Biosci. Biotech. Res. Comm. Special Issue No 1:60-67 (2017)

BBRC
Bioscience Biotechnology Research Communications

Investigation of photocatalytic degradation of diazinon using titanium dioxide (TiO2) nanoparticles doped with iron in the presence of ultraviolet rays from the aqueous solution

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ABSTRACT

Diazinon is one of organophosphate pesticides which it is classified as a relatively dangerous substance (Class II by World Health Organization). The aim of this study was to determine the efficiency of photocatalytic degradation of diazinon using titanium dioxide (TiO2) nanoparticles doped with iron in the presence of ultraviolet light in aqueous solution. This cross-sectional study was conducted at the laboratory scale in a 2 L reactor. The nanoparticles were synthesized by sol-gel method. The degradation of diazinon was evaluated in various amounts of pH, time, nanoparticles dosage, and the concentration of diazinon. The obtained results were analyzed using Excel2007 and SPSS V.16 software and regression test. The results showed that the increasing pH, reducing the concentration of diazinon and increasing contact time can lead to increase the removal efficiency. The optimum pH was obtained to be in neutral range and at the pH=7. In addition, the optimum amount for contact time, nanoparticle dosage and diazinon concentration was found to be 60 min, 100mg/L and 50mg/L, respectively. The maximum removal efficiency was 98.58%. Photocatalytic processes have high capacity in removing of diazinon and can effectively mineralized this compound. Furthermore, the TiO2 nanoparticles are not toxic and they can be used to remove the pollutants in various industries. Thus, doped nanoparticles due to the small amount of nano-materials, low energy consumption and high efficiency can be used as a good alternative to the removal of diazinon.

KEY WORDS: DIAZINON, PHOTOCATALYTIC DEGRADATION, DOPING, TIO2

ARTICLE INFORMATION:

**Corresponding Author: hamidhekmat60@yahoo.com*
Received 27th Nov, 2016
Accepted after revision 27th March, 2017
BBRC Print ISSN: 0974-6455
Online ISSN: 2321-4007
Thomson Reuters ISI ESC and Crossref Indexed Journal
NAAS Journal Score 2017: 4.31 Cosmos IF : 4.006

Online Contents Available at: http//www.bbrc.in/

INTRODUCTION

A variety of methods such as degradation by ultrasonic waves (Mahmoodi *et al.* 2007), biodegradation (Daneshvar, 2007), optical degradation (Čolović *et al.* 2011), ozonation (Wu, 2009; Yuk Sing and Chongyu, 2007), degradation by gamma rays (Yuk Sing and Chongyu, 2007), Fenton (Wang and Lemley, 2002), treatment with UV / H2O2 (APHA and WEF, 2005) and photocatalytic degradation (Li *et al.* 2002, Kansal *et al.* 2007) have been used to remove the diazinon.

The problems of these methods are including process complexity, high cost and high consumption of chemicals. However, the nano photocatalytic method has rarely applied. In general, common physical techniques such as flocculation, aeration, adsorption on activated carbon and reverse osmosis may effectively remove the contaminants; however, these methods are not able to destroy the pollutants and contaminants from water and they only can transfer these pollutants to another phase and this is led to the formation of a secondary environmental pollution which it is casused to require retreatment and increase total cost. Photocatalyst is an advanced oxidation technology with bright future and it has been utilized in Usa, Europe and Japan in order to purify the water of pollutants (Ugurlu and Karaoglu, 2009). Advanced oxidation processes produce a strong oxidizing agent (hydroxyl radicals) that they destroy the pollutants in wastewater completely (Mesgari *et al.* 2012).

One of the chief wastewater treatment process technologies is the photocatalysts and semiconductor that have been shown to be potentially useful for the treatment of wastewater contaminants (Liu *et al.* 2005; Mekprasart, 2011). Among the various semiconductor materials (oxides, sulfides), TiO2 has gained more popularity and attention due to high Photocatalytic activity, chemical stability, resistant against optical corrosion, economic acceptability, cost-effectiveness and lack of toxicity (Zhou *et al.* 2006; Sun *et al.* 2009). Although the efficiency of TiO2 with relatively high energy band gap (3.2eV) has limited, but various methods such as increasing surface to volume ratio, connecting TiO2 to other semiconductor particles, splashing of various types of TiO2 into the zeolite pores and doping the metal and non-metal ions with TiO2 have been developed to increase the photocatalysis activity of TiO2 particles (Zhu *et al.* 2006). The conductive ion metals can lead to the formation of doped energy level between the conduction and valence bands of TiO2 which it has identified as an effective way for increasing of the Photocatalytic activity of TiO2. Moreover, the doped ions may be act as electrons or holes traps and they boost the catalytic activity of TiO2 (Liu and Chen, 2009). Previous studies have clarified that the transition metal ions, e.g Fe+3 can be utilized to increase the Photocatalytic activity (Rezaei kalanteri *et al.* 2014; Fadaei and Sadeghi, 2013).

