Advanced Oxidation of the Endosulfan and Profenofos in Aqueous Solution Using UV/H$_2$O$_2$ Process

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Abstract

Degradation of two pesticides, endosulfan and profenofos, was investigated in aqueous solution using a combination of ultraviolet (UV) light and hydrogen peroxide (H$_2$O$_2$). Photochemical experiments based on the L$_9$ (3^4) three-level orthogonal array of the Taguchi method with four control factors including initial pesticide concentrations (10, 15 and 20 mg/l), UV irradiation times (30, 60 and 90 min), pH (5, 6.5 and 8) and H$_2$O$_2$ (0.1, 0.01 and 0.05 M) were conducted. The endosulfan and profenofos were analyzed using gas chromatography with electron capture detector (ECD) and gas chromatography with mass spectrometry (GC-MS) respectively. Under the optimum conditions, 96.5% of the endosulfan and 98.5% of the profenofos were removed in about 90 min. The study also showed that the oxidation rate was enhanced more during the UV/H$_2$O$_2$ process in comparison to direct photolysis. The results of our study suggested that the concentration of 0.1 molar H$_2$O$_2$ and 10 ppm of pesticide in the solution at pH 8 with 90 min UV irradiation time were the optimal conditions for the photochemical degradation of two pesticides. The photochemical degradation with UV/H$_2$O$_2$ can be an efficient method to remove the endosulfan and profenofos from aqueous solution.

Keywords: endosulfan; profenofos; UV irradiation; UV/H$_2$O$_2$

1. Introduction

Pesticides are substances or mixture of substances extensively used for prevention of harmful effects caused by pests. Among the large number of different pesticides, the organophosphate and organochlorine are of particular concern (Chelme-Ayala *et al.*, 2010; Androutsopoulos *et al.*, 2013). Organochlorine pesticides (OCPs) are one of the most important persistent organic pollutants (POPs) that could be bio-accumulated in humans and animals (Firestone *et al.*, 2005; Ozmen and Mor, 2012; Androutsopoulos *et al.*, 2013; Tao *et al.*, 2013). They have low volatility (Ozmen and Mor, 2012), high lipid solubility and moderate chronic and acute toxicities (Sharma *et al.*, 2012; Wang *et al.*, 2013). More recently, endosulfan was classified as a new POPs by the Stockholm Convention Committee and added to the Stockholm Convention in May 2011 (Androutsopoulos *et al.*, 2013; Gao *et al.*, 2013; Yu *et al.*, 2013). Endosulfan (6, 7, 8, 9, 10, 10-hexachloro-1, 5, 5a, 6, 9, 9a-hexahydro-6, 9-methano-2, 4, 3-benzo-o-dioxa-thiepin-3-oxide), a widely used OCPs, has been classified as highly toxic by the majority of environmental protection agencies (Ozmen and Mor, 2012; Sharma *et. al.*,

2012). Endosulfan belongs to the cyclodiene group (C$_9$H$_6$C$_{16}$O$_3$S), and is composed of a mixture of isomers known as, α-endosulfan (64-67%) and β-endosulfan (29-32%), (Carrigerand Rand, 2008; Tao *et. al.*, 2013). It is mainly used in cotton, soya, tea and coffee farms, and also for the production of vegetables in many parts of the world including India (Sharma *et al.*, 2012; Yonli *et al.*, 2012). According to the earlier studies, endosulfan can cause neurological effects, sperm abnormalities, reduced intra-testicular spermatid counts and cell death (McDaniel and Moser, 2004; Ozmen and Mor, 2012; Sharma *et al.*, 2012). Organophosphorous pesticides (OPPs) are an important source of environmental contamination. It is believed that most of the acute neurological effects of OPPs are due to inhibition of central and/or peripheral acetylcholinesterase (AChE) (McDaniel and Moser, 2004; Pirsaheb *et al.*, 2013) resulting in synaptic accumulation of acetylcholine and excessive stimulation of cholinergic neurons (Androutsopoulos *et al.*, 2013). Profenofos (O-4-bromo-2-chlorophenyl O-ethyl S-propyl phosphorothioate) (Abass *et al.*, 2007), an organophosphorous insecticide, widely used for household (Kavitha and Venkateswara Rao, 2009) and agriculture purposes especially in

