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Degradation of Organochlorine and Organophosphorus Pesticides by Photocatalysis: Chlorpiryfos and Endosulfan Case Study

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1. Introduction

In Colombia, the use of pesticides has increased vastly during the last two decades representing a large problem for the aquatic ecosystems and the water purification (Duarte 2009, Cáceres 2008 and and Cárdenas, 2006). Colombia possesses a large quantity of hydro resources, several of which have been transformed into reservoirs for energy generation and for use in drinking water plants. Most of the reservoirs are used for reception from agricultural regions, and therefore, the water arrives with large quantities of residual pesticides.

On the other hand, the amount of obsolete pesticides confiscated by the environmental authorities in Colombia is increasing year per year (Duarte 2009, Cáceres 2008 and ICA, 2006), this situation has produced a serious problems because there are not enough suitable sites for storage operations. Many stocks of these pesticides are being abandoned, burned, or added to water surfaces or stored in places without technical parameters for the collection of hazardous substances. For this reasons, technology is required to be able to destroy the pesticides and be sure that there are no residual organic compounds or minerals present therefore, hydro resources. One of these technologies is The AOP's (Advanced Oxidation Processes), and more specific The *Photocatalysis*, which uses ultraviolet radiation, oxygen and in some cases titanium dioxide like catalyst was selected. This presents numerous advantages in comparison with the conventional technologies: It achieves the destruction of a large number of organic pollutants and it also transforms them into innocuous products. It doesn't generate undesirable secondary compounds. It can be used for the treatment of water, air and contaminated ground; and it allows the employment of solar radiation as an energy source, adding to this process a significant environmental value.

The greatest amounts of pesticides confiscated by the environmental authorities in Colombia and the largest stocks of obsolete pesticides stored throughout the country are chlorinated organic substances, whose active ingredient is Endosulfan. (Gonzalez 2009 and

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Cárdenas 2006), for this reason this substance was selected for this study. The second substance selected was Attamix Sb because it is used massively and it is part of the phosphorous organic substances and the comparison between these two substances was interesting to test the technology.

This technology can be studied by two most common cases:

1, Homogeneous Photocatalysis: UV/H₂O₂ System: As described by Gonzalez and Braun (1996), the combination of ultraviolet radiation and hydrogen peroxide causes the breakage of the O-O bond by the action of radiation, to form two hydroxyl radicals. As Eq.(1)



Then the OH • radical attacks the hydrogen peroxide, resulting in the following sequence of reactions. Eq.(2,3,4). When organic compounds are present in the environment, the oxidation reactions start with the different radicals formed. (Fung, 1999)



The use of UV/peroxide offers great advantages: it is a very accessible, commercially available oxidant; it is thermally stable, and can be stored safely in the work place. Since it has an infinite solubility in water, there is no mass transfer problems associated with gases, as in the case of O₃. The capital investment is minimal and the operation is simple. The disadvantage is that it has low absorption at 254 nm, and high concentrations of H₂O₂ are required to maintain this process.

Heterogeneous Photocatalysis: This process is based on direct or indirect absorption of radiant energy (visible or UV) from a solid. This solid is known as photocatalyst, and usually a broadband semiconductor. In this process, the destruction or removal reactions of contaminants occur in the interfacial region between the solid and the solution, without the catalyst undergoes chemical changes.

In this research Dioxide of Titanium (TiO₂) was used as a catalyst, because, it has a high chemical stability and it works in a wide range of pH, being capable to produce electronic transitions by absorption of light in the near ultraviolet (UV-A) ((PSA-CIEMAT, 2001)

2. Materials and methods

2.1 Pesticides

2.1.1 Attamix S.B.

It has as active ingredient Chlorpyrifos, also known as Dursban 23, which belongs to the family of pesticides, insecticides and in turn to organophosphates. Chlorpyrifos is a crystal white solid with strong aroma, not very soluble in water, so it is usually mixed with oily liquids before being applied to crops or animals. In the United States, Chlorpyrifos was used for residential use until 2000 when it was restricted by the United States Environmental Protection Agency U.S. EPA. In 1999 in Colombia were sold around 2.1 million liters and 1.6 tons of Chlorpyrifos, which is the organophosphate with the biggest percentage of the total sales of this group of insecticides.

