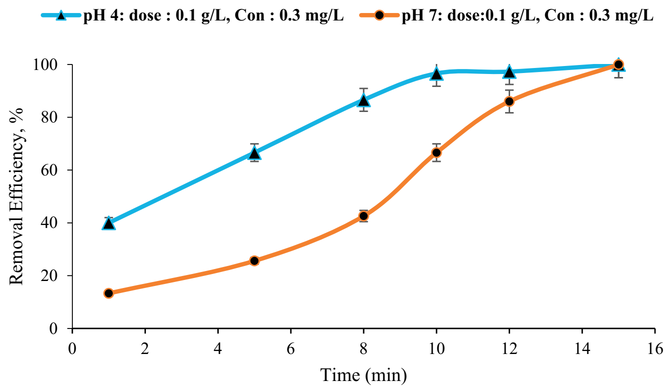


Fig. 3. Effect of pH and contact time on the efficiency of removal of diazinon by multi-walled carbon nanotubes.

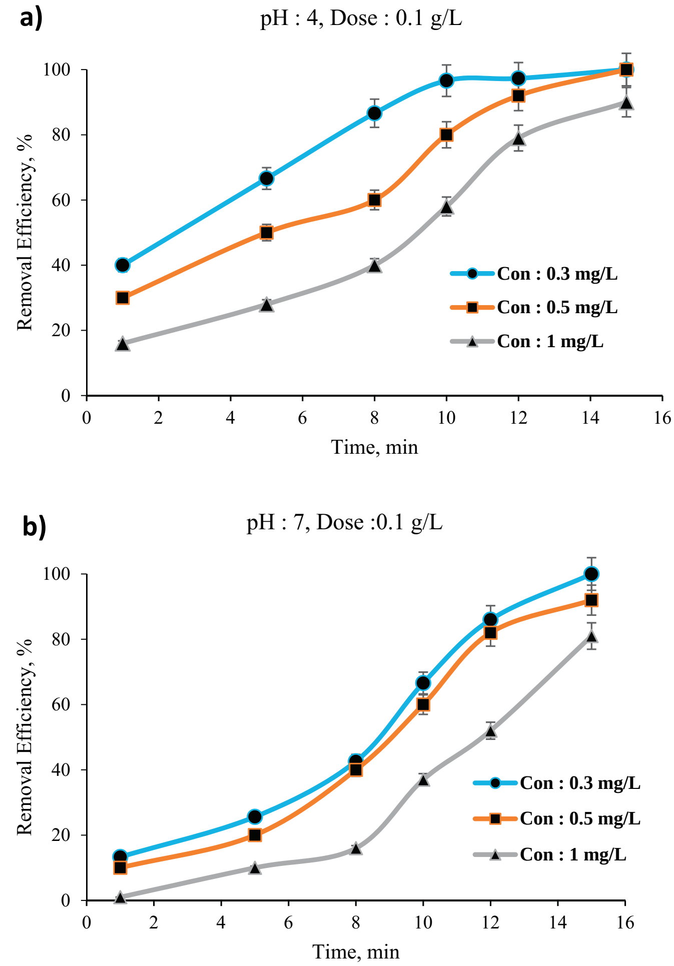


Fig. 4. Impact of contact time and initial concentration of pollutant on the efficiency of removal of diazinon by multi-walled carbon nanotubes.

protonation of nitrogen atoms on the diazinon primidine groups [6]. The results of studies of Liu et al. also showed that the highest percentage of diazinon removal was observed at pH = 4 [11] when analyzing the removal of diazinon using carbon nanotubes at pH = 2–10. In the study of Ku.et.al on diazinon pesticides with direct nano-photolysis, it was found that the removal efficiency is higher in acidic pH than alkaline and neutral at both pH [27]. Also, the results of Dehghani.et.al in analyzing the adsorption of diazinon from aqueous solutions showed that the removal of diazinon was in neutral < alkaline < acidic pHs respectively [7,28]. Another important parameter in the adsorption process is the contact time between the pollutant and absorbent molecules [29].

The results showed that in both investigated pHs, with increasing contact time, the efficiency of removal of diazinon increases, so that the highest diazinon removal efficiency at both pH = 4 and 7 at 15 min contact time was 100% and the lowest diazinon removal efficiency at 1 min contact time at both pH = 4 and 7 was 40% and 13.3%, respectively (Fig. 3). As it can be seen, at the beginning of the adsorption process, the efficiency of the removal of diazinon increases rapidly, which may be due to the presence of high and non-saturated active places at the external surface of adsorbent [1], and then, it declines gradually by increase of the tilt time. The amount of adsorption is approximately constant and has equilibrated after 15 min. A study by Liu et al. found that 96.16% of the efficiency of the removal of diazinon pesticides was achieved at 15 min contact time [11]. Therefore, in this study, the equilibrium contact time of the diazinon adsorption process is 15 min.