cotton, tobacco, corn, potato and beet farms and also for the production of vegetables in the India and other countries (Firestone *et al.*, 2005; Radwan *et al.*, 2005; Malghani *et al.*, 2009; Pandey *et al.*, 2011). Profenofos is extremely toxic to different organisms including mammals, insects (Gotoh *et al.*, 2001), fish, macro-invertebrates and also humans (Malghani *et al.*, 2009). It also has been classified as moderately hazardous (toxicity class II) pesticide by World Health Organization (WHO, 2006) and its acute toxic action is the inhibition of the acetylcholinesterase activity (Malghani *et al.*, 2009; Pandey *et al.*, 2011).

Considering that common treatment procedures cannot completely eliminate these compounds, other procedures like advanced oxidation processes (i.e. UV, UV/H_2O_2, UV/TiO_2, O_3/UV and O_3/H_2O_2) were considered in recent years. Among them, the UV/H_2O_2 combination is an effective method in destruction of a wide range of POPs (Alnaizy and Akgerman, 2000; Ghaly *et al.*, 2001; Wu and Linden, 2008; Abramovic *et al.*, 2010). In this process the photolysis of H_2O_2, generates oxidizing species (hydroxyl radical (•OH)), which oxidize various organic compounds quickly during the treatment process (Chen, 2009; Chelme-Ayala *et al.*, 2010). The UV/H_2O_2 process comprises direct photolysis, where the target compound is transformed through absorbing UV photons, and indirect photolysis, where the compounds react with OH radical produced via photolysis of H_2O_2 (Wu and Linden, 2008; Abramovic *et al.*, 2010). The removal of pesticides from aqueous solutions has been extensively covered in the literature. However, because the characteristics of pesticides are specific and because new pesticides are continuously being developed, research work dealing with their removal and/or degradation is still required. Degradation of endosulfan and profenofos has been reported by several authors (Cordeiro *et al.*, 2013; Ghadiri, 2001; Verma *et al.*, 2006). However, none of these papers dealt with the use of UV/H_2O_2 process for the degradation of these two pesticides.

The objective of this study was evaluation the oxidation of endosulfan and profenofos in aqueous solution during treatment with UV and UV/H_2O_2, over a various conditions of pesticides concentration, UV irradiation time, pH and H_2O_2 concentration according to the Taguchi method. Also the optimum conditions for the pesticides removal were investigated. Finally, the effect of UV/H_2O_2 and direct UV on the pesticides removal was also studied at different concentrations of the pesticides in constant conditions of other variants.

2. Materials and methods

2.1. Chemicals and solutions

Endosulfan and profenofos were purchased from Sigma Chemical Company and were used as soluble in ethanol. The chemical structures of the pesticides are presented in Fig. 1. Hydrogen peroxide (30%, w/w) was analytical reagent grade (Merck, Germany). All solutions were prepared with de-ionized water (resistivity > 18.2 MU cm). All other reagents were analytical grade and were used without further treatment.

2.2 Experimental

The photochemical degradation experiments were conducted in a batch photoreactor system. The photoreactor was a glass jar with 2 liters volume that a UV lamp with quartz sleeves (wavelength=254 nm, Arda150W.ca) were placed inside the reactor. A stirrer (Labinco-90-402) was used during the UV exposures to provide adequate mixing. The entire reactor was kept in an ice chamber where the temperature was controlled to 25±1°C. The pH was adjusted to desired values with 1 N H_2SO_4 and 1 N NaOH and measured by pH-meter (Metrohm Herisau-E520). Total organic carbon (TOC) analysis was conducted with using the SHIMADZU-TOC-VCSH analyzer (Combustion-infrared method) and in accordance with Standard Method 5310. Also chemical oxygen demand (COD) analysis was conducted

