

ORIGINAL RESEARCH PAPER

Investigation of Photo-Fenton-Like Process Efficiency in Diazinon Pesticide Removal from Aqueous Solutions

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ABSTRACT: Diazinon pesticide and its metabolites have been identified in aquatic systems worldwide. It was related to toxicity for aquatic organisms, animals, and humans. Waters contaminated with such persistent insecticides should be treated with suitable treatment processes. In this research, Diazinon removal from aqueous solutions by the photo-Fenton-like process (UV/H₂O₂/Fe⁰) was investigated. This experimental laboratory scale study was performed on synthetic wastewater in a batch system. The reaction was done in the cylindrical UV irradiation photo-reactor with 2.5 L useful volume at 25 ± 2 °C. The effects of H₂O₂, pH, ZVI, retention time and initial Diazinon concentration on removal efficiency were studied. The results showed that the optimal parameters for this process were as follows: [Fe⁰]/[H₂O₂] molar ratio of 1:1, pH 4, the reaction time of 30 min, and initial concentration of 10 mg/L. Diazinon removal and COD reduction in this condition were achieved 83.05% and 71.34%, respectively. Photo-Fenton like process can be suggested as a pretreatment step for the biological removal or post treatment of Diazinon and other pesticides with a similar structure in the aqueous environments. Also, considering the high potential of the photo-Fenton-like process in the reaction with pollutants, a modifying pilot system to ensure concurrent proper rotation speed and UV radiation seem to be necessary.

KEYWORDS: Diazinon, Photo-Fenton Like Process, AOPs, ZVI, Organophosphate Pesticide

Introduction

One of the organophosphate pesticides that is vastly used to treat a variety of insects in different regions is Diazinon [1]. Despite Diazinon has low persistence in the environment compare to the organochlorine pesticides, it is a non-specific insecticide and highly toxic to animals and humans. The main toxic action of Diazinon is inhibition of acetylcholinesterase activity [2]. Diazinon (O, O-diethyl O-[2-isopropyl-6-methylpyrimidin-4-yl] thiophosphate) is classified by the World Health Organization as “moderately hazardous” class II. It was related to toxicity for aquatic organisms in the concentration of 350 ng/L, with an LC₅₀ in killifish (48 h) of 4.4 mg/L. Fatal human doses were found to be in the range from 90 to 444 mg/kg. Diazinon is relatively water soluble (40 mg/L at 25 °C), non-polar, moderately mobile and persistent in soil. Hence, it is of concern for groundwater and surface resources derived drinking water [3–5]. Diazinon and its metabolites have been identified in aquatic systems worldwide [4]. Therefore, waters contaminated with such persistent insecticides should be treated with suitable treatment processes [6, 7].

Conventional water treatment processes have no enough efficacy for the degradation and removal of emerging water micro-pollutants including Diazinon [8]. In recent years, different methods have been developed and studied for removal of Diazinon such as biodegradation [9], sonochemical degradation [10], photodegradation [11], Fenton reagents and its derivatives [12–14], adsorption [15], electrocoagulation [4], and photocatalytic degradation [7]. A number of studies have

indicated the potential for using advanced oxidation processes (AOPs) to destroy micro-pollutants completely such as pesticides.

AOPs use combinations of oxidants, catalysts, and ultraviolet irradiation to produce hydroxyl radicals (OH[•]) in solutions and have offered interest for the degradation of non-biodegradable or hazardous organic pollutants in wastewater. The organic compounds are oxidized and mineralized by free radicals to carbon dioxide, water, and mineral salts. The Fenton reaction (Fe²⁺/H₂O₂), and Fenton-like reactions (Fe⁰/H₂O₂ or Fe³⁺/H₂O₂) have been widely applied in the treatment of biorefractory wastewater in the field of AOPs [13]. The main advantage of Fenton processes can be linked to the fact that the reaction occurs at room conditions of temperature and pressure which results a less expensive treatment. Furthermore, a short time is necessary for the reaction [16]. However, Fenton systems have two major limitations: (a) more iron sludge production and (b) slow Fe⁺³ ions reduction by H₂O₂ [13, 17]. Hence, ultraviolet (UV) irradiation is offered for dominance on the system limitations. The use of UV irradiation causes ferric iron (Fe⁺³) that had poor efficiency in Fenton process, returned to activity segment and transformed to ferrous iron ion in which enhance process efficiency. Moreover, UV irradiation affords hydroxyl radicals gain [13].