The following shows the structural form of chlorpyrifos as well as physical and chemical characteristics of the pesticide Attamix. The next figure shows the Structural form of chlorpyrifos:

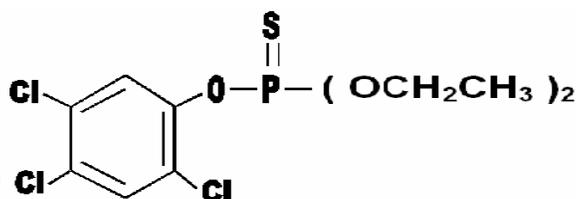


Fig. 1. Structural form of Chlorpyrifos. Organophosphorus pesticides (I): general aspects and toxicokinetics (Obiols, 2000)

Chlorpyrifos is used to control insect pests because inhibits acetylcholinesterase, is moderately toxic and its chronic exposure has been linked to neurological effects, developmental disorders, and autoimmune disorders. (Muller, 2000)

2.1.2 Endosulfan

The pesticide used in this study was obtained at ICA (Agriculture Colombian Institute) in Villavicencio City Branch, because in that place there were many containers confiscated by this institute, because they are the environmental authority in this zone of the country. The pesticide selected was Thiodan 35EC (I.A. Endosulfan). In the next pictures there are the containers used.



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Fig. 2. Thiodan 35EC used in the trials and Thiodan Confiscated by the authorities.

Endosulfan is an organochlorine compound that is used as an insecticide and acaricide. This colourless solid has emerged as a highly controversial agrochemical (ABC, 2009) due to its acute toxicity, potential for bioaccumulation, and role as an endocrine disruptor. Banned in more than 62 countries, including the European Union, several Asian and West African nations (Weekly Times, 2009) and soon in the United States, (Cone, 2010 and EPA 2010) it is still used extensively in many other countries including India, Brazil and Australia. Because of its threats to the environment, a global ban on the use and manufacture of Endosulfan is being considered under the Stockholm Convention (Earth Negotiations Bulletin, 2008).

Endosulfan is acutely neurotoxic to insects and mammals, including humans. The US EPA classifies it as Category I: "Highly Acutely Toxic" based on a LD₅₀ value of 30 mg/kg for female rats (EPA, 2002) while the World Health Organization (1984) classifies it as Class II "Moderately Hazardous" based on a rat LD₅₀ of 80 mg/kg. It is involved in the transfer of nerve impulses; symptoms of acute poisoning include hyperactivity, tremors, convulsions, lack of coordination, staggering, difficulty breathing, nausea and vomiting, diarrhea, and in

severe cases, unconsciousness (EPA, 2000), Doses as low as 35 mg/kg have been documented to cause death in humans, (International Programme on Chemical Safety, 2000) and many cases of sub-lethal poisoning have resulted in permanent brain damage (EPA, 2000). Farm workers with chronic Endosulfan exposure are at risk of rashes and skin irritation (EPA 2002). EPA's acute reference dose for dietary exposure to Endosulfan is 0.015 mg/kg for adults and 0.0015 mg/kg for children. For chronic dietary exposure, the EPA references doses are 0.006 mg/(kg.day) and 0.0006 mg/(kg.day) for adults and children, respectively (EPA, 2002).

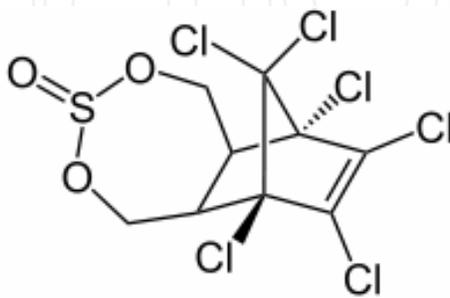


Fig. 3. Structural form of Endosulfan

2.2 Photochemical system

2.2.1 Reactor

The system used in the experiments was an annular type photochemical reactor (See Figures 4, 5 and 6). It consists of a cylindrical chamber, closed at each end, equipped with an inlet and an outlet for water, and a glass tube running through it from end to end by a central axis.

This tube protects the low pressure mercury ultraviolet lamp, connected to its corresponding electrical system.