(a)   (b)
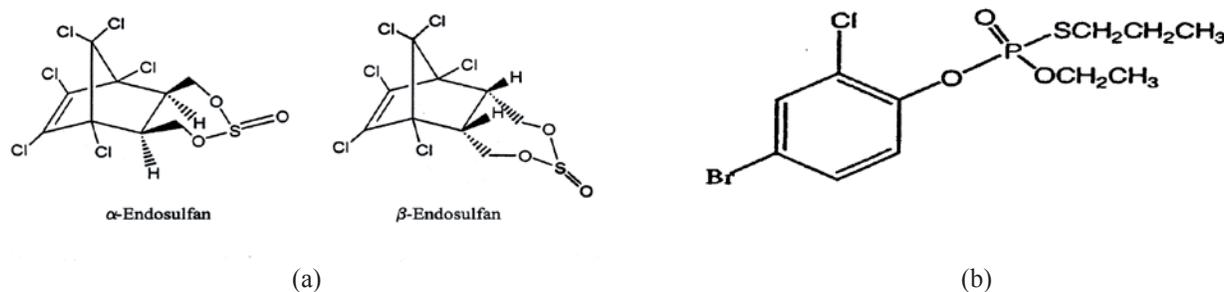
Figure 1. Chemical structures of endosulfan (a) and profenofos (b)

in accordance with Standard Method 5220. The amount of endosulfan and profenofos was measured in the aqueous solution using gas chromatography (Varian-CP 3800) with electron capture detector (ECD) and gas chromatography (Varian-Saturn 2200) with mass spectrometry (GC-MS) respectively. All experiments were carried out in triplicate at room temperature (20.0 ± 0.2 °C).

2.3. Design of experiments with Taguchi orthogonal array

In this study, the UV/H_2O_2 experiment design was based on the Taguchi method for determining the influence of control factors and to get the optimal experiment conditions for the UV/H_2O_2. Therefore, four control factors with three levels were selected based on the available literature. These factors were initial concentrations (10, 15 and 20 mg/l), UV irradiation times (30, 60 and 90 min), pH (5, 6.5 and 8) and H_2O_2 (0.1, 0.01 and 0.05 M). Nine test conditions were designed according to the $L_9 (3^4)$ orthogonal array. Under each test conditions, experiments were repeated three times. As shown in Table 1, different levels of variables were selected in such way that with very few testes could arrive to the best efficiency of removal.

3. Results

3.1. COD and TOC removal

The average of COD and TOC removal in Taguchi nine experiments was 67.8±10 and 77.6±8.4% for the endosulfan and 70.8±10.2 and 77.4±8.1% for the profenofos respectively. Also Fig. 2 shows the maximum and minimum efficiency of COD and TOC removal during the nine test conditions. The signal to noise ratio (S/N) test in the Taguchi method showed that the effect of variables in maximum efficiency removal of COD and TOC was in 10 ppm initial concentration, 90 min irradiation time, 0.1 molar H_2O_2 concentration and pH 8 for both the endosulfan and the profenofos. Therefore, the third test was the optimum conditions (Table 1).

3.2. Photodegradation of the pesticides

As shown in Fig. 3, the efficiency removal of the pesticides confirms the results from the COD and TOC testes. The average efficiency removal of the pesticides was 83.4±6.5 and 86.8±6.6% for endosulfan and profenofos, respectively. Under the optimum conditions, 96.5% of the endosulfan and 98.5% of the profenofos were removed in about 90 min. Also the final concentration of the pesticides decreased to 0.34 ppm for the endosulfan and 0.14 ppm for the profenofos from the 10 ppm initial concentration.

3.3. Effect of the pesticide concentrations

One of the important parameters could effect on degradation efficiency during the AOPs is concentration. Therefore the impact of the pesticide concentration was investigated in constant conditions of other variants such as irradiation time, H_2O_2 concentration and pH. As shown in Fig. 4, when the initial concentration increased from 10 to 30 ppm, efficiency of COD removal was decreased from 55 to 40% during UV/H_2O_2, and 42 to 21% during direct UV for endosulfan. This reduction for profenofos was from 63 to 40% for UV/H_2O_2 and 51 to 25% for direct UV. Also the Pearson correlation test showed an inverse linear relationship between the pesticides concentration

Table 1. Variables under the nine test conditions of the Taguchi $L_9 (3^4)$ orthogonal array