Thus, Fenton systems plus UV irradiation considering advantages such as favorite health and environmental aspects, high efficiency for various pollutants removal from water and wastewater, and other advantages have a very desirable prospect in water and wastewater industries. The presence of high concentrations of chemical, synthetic, and toxic pollutants, which have an inhibitory effect on microorganism's biological activity, have made frequent use of these systems in treating industrial wastewater. Although, great studies have been performed heretofore about AOPs application sake pesticides

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elimination from aquatic environments; anyone has been paid to evaluate and survey pesticides removal via Fenton-like processes plus UV irradiation especially using zero-valent iron (Fe^0) in Diazinon removal. Moreover, optimal values of affecting parameters in this process have been implied less. Thus, the objectives of this study were: (a) investigation of photo-Fenton-like ($\text{UV}/\text{H}_2\text{O}_2/\text{Fe}^0$) process efficiency in Diazinon removal from aqueous solutions and (b) optimization of affecting parameters on the process at different concentrations of proposed toxin.

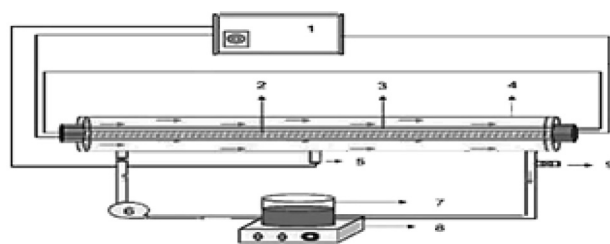
Materials and methods

Materials and equipment

In this laboratory experimental study, Diazinon primary matter for preparing different initial concentrations was applied from Merck. Ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) and zero-valent iron (ZVI) powder for activation of producing OH^\bullet ; hydrogen peroxide solution (30% w/w) as oxidant; potassium hydrogen phthalate (KHP), $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (FAS), $\text{K}_2\text{Cr}_2\text{O}_7$, HgSO_4 and AgSO_4 for COD test; H_2SO_4 and NaOH for proper pH adjustment; and sodium thiosulfate for samples maintenance were also purchased from Merck. Methanol as the mobile phase of HPLC (high-performance liquid chromatography) was obtained from Caledon Company. The Diazinon concentrations were measured using a CECIL HPLC with a UV detector (CECIL, England, CE4100 model) and column: C18 (250 mm \times 4.6 mm I.D.) and elution process were done using gradient mode. The chemical oxygen demand (COD) was determined by COD reactor HACH DRB200 and CECIL Aquarius spectrophotometer [18]. The pH meter used for pH adjustment was HACH HQ40d model.

Photo-reactor characteristic

The schematic of photoreactor is shown in Fig. 1. The cylindrical UV irradiation photo-reactor with 2.5 L useful volume was made from very slick and smooth steel for maximum rays repercussion inside of the reactor. UV rays were supplied by using low-pressure mercury vapor UV lamp about the power of 55 W (Netherland product). The lamp was put as the central inside of a transparent quartz shield with 3 cm diameter along the reactor. The photoreactor was connected to a storage vessel by 2.5 L volume for sample loading and sampling. A jar test set at 200 rpm was used for mixing. A peristaltic pump was applied for continuous flow of reactor contents. The UV lamp and photo-reactor characteristics used in this research are presented in Table 1.