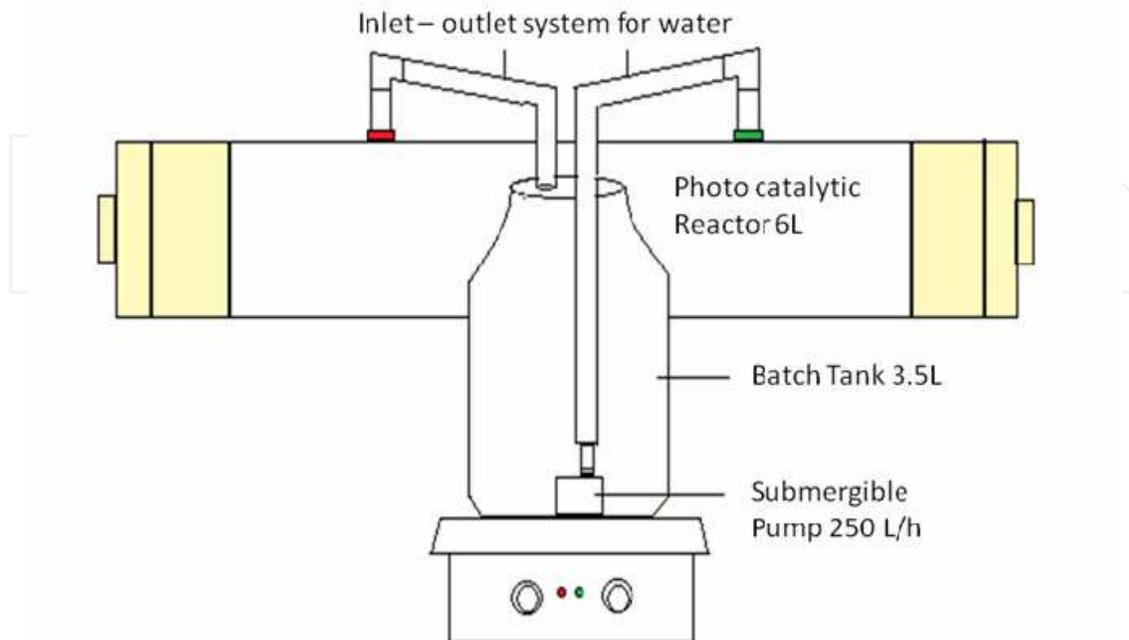


Fig. 4. Diagram of the Photocatalytic treatment units used at the laboratory

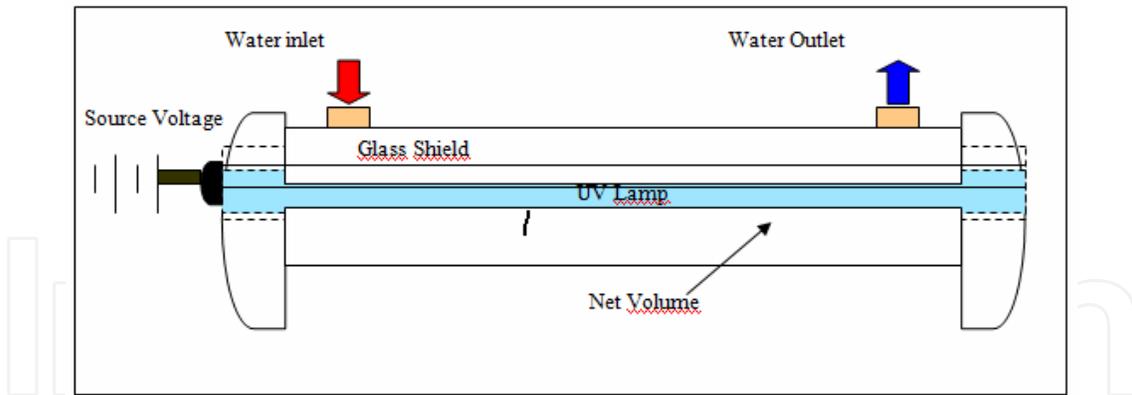


Fig. 5. Annular Photochemical Reactor



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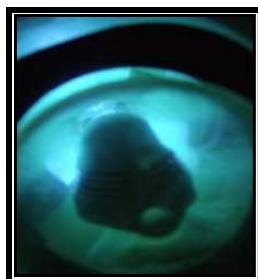
Fig. 6. Photocatalytic System