Test conditions	Variables				Removal efficiency (%)					
	Pesticide (ppm)	Time (min)	pH	Peroxide (molar)	Pesticide		COD		TOC	
					Endosulfan	Profenofos	Endosulfan	Profenofos	Endosulfan	Profenofos
1	10	30	5.0	0.01	74.1	79.1	55.2	63	63.6	70.7
2	10	60	6.5	0.05	88	94	72.2	79.6	81.3	82.3
3	10	90	8.0	0.10	96.5	98.5	89.1	93.3	92.3	95.4
4	15	30	6.5	0.10	81.4	81	60.8	60.7	72.1	69.6
5	15	60	8.0	0.01	84.4	88.4	66.9	68.6	75.7	75.2
6	15	90	5.0	0.05	88.1	91.1	74.5	74.2	80.7	81.5
7	20	30	8.0	0.05	75.1	78	54.7	59.2	67.8	66.7
8	20	60	5.0	0.10	81.3	87	66.2	65.3	79.4	76.5
9	20	90	6.5	0.01	82	84.2	70.6	72.9	85.9	78.4

(a)   (b)
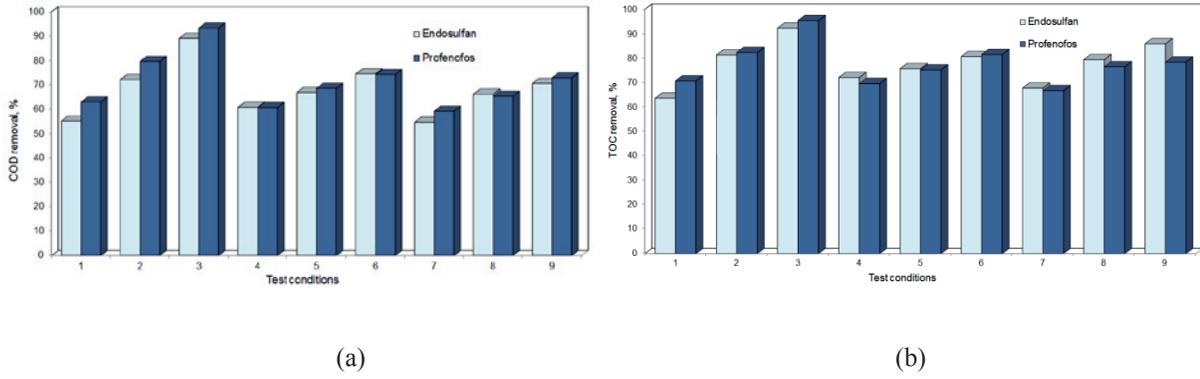
Figure 2. Removal efficiency of COD (a) and TOC (b) for endosulfan and profenofos during the UV/H_2O_2 process in nine test conditions (See Table 1. for test conditions)
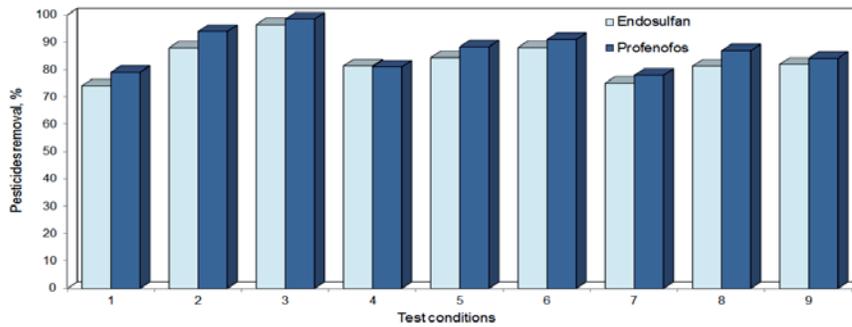
Figure 3. Endosulfan and profenofos efficiency removal during the UV/H_2O_2 process in nine test conditions (See Table 1. for test conditions)
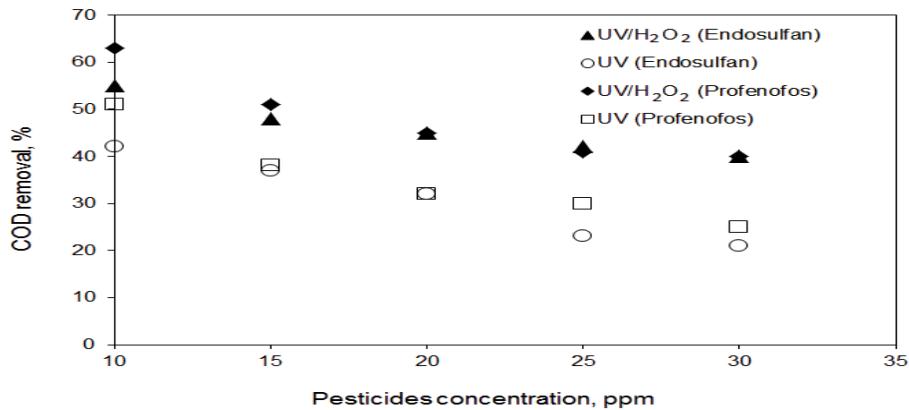
Figure 4. Relation between initial concentration and COD removal during UV and UV/H_2O_2 (30 min irradiation time, pH 7 and 0.01 M H_2O_2)
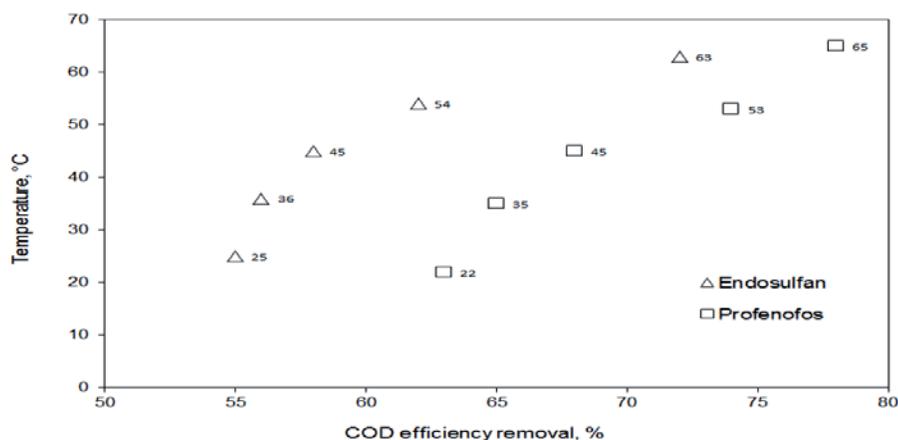
Figure 5. Relation between test temperature and COD efficiency removal during UV/H$_2$O$_2$ treatment