Metal ions Fe+3 can be easily accommodated among TiO2 network due to half-filled electron configurations and with an ionic radius close to the ionic radius of Ti+4 (Sorouri Zanjani *et al.* 2009) and it is caused to increase the photocatalytic activity in the visible light region. In addition, Fe+3 ions can create a surface trap on TiO2 network for electrons and holes arising from radiation, thereby it can increase Kvantayy efficiency and photocatalytic activity by reducing the recombination of generated electrons and holes. Thus, Fe+3 ions are considered as a striking doping factor (Rezaei kalanteri *et al.* 2014, Samadi *et al.* 2010).

For many years, the mankind uses the various types of chemicals to eliminate the pets. This material has brought severe and irreparable damage to nature, environmental health, balance and stability of ecosystems and living creatures (Balschmiter *et al.* 1983). A part of pollutants such as organic material are often degraded through the biological processes but other materials such as pesticides are resistant against degradation and remain in the aquatic environment for a long time (EsmailiSari, 2001). Organophosphates, as a group of pesticides, were replaced with organochlorine a few decades ago due to their lesser resistant and stability (Girón-Pérez *et al.* 2007). These poisons are capable to create serious effect on non-target animals such as invertebrates, mammals, birds and fish due to widespread distribution in the aquatic environment (Vandergeest *et al.* 1997; Castano *et al.* 1986). The exposure of fishes with fatal doses of diazinon is caused to anemia (Anees *et al.* 1978), reduction of DNA, RNA and protein in the liver (Ansari, 1988), effect on the nervous system, the anomaly in the gills, increasing the amount of macrophages and the effect on the reproductive behavior (Dutta and Maxwell, 2003). Diazinon is partially soluble in water (40 mg/L at 25°C); non-polar and resistant against degradation in soil (APHA and WEF, 2005) which its characteristics are given in Table 1. Unlike to chlorinated pesticides, they have not accumulative nature in the body and are faster degraded in the environment (Shemer and Linden, 2006).

Diazinon is one of the Organophosphate insecticides which are classified as relatively dangerous materials (Class II by the World Health Organization). It makes toxicity for aquatic organisms at a concentration of 350 ng/l (Li *et al.* 2002), and its LC50 for fish is 4.4 ppm (Zhang and Pehkonen, 1999). More than 13 million pounds of diazinon are annually used in the United States (10). Thus, the releasing of this compound into the groundwater is one of the major concerns. The toxic effect of Diazinon, like other organic phosphorus pesti-

cides, is to stop the acetylcholinesterase (Li *et al.* 2002, Zhang and Pehkonen, 1999). It was also reported that diazinon has a negative effect on the immune system (Immunotoxic), cells (Cytotoxic) and genes (Genotoxic) (Mahmoodi *et al.* 2007).

Since the diazinon is the most widely use and dangerous pesticides for the environment, especially aquatic organisms, thus, the aim of this study was to determine the efficiency of photocatalytic degradation of diazinon using titanium dioxide nanoparticles doped with iron in the presence of ultraviolet light is the aqueous medium.

The most conducted studies in Iran have revealed that the concentration of diazinon in the water is more than standard levels. Shaeghi *et al* has found that the diazinon concentration in Gharehsou and Gorgan rivers in Golestan province was 22.4ppm and 6.74ppm, respectively which it was higher than the standard levels (Daneshvar, 2007). Khazaeii and colleagues has also observed that the concentration of diazinon in a number of water samples was higher than standard levels (Wu, 2009).

MATERIALS AND METHODS

In this study, a reactor made of Pyrex with the overall volume of 2.7 liters was used. The diameter and height of this reactor was 14 cm and 18 cm, respectively. A medium pressure Lamp UV (125 watt, length of 12 cm and a diameter of 1 cm and coating quartz with external diameter of 2.5cm, internal diameter of 2 cm and a length of 12.5 cm) has been installed in the middle of the reactor lid made of multilayer aluminum foil. There was another hole on the reactor lid which it was for sampling and it was covered during the process. The maximum wavelength emitted by the UV lamps was 247.3 nm and in UV-C range. The reactor was covered by aluminum foil to protect against the radiation. Mixing in the reactor was carried out using a magnetic stirrer and magnet. Free height of 5.5 cm inside the reactor was intended to move the magnet. Samples with different concentrations of diazinon, which was prepared by diluting the diazinon 60 percent, were entered into the reactor and the samples were taken under different conditions and at different times.