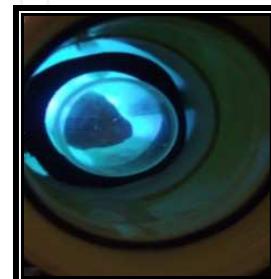
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Fig. 7. UV Lamp

2.2.2 UV Lamp

The lamp used was a germicide UV lamp of mercury at low pressure. To protect the lamp from the water was used an isolation of Duran™ glass completely close in one side, the other side had a movable piece to do the maintenance. The wavelength used was 254 nm. The figure 7 shows the lamp.

2.2.3 Batch tank

To carry out the recirculation and homogenization of the chemicals, one glass container was used, because glass material is less reactive to chemicals than other materials. It was 25.8 cm of length and 50 cm of diameter.

The next figure shows the batch tank:



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Fig. 8. Batch Tank

2.2.4 Recirculation system

The water recirculation was done by a submersible pump of 250L/h, with an input of 60Hz 3 Watts and Voltage of AC 110V -120V

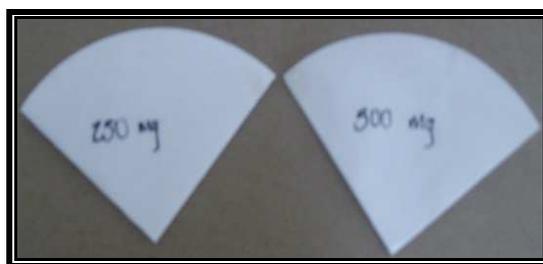
2.2.5 Homogenization system

To obtain the homogeneity of the Photocatalytic system it was used a magnetic stir.

2.3 Chemicals

2.3.1 Catalyst

It was used Titanium Oxide TiO_2 Anatase type, because it has good photo adsorption properties. The concentrations used were between 100 and 500 mg/L and how this chemical is used in a solid form it was design a package to prevent the increment in the turbidity and the decrement of efficiency of the system. This package was elaborated with filter paper with 100 mm of pore diameter and 65 g/m² of surface area.



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Fig. 9. Catalyst Package

2.3.2 Hydrogen Peroxide

In this kind of studies is common the used of Hydrogen Peroxide as strong oxidant. It was used 30% v/v and the concentrations in the system were from 1 to 10mL/L.

2.4 Other characteristics

2.4.1 Residence time

Is the time in which the pesticide will expose to radiation in its way inside the reactor, it was determined 120 min per trial because previous studies (Barrios 2005) suggested that.

2.4.2 Temperature

This process is not sensible to the temperature because the activation energy ($kT = 0.026$ eV), is very low compared with the activation energy of TiO_2 (3.2eV) and its contribution to generation $e-h^+$ is very low. For this reason heat was not added to the system. The temperature of the system was 22°C. (Herrmann 1991)

2.5 Test performed

2.5.1 Chlorpyrifos

In the next table there are the concentration of Hydrogen Peroxide and Titanium Dioxide used in the degradation of Chlorpyrifos. The trials had an irradiation time of 120 min and 22°C, the 15th trial was divided in three parts 40, 80 and 120 min to check the influence of the irradiation time in the efficiency.

Concentrations Trial	Hydrogen Peroxide (H_2O_2) (mL/L)	Titanium Dioxide (TiO_2) (mg/L)
1	2.5	250
2	1	100
3	1.5	100
4	2	100
5	2.5	200
6	1	200
7	1.5	200
8	2	200
9	2.5	200
10	1	250
11	2	250
12	3	100
13	5	100
14	7	100
15A	10	100
15B	10	100
15C	10	100

Table 1. Concentration of Hydrogen Peroxide and Titanium Dioxide used in the degradation of Chlorpyrifos

In each trial was used 20 g of Attamix and 3L of the solution was added to the batch tank. The catalyst was adhered to the reactor and the Hydrogen Peroxide volume was added

then. Was used and homogenization time of 10 min and the lamp was turned on. After 120 min an aliquot was extracted from the batch tank and analyzed by gas chromatography.