1. Transformer
2. Low-pressure Hg UV lamp
3. Quartz jacket
4. Stainless steel jacket
5. Photocell
6. Pump
7. Synthetic wastewater vessel
8. Shaker
9. Sampling tube

Fig. 1. Schematic of photo-reactor

Experimental procedure

This experimental laboratory scale study was performed on synthetic wastewater in a batch system. Reaction on the sample was done in the space through UV lamp and steel cover (Fig. 1) at 25 ± 2 °C. Degradation of Diazinon during photo-Fenton-like oxidation was performed under experimental conditions including: H_2O_2 (in 0.3, 0.6, 1, and 2.5 mM), pH (the values of 4, 5.37, 7, and 10), ZVI (in 0.3, 0.6, 1, and 2.5 mM) and initial Diazinon concentration (in 10, 20, 40, and 50 mg/L). Reaction time was uncertain up to 30 minutes (1, 2, 4, 7, 15, and 30 min). Experiments were done at five discrete steps including determination of optimum values of H_2O_2 , pH, ZVI, reaction time, and effect of initial Diazinon concentration on removal efficiency. In each step, the optimum value for considered parameter was determined via poisoning all variables and changing one variable. Diazinon standard solution with a concentration of 50 mg/L was used at the first because of detecting the maximum concentration of this toxin in environmental wastewaters. Final assessment of process was based on evaluating Diazinon ultimate concentration using HPLC set. The samples were taken at selected reaction times and analyzed by HPLC. Diazinon mineralization quantity was also appointed.

Table 1. Characteristics of UV lamp and photo-reactor

UV Lamp	characteristic
Power (W)	55
Useful lifetime (h)	5000
Maximum irradiation area (nm)	253.7
Quartz shield diameter (cm)	2
Radiation intensity ($\mu\text{Ws}/\text{cm}^2$)	50000
Flow (A)	0.8
Frequency (Hz)	50 – 60
Voltage (V)	220 – 240
Photo-reactor	characteristic
Length (cm)	92
Diameter (cm)	7.6
Body substance	Rustproof steel
Influent and effluent pipes size (in)	0.75
Tolerable pressure (bar)	5
Useful volume (L)	2.5

Conclusively, the COD reduction was considered under optimal conditions (according to 5220B method)[18]. All experiments were run in duplicate. After experiments completion, results were analyzed and related diagrams were depicted using Excel software.

Results and discussion

Effect of H_2O_2

The effect of H_2O_2 dosage changes on Diazinon removal efficiency is illustrated in Fig. 2. Adequate dosages of H_2O_2 extremely affect the reaction rate. As it is obvious, Diazinon removal efficiency firstly enhanced to 76.45% with H_2O_2 increasing from 0.3 mM to 0.6 mM at a constant dose of Fe^0 . Then, it reduced with more H_2O_2 adding from 0.6 mM to 2.5 mM. The use of H_2O_2 mid iron ion results in producing a large number of OH^\bullet causes a rapid reduction of organic compounds (Eq.1) [19, 20].



Hence, removal efficiency enhanced at the first, but additional amounts of H_2O_2 reacted with produced OH^\bullet in the process (Eq. 2) and redounded upon weaker radicals output that

had lower activity rather than OH^\bullet [20]. Another reason for the decline in the efficiency of the process can be spontaneous decomposition of H_2O_2 to water and oxygen (Eq. 3). Produced O_2 trapped in the sludge matrix and led to its flotation [21]. Excess H_2O_2 in wastewater causes the COD rise [22].

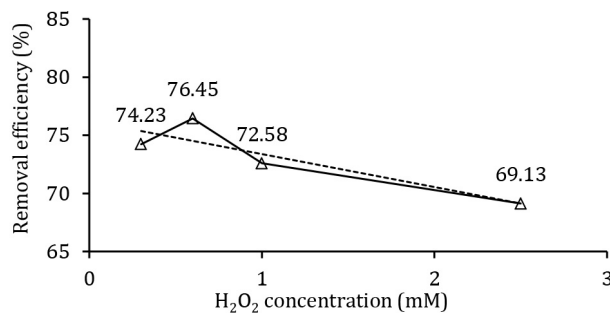


Fig. 2. Effect of different concentrations of H_2O_2 on Diazinon removal: $[\text{H}_2\text{O}_2]$ = varying, $[\text{Fe}^0]$ = 0.6 mM, pH= 5.37 (neutral), reaction time= 30 min, Diazinon: 50 mg/L. The standard deviation of all data samples was below 5.