The detection of diazinon level was performed by reverse phase method of high performance liquid chromatography (HPLC). Chromatographic conditions were as following: mobile phase of methanol + water was applied at a ratio of 70:30 and C18 was used as the columns. The determination of dizazinon level was performed with a UV detector at 220 nm. Diazinon was prepared from Sigma-Aldrich CO, USA. A hanger radiometer instrument (ECL-X model) which it was to measure the intensity of light in UV-C range was utilized to determine the intensity of the lamp UV125 watt medium pressure used in reactors radiation. Intensity of lamps were measured and controlled in half the diameter of the reactor (about 7 cm) at different times.

The formula for calculating the radiation intensity:

D=L ×T T: time (s) L: radiation intensity (mw/s/cm2)

The diazinon removal percentage is calculated by following equation:

$$\text{degradation } (\%) = 100 \times \left(\frac{C_0 - C}{C_0}\right)$$

Where C0 and C are the initial and final concentration of Diazinon, respectively.

Test method and statistical analysis

Samples were taken in different states from the reactor and were centrifuged at 4000 rpm for 30 minutes and then were filtered with a 0.23µm filter to remove particles of TiO2. The DX8 and SPSS V.16 software and the ANOVA test were applied for design of experiments, drawing graphs and statistical analysis of results and LSD POST HOC was used to distinguish between different modes. Also, DX8 software was used to determine the optimal mode and model.

Nanoparticle characterization

Fig 2&3 depict the SEM images and diameter distribution of TiO2 nanoparticles before doping with Fe, respectively. The diameter measurement of nanoparticles was performed with the Measurement software and their average diameter was determined and it is approved that they are nanoparaticles. In this case, the average diameter of the nanoparticles was determined to be 42.4 nm.

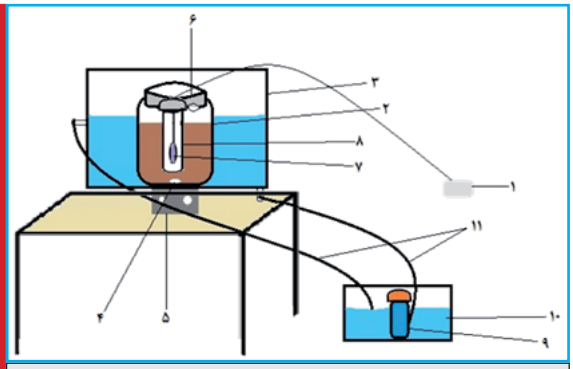

FIGURE 1. Schematic of reactor used in the process (1) Trans of lamp 150 watt, (2) glass reactor, (3) cooling water, (4) Magnet (5) magnetic stirrer (6) reactor doors, (7) UV lamp 150 watt medium pressure (8) covering quartz, (9) pump, (10) the water tank (11) pipes for water.

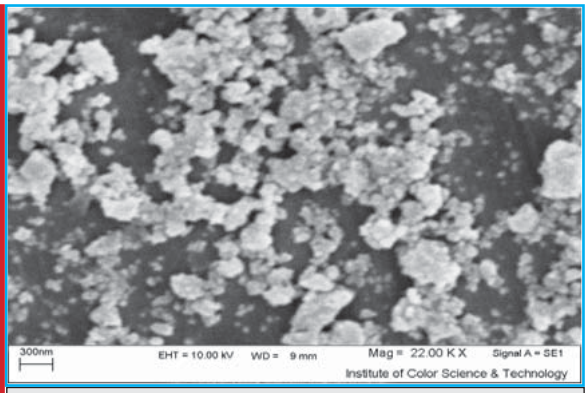

FIGURE 2. SEM images of nanoparticles

The X-ray diffraction pattern (XRD) for doped TiO2 nanoparticles with Fe is represented in Fig 6. The observed peaks (maximum peak at 250) in XRD pattern indicates that TiO2 doped with Fe has Anatase structure.