2.5.1.1 Sample Extraction

Each sample obtained from the reactor was extracted by the EPA 3510 Method using Dichloromethane (CH_2Cl_2). The solution pesticide-solvent was concentrated in Randall equipment to a 3 mL volume.



Fig. 10. Randall equipment

2.5.1.2 Gas chromatography

It was used a Thermo Finnigan model Trace GC Ultra Chromatograph, with a column RTX5 30m long and internal diameter 0.32mm and 0.5 μm of phase, detector FID and an auto injector. The makeup gas was Nitrogen at 35 mL/min and the carrier gas Hydrogen at 30 mL/min. The injection volume was 2 μL and splitless mode. The standard was a Restek 18000235 and the temperature program was:

	Flow °C/min	Temperature °C	Heat Time min
Initial		60	1
Ramp 1	7	210	10
Ramp 2	5	250	0

Table 2. Temperature Program

2.5.2 Ensosulfan

To perform the degradation of Endosulfan, the tests were different than Chlorpyrifos tests, because this compound is totally prohibited in Colombia and the authorities find this chemical in different presentations and with different level of contamination. Below there are the trials done:

Hydrogen Peroxide concentrations: 1ml/L, 2ml/L

Titanium Dioxide concentrations: 250mg/L, 500mg/L

Reaction Time: 30, 60, 120 min

Pesticide concentrations: 1000, 750, 500, 250 y 100 mg/L

The system - reactor conditions were the same as with Chlorpyrifos, but the differences are that also for Endosulfan was used Homogeneous Photocatalysis, it means without catalyst and the extraction was done using Hexane.

The same trials were done by Homogeneous to compare with heterogeneous.

3. Results

3.1 Chlorpyrifos

The results were expressed by the Elimination Percent and it is 100 minus the relationship between the difference of the initial and final concentration over the initial concentration. In the next table are the concentrations and elimination percent of each trial. How it was used 20gr of pesticide, the initial concentration was 416 mg/L of Chlorpyrifos (the active ingredient).

TiO ₂ Concentration (mg/L)	Hydrogen Peroxide (mL/L)	Trial	Pesticide Concentration (mg/L)	Elimination Percent (%)
100	1	2	0.646	84.5
100	1.5	3	0.633	84.81
100	2	4	0.612	85.31
100	2.5	5	0.569	86.35
200	1	6	0.751	81.98
200	1.5	7	0.702	83.15
200	2	8	0.644	84.55
200	2.5	9	0.638	84.69
250	1	10	0.765	81.64
250	2	11	0.731	82.46
100	3	12	0.562	86.51
100	5	13	0.553	86.73
100	7	14	0.523	87.45
100	10	15A	0.5	88
100	10	15B	0.497	88.08
100	10	15C	0.456	89.06

Table 3. Elimination percent for each trial performed

The table before shows the elimination percent increases if the volume of Hydrogen Peroxide increases. For 100 and 200 mg/L of Titanium Dioxide The figure 11 shows the elimination percent.

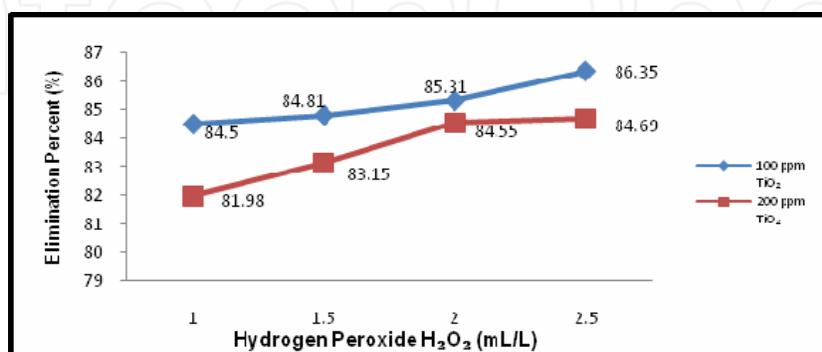


Fig. 11. Elimination Percent of Chlorpyrifos for 100 and 200 mg/L of TiO₂

The trials with 250 mg/L of Titanium Dioxide showed the lowest elimination percent, because the turbidity presented by the catalyst. The best elimination percent was 89.06% in