and efficiency of COD removal. These were (*p-value < 0.05, r= 0.985*) and (*p-value < 0.05, r= 0.996*) for endosulfan and (*p-value < 0.05, r= 0.995*) and (*p-value < 0.05, r= 0.951*) for profenofos respectively during the UV/H$_2$O$_2$ and UV.

3.4. Hydrogen peroxide, pH and irradiation time effect

The S/N test in the Taguchi method showed that the 0.1 molar of H$_2$O$_2$ was the optimum concentration in the pesticides removal. Also in comparison between UV/H$_2$O$_2$ and direct UV the mean of COD removal of the endosulfan was 46±5.3% for the UV/H$_2$O$_2$ while for direct UV was only 31±8%. These values for profenofos were 48.2±8.3 and 33.4±6% for UV/H$_2$O$_2$ and direct UV respectively that shows H$_2$O$_2$ can improve the removal efficiency. Also all of the S/N ratio curves showed that during the all test conditions, increasing the irradiation time and pH values showed the better results on the pesticides removal.

3.5. Temperature effects on photodegradation

The results in Fig. 5 showed that increasing in the sample temperature cause to raise the efficiency of COD removal of the pesticides. Pearson correlation test showed a direct linear relationship between temperature and efficiency of COD removal that was (*p-value < 0.05, r= 0.959*) for endosulfan and (*p-value < 0.05, r= 0.984*) for profenofos.