Strong peaks at 27, 36 and 55 degrees are indicative of TiO2 in the rutile phase. On the other hand strong peaks at 25 and 48 degrees is represented TiO2 is in anatase. The Fig 6 shows that highest amounts are related to anatase phase while rutile phase exists with anatase phase, heterogeneously. Titanium dioxide can be observed in 3 forms including Anatase, Rutile and Brucite which anatase and rutile have light catalytic activity. The Anatase shows more light activity than Rutile; thus, it is more applicable.

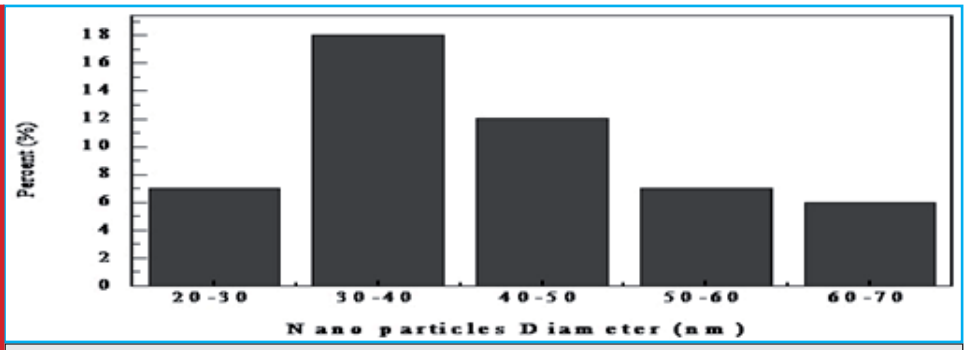

FIGURE 3. The diameter distribution of nanoparticle

In addition, Fig 4&t5 are related to the SEM images and diameter distribution of the doped TiO2 nanoparticles with Fe, respectively. The average diameter of nanoparticle was obtained to be 37.89 nm. The comparison of the Fig 2 and Fig 4 show that the doped nanoparticle structure is smaller while it was bulky before doping. Furthermore, it was clarified that the average size of doped nanoparticles (Fig5) is smaller than un-doped nanoparticles (Fig 3).

To obtain the optimum pH, the experiments were conducted in different pH (3, 5, 7, 9, 11) while other parameters including time, Diazinon concentration and nanoparticle dose were kept constant. The results are presented in fig 7. The results clarify that the maximum removal efficiency was achieved to be 97.52% at pH=7 and therefore, this pH was considered as optimum pH for next experiments. It is also observed that the removal efficiency was decreased at the pH<5 and pH>9.