the 15th trial with a concentration of Titanium Dioxide of 100 mg/L using Hydrogen Peroxide of 10mL/L with 120 min of irradiation UV at 254 nm. At 120 min of reaction time all the experiments showed more than 80% of elimination percent when the pesticide Attamix is degraded with the Heterogeneous Photocatalysis. With the better trial (15) were performed two additional test at 40 and 80 min and the best elimination was at 120 min, but the difference between them are so low (88 - 89%).

This part of the investigation concluded that the pesticide (Attamix SB) can be degraded efficiently using Heterogeneous photocatalysis.

3.2 Endosulfan

The results showed that two species were presented in the pesticide α and β endosulfan.

3.2.1 Homogeneous photocatalysis

The next figure shows the trials performed and the next table presents the elimination percents obtained:

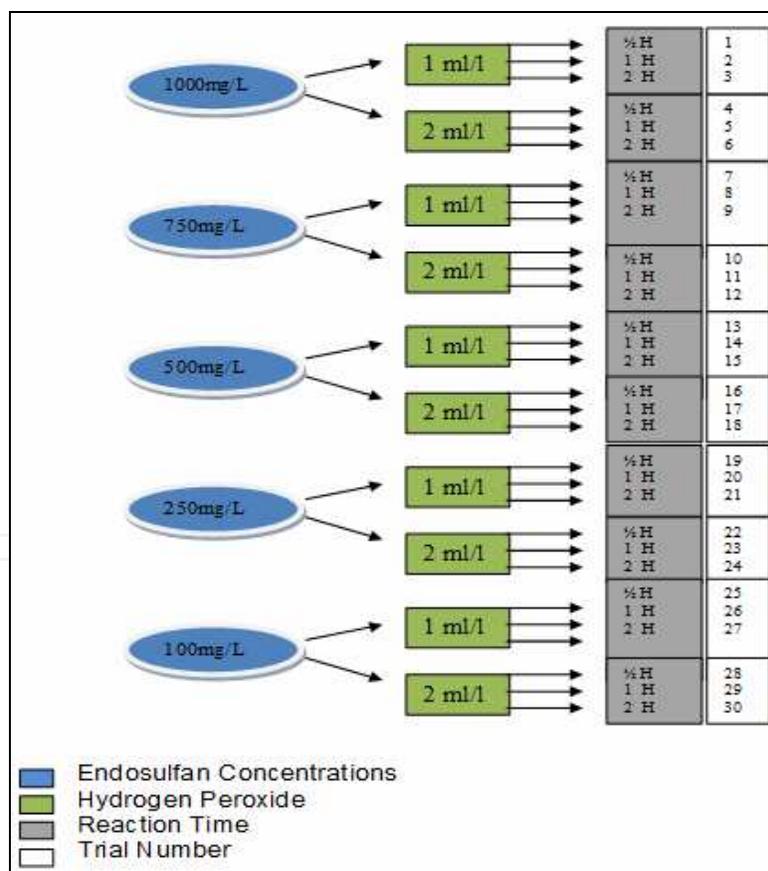


Fig. 12. Homogeneous Photocatalysis Trials

Pesticide Concentration	Trial	Elimination % α - Endosulfan	Elimination % β - Endosulfan
1000ppm	1	8,87	8,10
	2	12,76	11,48
	3	13,15	12,33
	4	8,88	8,59
	5	12,37	11,66
	6	13,58	12,50
750ppm	7	10,78	8,80
	8	11,12	10,93
	9	11,55	12,22
	10	10,90	7,75
	11	13,43	10,58
500ppm	12	13,66	13,05
	13	10,12	9,35
	14	12,32	10,87
	15	13,92	13,48
	16	10,94	11,65
	17	12,42	12,82
	18	13,72	13,36
250ppm	19	12,72	12,43
	20	16,08	14,93
	21	17,42	17,31
	22	12,80	12,66
	23	16,22	15,41
	24	17,44	17,87
100ppm	25	17,79	17,59
	26	18,37	18,65
	27	19,71	19,69
	28	18,07	17,61
	29	19,08	18,82
	30	19,80	19,70

 Table 4. Elimination Percent of α and β Endosulfan using Homogeneous Photocatalysis

For the homogeneous photocatalysis system UV/H₂O₂, it was obtained the higher elimination percent for α y β Endosulfan of 19.7% y 19.8%, with the lowest concentration of pesticide 100 mg/L, with the highest reaction time 120 min and the highest volume of Hydrogen Peroxide 2 ml/L .