With the above, determination of the optimum value for the maximum economy and efficiency OH^\bullet production is important. So, it seems that in this study, the maximum removal efficiency among the four studied concentrations of Diazinon in the process (0.3, 0.6, 1, and 2.5 mM) belonged to the 0.6 mM. Therefore, the optimal value was used in subsequent experiments. Murray and Parsons (2004) demonstrated that organic matter removal efficiency enhanced at a fixed concentration of Fe (II) with increasing H_2O_2 and then decreased with further increasing the H_2O_2 concentration [23]. Also, Wu et al. (2010) believe that this is due to the combination of OH^\bullet induced proliferation of H_2O_2 and addition production of OOH^\bullet which is OH^\bullet consumer [24].

Effect of pH

The effect of pH values in four ranges of acidic, alkaline, neutral and natural (values of 4, 7, 10, and 5.37) with an optimal concentration of hydrogen peroxide (0.6 mM) and certain iron concentration (0.6 mM) for Diazinon removal was examined. As shown in Fig. 3, the maximum removal efficiency equaled to 81.46% which was considered at pH= 4. Studies have shown that the best range of pH for the Fenton reaction is 3 to 4 and the process reactions are pH-dependent [25]. Researchers have also shown that pH declining to a value of 2 and lower causes decreasing the process efficiency due to $\text{OH}^{\cdot 2}$ formation and consequently Fe^{+3} and OH^\bullet production also decrease [26]. Wu et al. (2010) remarked that the removal efficiency decline at pH= 2 is related to complex species formation that have less reactivity with H_2O_2 [24]. At pH= 4, $\text{Fe}(\text{OH})^+$ ion is formed which have activity more than Fe^{+2} in the Fenton process [27]. Another reason could be that the soluble part of the iron ion and the hydroxyl radical oxidation potential are more at the acidic pH [19] and hydrogen peroxide is also decomposed easily into hydroxyl radical [27].

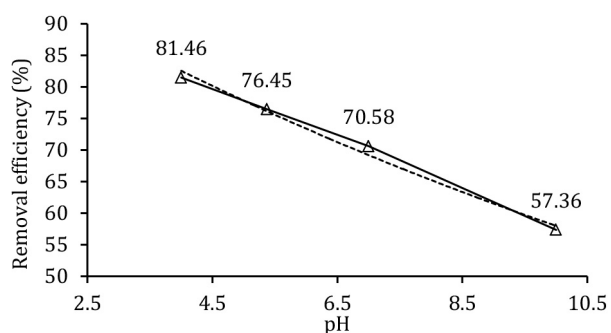


Fig. 3. Effect of pH on Diazinon removal: $[\text{H}_2\text{O}_2]$ = 0.6 mM, $[\text{Fe}^0]$ = 0.6 mM, pH= varying, reaction time= 30 min, Diazinon: 50 mg/L. The standard deviation of all data samples was below 5.

As was observed, removal efficiency decreased with increasing pH in the studied concentration of Diazinon. At pH above 4, Fe^{+3} precipitates as $\text{Fe}(\text{OH})_3$ causing decomposition of hydrogen peroxide into water and oxygen [25]. Moreover, H_2O_2 in presence of H^+ at acidic pH transforms to peroxone ion that affords H_2O_2 persistence and prohibits from H_2O_2 with iron ions reaction [28]. In this study, pH= 4 was selected as optimal pH in order to provide a favorite condition for Diazinon removal which is consistent with most studies.

Effect of ZVI

Given the dominant role of iron in the reaction with hydrogen peroxide which causes increasing production of OH^\bullet , it is very important to determine the optimum dose. Iron ions are as ferrous ion (Fe^{+2}), ferric ion (Fe^{+3}), and zero-valent iron (Fe^0) forms. ZVI powder was used in the photo-Fenton-like process. The effect of different concentrations of ZVI on the photo-Fenton-like process in Diazinon removal is specified in Fig. 4. ZVI powder addition to 0.6 mM (33.6 mg/L) increased the toxin removal efficiency to 81.46%. More amounts of ZVI decreased the process efficiency. With increasing the concentration of Fe^{+2} , more OH^\bullet produced and removal rate and efficiency of various pollutants enhanced. Other studies have also shown that excessive concentrations of iron (Fe^{+2}) have been an inhibitory effect on the production of hydroxyl radicals and reduced the rate and efficiency of the chemicals destruction [29]. The use of iron ions with hydrogen peroxide for producing large amounts of OH^\bullet and thereby increasing oxidizing potential of H_2O_2 suddenly decrease the concentration of organic compounds [21]. This causes the removal efficiency firstly enhanced with increasing concentration of the reactants. But with increasing iron concentration (higher than optimal value), the metal ion is combined with OH^\bullet and it makes the reaction environment [20].