4. Discussion

This study represented the UV/H$_2$O$_2$ performance for degradation of two important pesticides, endosulfan and profenofos. Our results demonstrated that the UV/H$_2$O$_2$ process could adequately remove endosulfan and profenofos from aqueous solution. For the COD removal, profenofos showed a higher removal capacity than endosulfan during the same test conditions, as shown in Fig. 2 (a). Also the TOC and direct measurement of the pesticides confirmed that, profenofos was more degradable than endosulfan during the UV/H$_2$O$_2$ (Fig. 2(b) and Fig. 3.). As shown in Fig. 4, there is an inverse relation between COD removal and the pesticides initial concentration during both UV and UV/H$_2$O$_2$. Lapertot *et al*. (2006) reported a similar inverse relationship between concentration increase and removal efficiency during the UV/TiO$_2$ process in their study. Their study showed that the removal efficiency of the alachlor, atrazine, chlorfenvinphos and diuron was double in 10 ppm more from 30 ppm concentration (Lapertot *et al*., 2006). Photodegradation of both endosulfan and profenofos was greatly enhanced with addition of hydrogen peroxide due to the production of the strong oxidizing species, hydroxyl radical. The low direct photolysis rate was due to the lower energy of UV irradiation (Chen, 2009). With increasing in H$_2$O$_2$ concentration the removal rate has been increased (Yu *et al*., 2013). The different degradation rate between UV/H$_2$O$_2$ and direct UV photolysis was related to the hydroxyl radical concentration (Wu and Linden, 2008; Chelme-Ayala *et al*., 2010). In a study by Changlong wu

et al. (2008) that was done on removal of the parathion by UV and UV/H_2O_2, the results showed that with increase in H_2O_2 concentration from 10 to 50 mg/l, the efficiency removal was increased more than three times (Wu and Linden, 2008). Irradiation time is another critical factor to control the oxidation. Increase of the irradiation time showed a better result on the pesticides removal. This phenomenon can be related to more exposure to radiation and more opportunity of the OH radicals to practice. Therefore according to the Taguchi method the time of 90 min was selected as the optimum time in all of the test conditions. Other studies showed the similar results with our study (Gao *et al.*, 2013; Mircioiu *et al.*, 2013). Considering that the reactivity of H_2O_2 and the reaction rates of the compounds degradation depend on the solution pH (Abramovic *et al.*, 2010), three pH values (5, 6.5 and 8) were selected. The S/N curve of both pesticides indicated that the reaction rate of decay increased with increasing pH, and the pH 8 was optimum; although the pH effect was not as strong as other parameters according to the less slope curve (Figs. 6, 7 and 8). These results were similar to the Kusic *et al.* (2006) that showed with increasing the pH values from 3 to 8, the phenol removal increased from 80 to 95% during the UV/H_2O_2 and O_3/H_2O_2 (Kusic et al., 2006). Temperature was another critical factor to control the oxidation. Typically, an augment in temperature boosted the oxidation efficiencies of the COD. As shown in Fig. 5, once the temperature was increased, an improvement in the COD removal was shown. This increase has also been reported by other researchers. Castrantas *et al.* (1990) observed an increase in the efficiency degradation of phenol up to 10% with increase in temperature from 25 to 60 °C (Castrantas and Gibilisco, 1990).
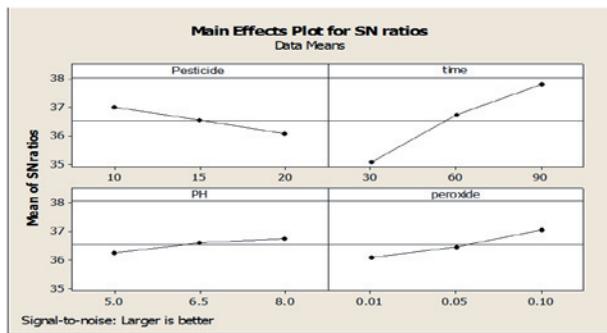
(a)
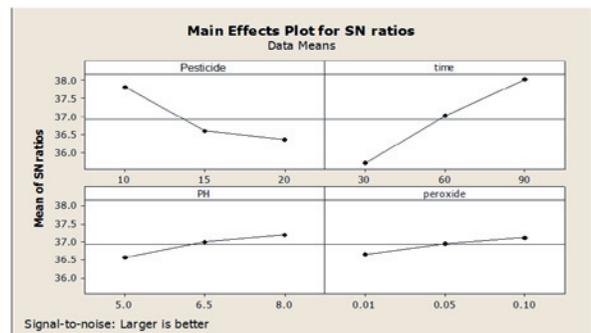
(b)