3.2.2 Heterogeneous photocatalysis

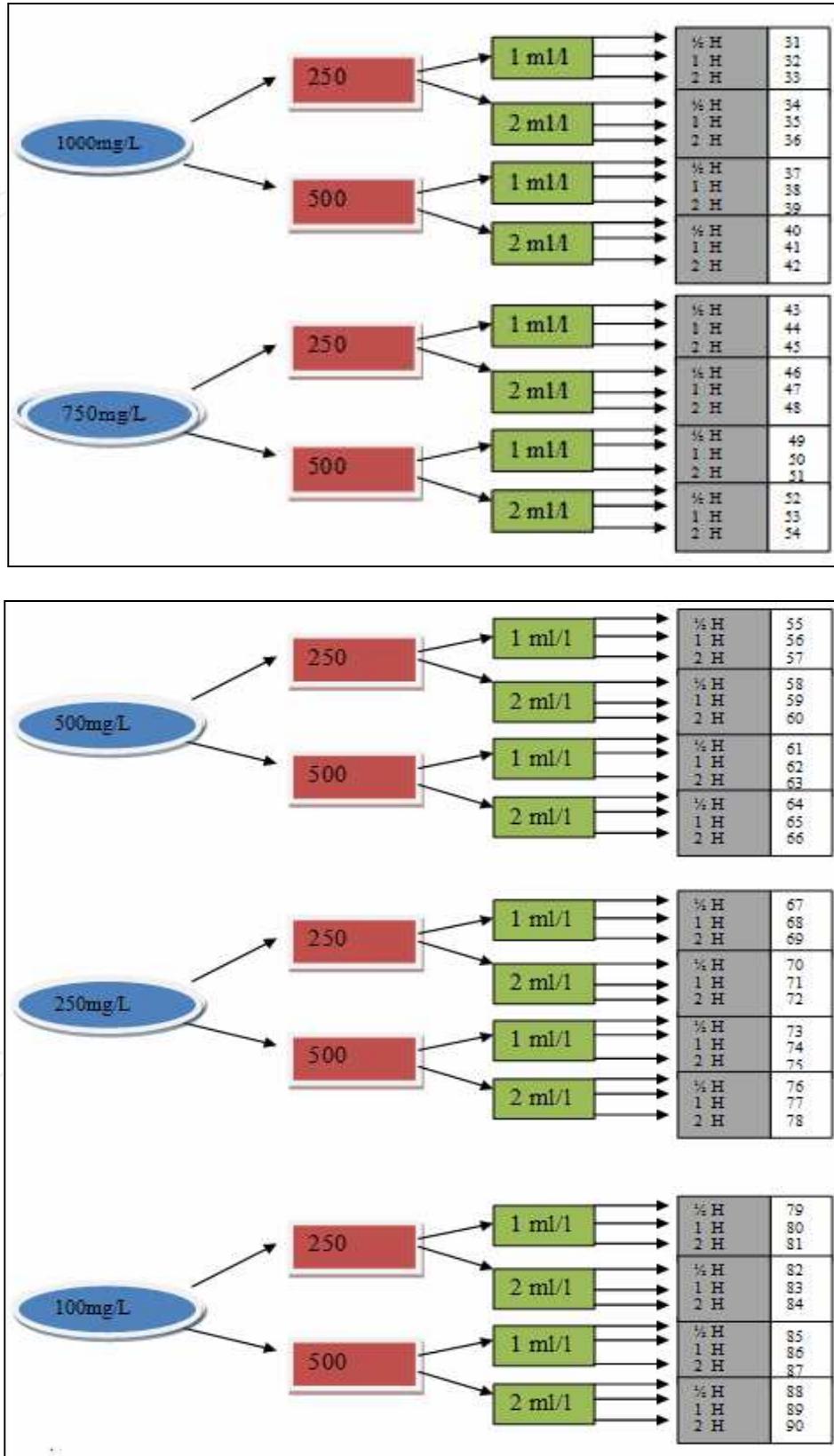


Fig. 13. Heterogeneous Photocatalysis Trials

Pesticide Concentration	Trial	Elimination % <i>a</i> - Endosulfan	Elimination % <i>β</i> - Endosulfan
1000ppm	31	62,49	64,72
	32	63,29	64,44
	33	63,28	65,28
	34	63,05	65,54
	35	63,08	66,09
	36	63,71	66,84
	37	61,72	61,00
	38	62,13	60,99
	39	61,92	61,35
	40	62,11	64,68
	41	62,32	64,89
	42	62,12	65,32
750ppm	43	62,68	67,93
	44	62,70	67,93
	45	62,81	67,98
	46	62,95	69,92
	47	63,19	69,89
	48	63,41	69,87
	49	62,14	66,22
	50	61,56	65,54
	51	62,36	64,87
	52	61,39	67,20
	53	61,15	67,58
	54	62,30	67,38
500ppm	55	72,66	66,22
	56	72,39	65,54
	57	75,31	64,87
	58	73,26	70,79
	59	73,89	70,82
	60	75,86	70,61
	61	71,13	59,22

Pesticide Concentration	Trial	Elimination % <i>α</i> - Endosulfan	Elimination % <i>β</i> - Endosulfan
	62	71,96	59,27
	63	73,30	61,32
	64	72,64	65,50
	65	73,89	66,19
	66	73,53	66,17
250ppm	67	81,54	88,45
	68	82,08	89,10
	69	82,30	88,62
	70	81,72	90,06
	71	83,17	90,63
	72	85,02	92,08
	73	81,56	87,64
	74	81,32	87,60
	75	83,02	88,48
	76	81,51	91,05
	77	83,02	91,26
	78	83,28	91,47
100ppm	79	87,26	92,11
	80	88,10	92,60
	81	89,25	93,10
	82	91,25	93,59
	83	92,36	94,09
	84	93,46	94,58
	85	85,00	90,23
	86	85,01	90,49
	87	85,00	90,29
	88	85,56	92,69
	89	85,03	93,21
	90	85,02	93,55

Table 5. Elimination Percent of α and β Endosulfan using Hetrogeneous Photocatalysis