3.3. The effect of the initial concentration of diazinon

The effect of the initial concentration of diazinon in pH values of 4 and 7 and 1–15 contact times on the process of diazinon adsorption was investigated by multi-walled carbon nanotubes (Fig. 4). The results showed that at pH = 4 and 0.3 mg/L initial concentration of diazinon at 1 min contact time, it was 40% and in 15-min contact time, it was 100%. While at initial concentration of 0.1 mg/L at the contact time of 1 min, it was equal to 16%, and at a contact time of 15 min, it was 90%. Also, at pH 7 and 0.3 mg/L initial concentration of diazinon at 1 min contact time, it was 13.3% and at the contact time of 15 min, it was 100%. At an initial concentration of 1 mg/L at 1-min contact time, it was 1% and in a contact time of 15 min, it was 81%. Thus, by increasing the initial concentration of diazinon at pH values of 4 and 7 at studied contact times, the efficiency of the removal of diazinon by multi-walled carbon nanotubes was reduced.

Reducing the removal percentage by increasing the concentration of pollutants can be attributed to the fact that the number of active sites on the adsorbent is constant against the increase in the number of pollutant molecules. In other words, the saturation of the adsorbent surface at high concentrations of diazinon has been a major cause of this declining process. On the other hand, at high concentrations, more amounts of diazinon ions remain unabsorbed in a certain volume of solution and thus, diazinon removal efficiency in the solution decreases. In other words, in the low concentrations of diazinon ions, the ratio of the initial dissolving moles (diazinon ions) to the absorbent surface area is low. Therefore, the diazinon ions are readily absorbed by the active sites, thereby increasing the level of removal of diazinon ions [30].

3.4. The effect of adsorbent dose

The adsorbent dose is one of the key parameters in determining the quantitative removal of the pollutant selected in the adsorption process [5]. Therefore, the effect of adsorbent dose in the amounts of 0.1 and 0.3 g/L on diazinon adsorption was investigated using a solution containing 0.3 mg/L diazinon at pH values of 4 and 7. As shown in Fig. 5, with increasing adsorbent dosage from 0.1 to 0.3 mg/L, diazinon removal efficiency was increased at all contact times. Increasing the removal efficiency in this case is due to an increase in the adsorbent level and, consequently, an increasing access of adsorbate molecules to adsorption sites on multi-walled carbon nanotubes [1].

100% removal of diazinon by multi-walled carbon nanotubes was observed in a set of adsorption experiments at different concentrations, pH, and contact times. In a pH of 4, 3 samples with 0.3 mg/L initial concentration of diazinon and 1 sample with 0.5 mg/L initial concentration of diazinon as well as at pH of 7, 2 samples with initial concentration of 0.3 mg/L and 1 sample with the initial concentration

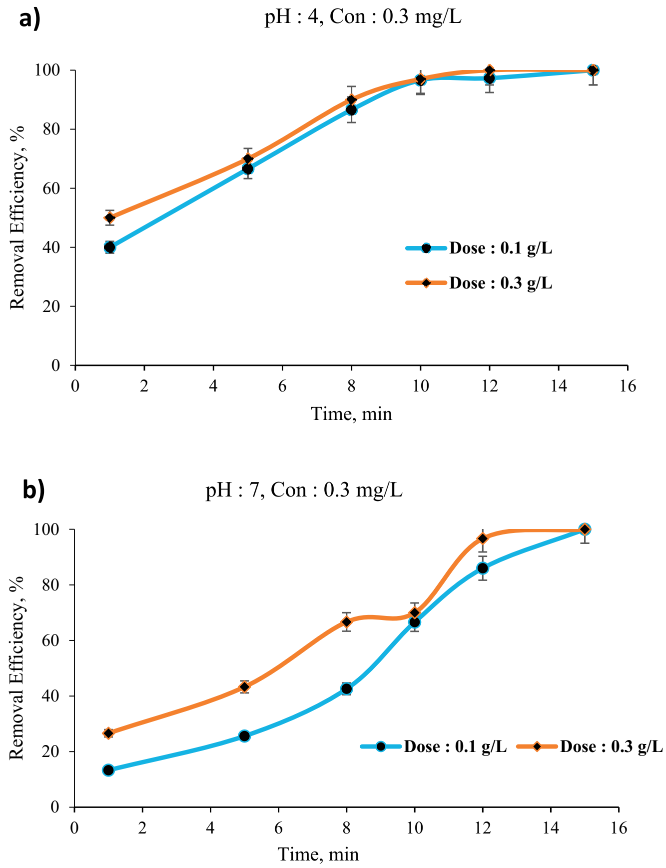


Fig. 5. The effect of contact time and adsorbent dose on the efficiency of diazinon adsorption by multi-walled carbon nanotubes.