Effect of $[\text{Fe}^0]/[\text{H}_2\text{O}_2]$ molar ratio

Perusing relationship between changes in Diazinon removal efficiency and changes in $[\text{Fe}^0]/[\text{H}_2\text{O}_2]$ molar ratio in the reaction showed that increasing the $[\text{Fe}^0]/[\text{H}_2\text{O}_2]$ molar ratio from 0.5 to 1 in the process led to increase efficiency and achieve removal efficiency of 83.46%. It is also revealed that increase in the ratio from 1 to 4.17 and higher is conducive to decline efficiency. This means that hydrogen peroxide in higher proportion than optimal value had an inhibition role for the production of hydroxyl radicals and decreased the process efficiency [21, 22].

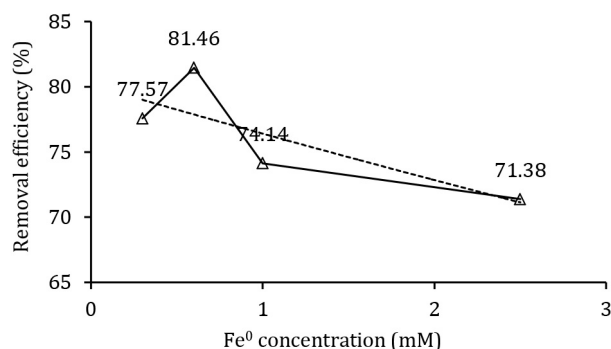


Fig. 4. Effect of different concentrations of ZVI on Diazinon removal: $[\text{H}_2\text{O}_2]=0.6$ mM, $[\text{Fe}^0]=$ varying, pH= 4, reaction time= 30 min, Diazinon: 50 mg/L. The standard deviation of all data samples was below 5.

The optimal molar ratio of $[\text{Fe}^0]/[\text{H}_2\text{O}_2]$ is important not only in terms of achieving effective degradation of Diazinon but also minimizing chemicals consumption and residuals resulting from the completion of the reaction. Hence, in order to achieve higher efficiency in Diazinon removal, according to the molar ratio of $[\text{Fe}^0]/[\text{H}_2\text{O}_2]$ in the photo-Fenton-like process was chosen 1:1.

Effect of retention time

Diazinon removal rate in various retention times (1, 2, 4, 7, 15, and 30 min) was investigated. Results demonstrated that removal efficiency enhanced with varying retention time from 0 to 30 min. Maximum removal efficiency (83.05%) was achieved at a reaction time of 30 min under optimal condition (Table 2). Also, the maximum removal in all steps was observed after 30 min that was used in the determination of optimum values. It should be noted that short treatment time may need higher chemicals consumption. Moreover, the long treatment time can enhance the reactor volume and construction costs [14]. Considering the results, 30 min was selected as appropriate reaction time in this study.

Table 2. Optimal parameters of photo-Fenton-like process for Diazinon removal

Parameter	Optimum value
H_2O_2 (mM)	0.6
pH	4
Fe^0 (mM)	0.6
$[\text{Fe}^0]/[\text{H}_2\text{O}_2]$ molar ratio	1:1
Reaction time (min)	30
Initial Diazinon concentration (mg/L)	10
Maximum COD removal (%)	71.34
Maximum Diazinon removal (%)	83.05