For the heterogeneous photocatalyst system UV/TiO₂, it was obtained the higher elimination percent for α y β Endosulfan of 93.5% y 94.6% , with the lowest concentration of pesticide 100 mg/L, with the highest reaction time 120 min and the highest volume of Hydrogen Peroxide 2 ml/L and the concentration of TiO₂ of 250 mg/L.

This part of the investigation concluded that to obtain an optimum degradation for the pesticide Endosulfan is necessary the presence of a catalyst in this case of TiO₂ Anatase type.

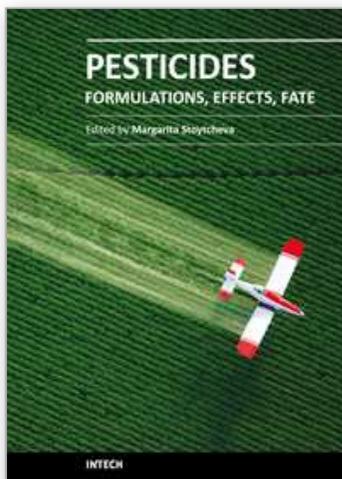
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This book provides an overview on a large variety of pesticide-related topics, organized in three sections. The first part is dedicated to the "safer" pesticides derived from natural materials, the design and the optimization of pesticides formulations, and the techniques for pesticides application. The second part is intended to demonstrate the agricultural products, environmental and biota pesticides contamination and the impacts of the pesticides presence on the ecosystems. The third part presents current investigations of the naturally occurring pesticides degradation phenomena, the environmental effects of the break down products, and different approaches to pesticides residues treatment. Written by leading experts in their respective areas, the book is highly recommended to the professionals, interested in pesticides issues.

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