Figure 6. The Taguchi test results for the optimum conditions of COD removal. Endosulfan (a), profenofos (b)
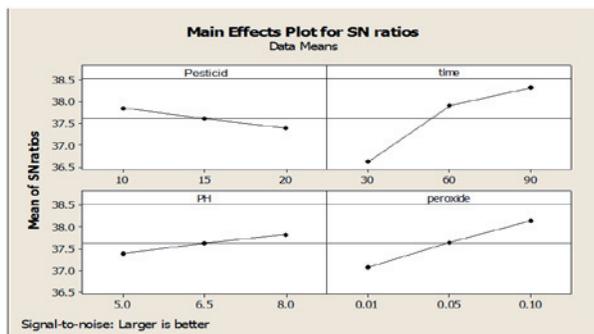
(a)
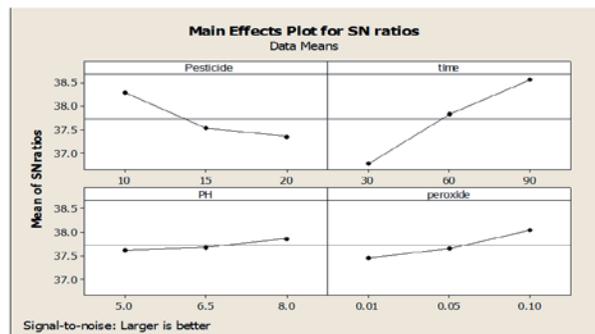
(b)

Figure 7. The Taguchi test results for the optimum conditions of TOC removal. Endosulfan (a), profenofos (b)

(a)   (b)
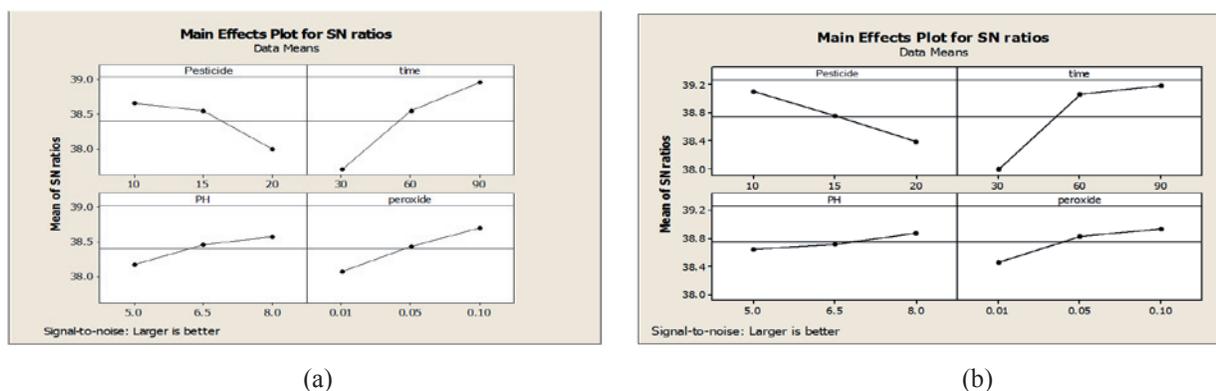
Figure 8. The Taguchi test results for the optimum conditions of pesticide removal. Endosulfan (a), profenofos (b)

5. Conclusions

The photodegradation of the endosulfan and profenofos was investigated in aqueous solution by combination of UV and hydrogen peroxide. The results showed that under optimum conditions (10 ppm initial concentration, 90 min UV irradiation time, 0.1 molar of H_2O_2 and pH 8) according to the Taguchi method, 96.5 and 98.5% degradations were observed for the endosulfan and profenofos, respectively. The impact of direct oxidation with UV and combination of UV and hydrogen peroxide on COD removal of two pesticides was also conducted. The outcomes showed an increase in COD removal of two pesticides during UV/H_2O_2 process compares to the direct UV, so that this rate was 15 and 27% more for endosulfan and profenofos respectively.

Acknowledgements

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