Effect of initial Diazinon concentration

Diazinon oxidation procedure by using a photo-Fenton-like process in different initial concentrations of the toxin (10, 20, 40, and 50 mg/L) at the optimal values of the studied variables was performed. The effect of the initial Diazinon concentration on removal efficiency is presented in Fig. 5. The maximum removal efficiency for the concentration of 10 mg/L using optimized condition was achieved 83.05%. The process efficiency for concentrations of 20, 40, and 50 mg/L were 82.33, 81.62, and 81.46 percent, respectively. Thus, the removal efficiency was declined by increasing the concentration of the toxin. Increase in concentration of the toxin caused a reduction in the reaction rate (Table 3). The reaction rate in the process for the concentration of 10 mg/L ($k=0.533$ 1/min) was more than the reaction rate of 50 mg/L ($k=0.097$

1/min). Because of increasing concentration of Diazinon, the numbers of pesticide molecules in an aqueous environment enhanced and competition between these molecules on the hydroxyl ions increased [30]. Another cause of declined detoxification efficiency in higher concentrations in photolysis processes (such as photo-Fenton-like) is that with increasing concentrations of toxin, UV photons penetration into the solution becomes less and seek to reduce the influence of the photons. Hence, the decomposition of H_2O_2 to OH^\bullet is reduced. Thus, whatever the level of toxin concentration increases, penetration of UV photons and subsequently production of hydroxyl radicals and removal efficiency will be reduced [31]. In its decomposition procedure, organic matter creates compounds which cause the breakdown of aromatic rings and effectively promote the removal efficiency by H_2O_2 over the time [30].

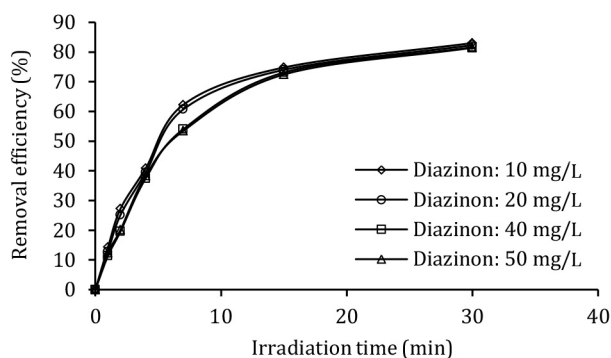


Fig. 5. Effect of different initial concentrations of Diazinon on removal efficiency: $[\text{H}_2\text{O}_2]=0.6$ mM, $[\text{Fe}^0]=0.6$, pH= 4, reaction time= 30 min. The standard deviation of all data samples was below 5.

Table 3. The reaction rate in different initial concentrations of Diazinon (t/C_i changes versus time): $[\text{H}_2\text{O}_2]=0.6$ mM, $[\text{Fe}^0]=0.6$, pH= 4, reaction time= 30 min.

Diazinon concentration (mg/L)	reaction rate constant, k (1/min)	Time (min)	Correlation coefficient (R^2)
10	0.533	30	0.9464
20	0.256	30	0.9476
40	0.123	30	0.945
50	0.097	30	0.9436

Conclusion

In this study, degradation of Diazinon by photo-Fenton-like process was investigated and optimal values of affecting parameters on the process were explained. The results showed that this process could successfully degrade Diazinon under optimal conditions (83.05% at 10 mg/L initial concentration, 30 min UV irradiation time, $[\text{Fe}^0]/[\text{H}_2\text{O}_2]$ molar ratio of 1:1, and pH₄). The rate of mineralization was surveyed and 71.34% COD reduction on optimal conditions was observed. Also, Diazinon removal in the concentration of 50 mg/L that was detected as the maximum level of the toxin in the environmental wastewaters was achieved up to 81.46%. Photo-Fenton like process ($\text{UV}/\text{H}_2\text{O}_2/\text{Fe}^0$) can be suggested as a pretreatment step for the biological removal or post treatment of Diazinon and other pesticides with a similar structure in the aqueous environments. However, due to the influence of other compounds in the wastewater of industries producing pesticides, it is recommended that a pilot-scale study on real wastewater from industries that have the problem of toxic effluents discharge into the environment be implemented. Also consider-

ing the high potential of the photo-Fenton-like process in the reaction with pollutants, modifying the pilot system to ensure concurrent proper rotation speed and UV irradiation seems to be necessary.

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