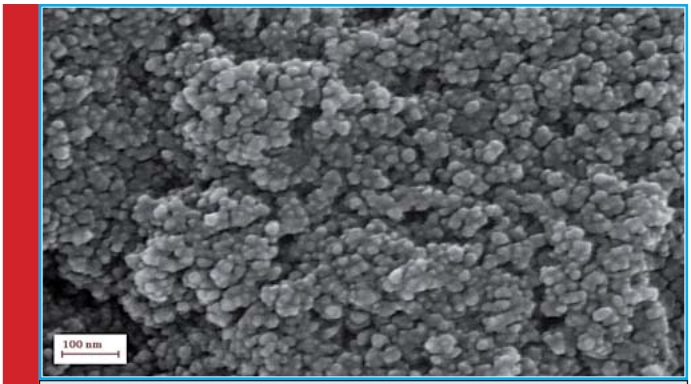

FIGURE 4. SEM images of Fe-TiO2 nanoparticles

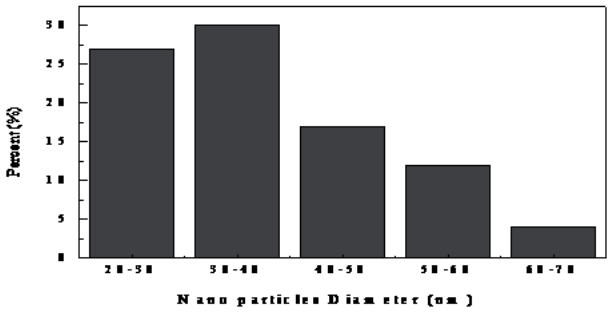

FIGURE 5. The diameter distribution of Fe-TiO2 nanoparticle

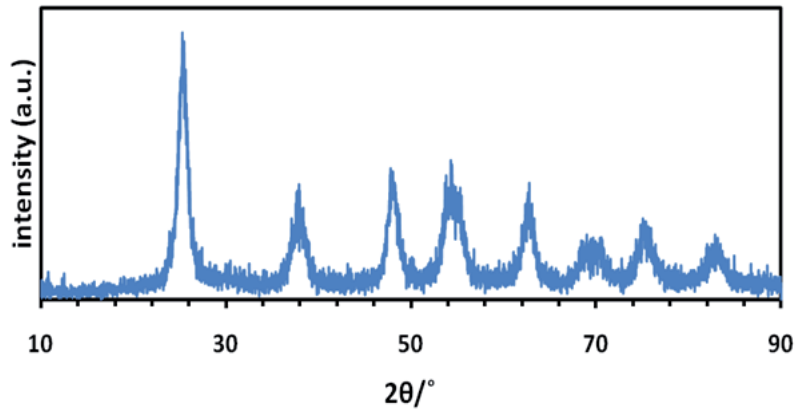

FIGURE 6. X-ray diffraction pattern for TiO2 nanoparticles doped with Fe

To determine the optimum time, the studied parameter were kept constant except the time. It was varied from 5 to 90 min. it was observed that best removal efficiencies was obtained in 60, 75 and 90 min. the removal efficiency percentage in 60, 70 and 90 min was obtained to be 98.57%, 98.06% and 98.66%, respectively. In this study, 60 min was selected as optimum time. The obtained results were presented in fig 8.

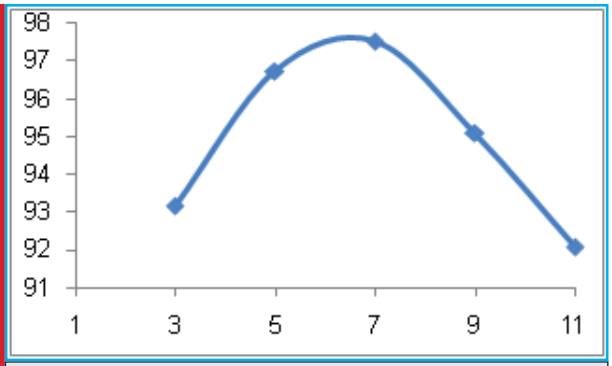

FIGURE 7. The effect of pH on diazinon removal efficiency (time= 60 min, diazinon concentration = 50 mg/L, nanoparticle dosage=100 mg/L)

The optimum diazinon concentration was determined by keeping constant of the pH, time and nanoparticle dose and the varying of the diazinon concentration in range of 1-100 mg/L. The highest removal efficiency was related to diazinon concentration of 50 mg/L (97.52%) and 25mg/L (97. 40%). It is clear that the removal efficiency for 50 mg/L is slightly more than 25mg/L; thus the 50 mg/L was accepted as optimum diazinon concentration. The results of this section are shown in Fig 9.

The optimum dose of nanoparticle was determined by varying the nanoparticle doses between 50-300 mg/L and keeping constant of other parameters. The best results were observed in the dosage of 100 mg/l (97.52%), 150mg/L (98.16%) and 200 mg/L (98.58%). Since there is no significance difference in diazinon removal efficiency in mentioned concentrations; therefore, there the nanoparticle dosage of 100 mg/L were selected as optimum dose. The results are shown in fig 10.

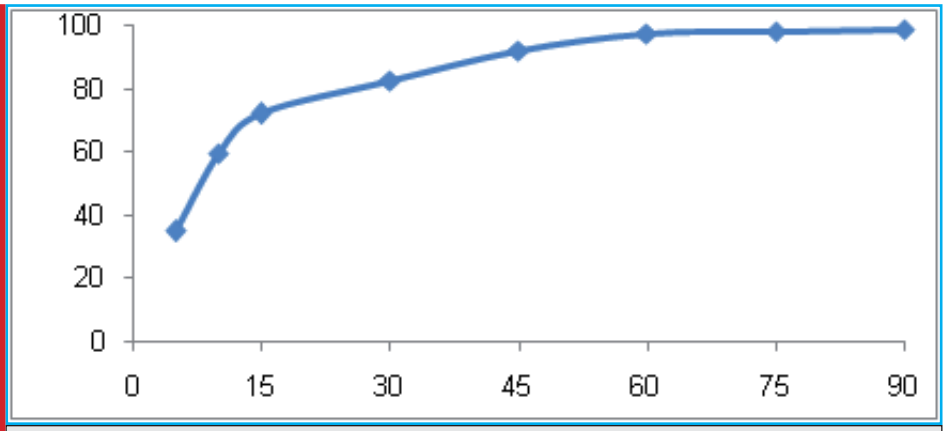

FIGURE 8. The effect of time on diazinon removal efficiency (pH=7, diazinon concentration = 50 mg/L, nanoparticle dosage=100 mg/L)