Table 1
Comparison of different adsorbents in diazinon removal.

Adsorbent	Adsorbent dose (g/L)	Contact time (min)	pH	Efficiency of removal (%)	Reference
Fe_3O_4	0.002	1 h	7	23	[1]
MPS-MCM-41	2	1 h	6	87.2	[6]
Activated carbon	1	8 h	5.6	85	[31]
CHN-CNT	0.5	60 min	5.5	82.5	[32]
M-M-ZIF-8	0.015	15 min	4	97.8	[11]
MWCNTs	0.1	15 min	4	**100**	**This study**
			7	**100**	

of 0.5 mg/L reached the maximum removal of diazinon (100%) for different amounts of 0.1 and 0.3 g/mL of carbon nanotubes as adsorbents within 1 to 15 min.

The most important factor affecting the removal of diazinon is the ratio between the initial concentration of diazinon and the nano-adsorbent concentration. As a result, in lower concentrations of diazinon and more amount of adsorbent, the percentage of diazinon removal is higher. But what should be considered here is that increasing the amount of adsorbent is more expensive, which will not be desirable. Therefore, the researchers are trying to reduce the amount of adsorbent with the highest removal efficiency.

Therefore, maximum removal of diazinon should be considered in low concentrations of adsorbents. Generally, maximum removal of diazinon were obtained at contact time of 15 min, 0.1 g/L concentration of multi-walled carbon nanotubes and 0.3 mg/L initial concentration of diazinon.

Table 1 shows the results of the present study on the diazinon removal efficiency by multi-walled carbon nanotubes in comparison with other studies. As we can see, a wide range of different adsorbents was investigated in the removal of diazinon from aqueous solutions. The optimum pH in studies has been between 4 and 7, but most studies have considered acidic pH as optimal pH. The effect of different amounts of adsorbent in the range of 0.002 to 2 g/L has been investigated. The results also show that many of the adsorbents used in the long run had a low efficiency in removing diazinon from aqueous solutions, while the results of this study showed a higher removal of diazinon pesticide in a shorter time period by multi-walled carbon nanotubes.

It can also be noted that the different performance of various adsorbents in the removal of diazinon pesticide can be due to different characteristics such as structure, functional groups, surface area and pores of adsorbents, amounts of used adsorbents, pH, initial concentration of pesticide, operating temperature and contact time. As you can see, multi-walled carbon nanotubes are a better adsorbent in removing diazinon from aqueous solutions than other adsorbents used in other studies. Therefore, multi-walled carbon nanotubes can be used as an alternative absorbent to conventional activated carbon for the removal of environmental pollutants, including pesticides.

3.5. Diazinon adsorption from real samples

The applicability of multi-walled carbon nanotubes for the adsorption of diazinon from real water samples used in agriculture (with an electrical conductivity of 720 μs/cm, a total hardness of 250.2 mg/L $CaCO_3$ and a turbidity of 12 N.T.U) was tested at initial concentration of diazinon is 0.3 mg/L, equilibrium contact time, adsorbent dose of 0.1 g/L and pH values of 4 and 7. The rate of removal of diazinon at pH 4 and 7 was 99.1% and 98.5%, respectively.

Therefore, multi-wall carbon nanotubes could be considered as an efficient adsorbent for the removal of diazinon pesticide from real water samples. The above mentioned results also showed that removal of diazinon in synthetic samples was higher than real water samples. It seems that the presence of various impurities in real water samples probably decreased the mass transfer of diazinon molecules onto the adsorbent, so that real water matrices needs more time to achieve equilibrium conditions than those observed for deionized water [33–46].

4. Conclusion

In this study, the application of adsorption process to remove diazinon pesticide from water was investigated in batch experiments using multi-walled carbon nanotubes. The effect of parameters of pH, contact time, initial concentration of diazinon and adsorbent dose on the efficiency of diazinon removal was evaluated. The results showed that multi-walled carbon nanotubes with desirable quality, low cost of operation (in comparison with traditional methods) can remove 100% of diazinon from water sources under conditions of 15-min contact time, 0.1 g/L adsorbent dose, and 0.3 mg/L initial concentration of diazinon at ambient temperature of 24 ± 2 °C. In general, it can be concluded that multi-walled carbon nanotubes have a high potential for removal of organophosphorus pesticides, such as diazinon from water.

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Conflict of interest

The authors of this article declare that they have no conflict of interests.

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