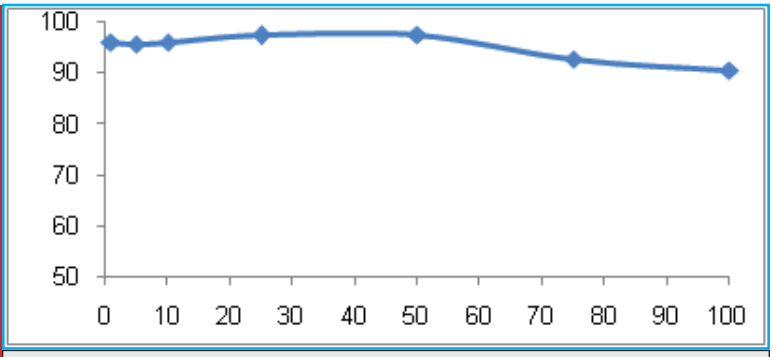

FIGURE 9. The effect of diazinon concentration on diazinon removal efficiency (time= 60, pH=7, nanoparticle dosage=100 mg/L)

DISCUSSION AND CONCLUSION

In the present study, photocatalytic decomposition of diazinon using TiO2 nanoparticles doped with iron in the presence of ultraviolet rays from the aqueous medium was studied. In addition, the effect of different parameters including pH, dose of TiO2 nanoparticles doped with iron, reaction time and concentration of diazinon was discussed.

The results indicated that the increasing of pH, decreasing of the diazinon concentration and increasing of time is resulted in higher removal efficiencies.

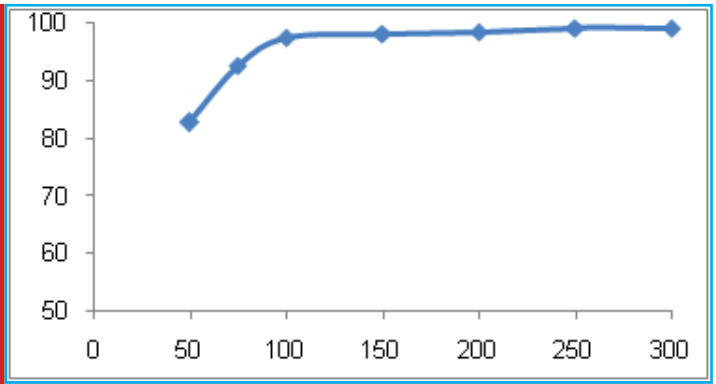

FIGURE 10. The effect of nanoparticle dosage on diazinon removal efficiency (time = 60, pH=7, diazinon concentration = 50mg/L)

Best pH for diazinon removal is obtained to be in neutral range and t pH of 7 which it is in accordance with Merabat study (2009); in his study, the photocatalytic decomposition of Indole was evaluated and the optimum ph was found to be in range of 6-7.

Daneshvar *et al* (2007) has investigated the photocatalytic decomposition of diazinon with UV-C/ZNO and the results indicated that the 80 min is required to remove 80% of diazinon; it shows that the results of present work is better than their results because the optimum time of present work is 60 min. furthermore, the results showed that the best dosage of nanoparticles to obtain the highest removal rate was 100 mg/L in addition, 50 mg/L of diazinon was selected as optimum concentration. Zhang *et al*. (2011) has neeb reported that TiO2 is effective on photocatalytic removal process that it is agreed with the results of present study. According to the results, the TiO2 dosage and contact time have positive role on diazinion removal efficiency.

Bazrafashan *et al* (2007) has found that higher electrical potential or contact time is needed to remove the higher concentration of diazinon. For any specified time, the removal efficiency has significantly increased by increasing the voltage. The highest Electrical potential (40 V) was led to fastest treatment with an over 99% of removal efficiency of diazinon after 60 minutes which it is consistent with present study. The removal efficiency was 97.52% after optimum time of 60 min.

Diazinon is one of widely used as well as most dangerous pesticides for environment and especially aquatic organisms. The photocatalytic processes have high capacity in removal and effective mineralization of diazinon. Besides, the TiO2 nanoparticles can effectively use to remove the toxic pollutants in various industries because they are not toxic; thus, the doped nanoparticle can be used as a suitable alternative to remove the diazinon due to small amounts of nano-materials, low energy consumption and high efficiency.

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