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Light effects on Carbofuran and Fluometuron: Impact on natural waters. Spectrophotometric study

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Abstract

The aim of this work is to study the effect of light on Carbofuran and Fluometuron in various solvents, and to assess their ability to contaminate surface and ground waters. The results of this study show that these products are notoriously unstable under the effect of irradiation. Using water as solvent, we observed that the photodegradation rate of these products was slow, so they can lead to surface waters contamination. It was also observed that the photodegradation of Carbofuran in water was pH dependent. Indeed, Carbofuran is stable in neutral and acidic media, but undergoes photodegradation in basic medium. The kinetics of photodegradation showed that Fluometuron was first order kinetics in all solvents used, while for Carbofuran the kinetics was zero order reaction in the same solvents. Thus, at low concentrations, photodegradation of Carbofuran took place, very quickly. The measurements of the infiltration rate of these two products showed high mobility in the soil. For Carbofuran, the infiltration rate was 0.88 cm/min, and for Fluometuron it was 0.66 cm/min. As a result, these products can contaminate groundwater.

Keywords: Carbofuran, Fluometuron, photodegradation, contaminate, kinetics, mobility

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1. Introduction

Carbofuran is an insecticidal and a nematicidal product of the family of Carbamates. Its chemical formula is $C_{12}H_{15}NO_3$. It is frequently detected in surface waters (Bushway, and al., 1992; Baup, 2000; Bretaud, Saglio & J-P. Toutant, 2001). It's solubility in water is 350 mg /L at 25 °C (WHO, 2004). Fluometuron belongs to the phenyl urea herbicides family and is widely used in cotton cultivation. Its chemical formula is $C_{10}H_{11}NF_3$.

Many studies have been conducted to evaluate the degradation of pesticides and to determine their future in the environment (Trotter Kent and Wong, 1991; Kim, In Seon, et al., 2004; Otieno, Lalah, Virani, Jondiko and Schramm, 2010; Hernandez-Moreno, Perez-Lopez, Solera, Gravato and Guilhermino, 2011; H'meidy O. I. et al., 2012; Benicha, Mrabet and Azmani, 2013). These results showed that carbamates are stable in acidic or neutral media. However, some products of degradation seem more persistent than the molecule from which they originate; it is the case of Carbofuran whose duration of action in the soil can reaches 50 days (Baudet and Lejeune (2013). In the case of herbicides, the degradation may depend considerably on the type of soil, (Brown, Hayes, Tyler, and Mueller, 1994; Reddy et al., 1995; Zablotowicz, Locke and Smeda, 1998; Wilson, Yunsheng, Essington, Tyler and Selim, 1998; Matocha et al., 2006). Indeed, the studies on the effect of the tillage on Fluometuron degradation revealed that this product degrades faster in soil with conventional tillage than in soil with conservation tillage, (Locke, Zablotowicz, and Gaston Available on http://www.aq.auburn.edu/auxiliary/nsdl/scasc/.../Locke.pdf;; Bradley, York, Yelverton, Culpepper and Batts, 2001).

Also, several studies have been conducted to evaluate the toxicological effects of these two products. Those done on Carbofuran with several experimental animals showed that it is an inhibitor of acetyl cholinesterase (AChE), (National Research Council of Canada, 1979; FAO/OMS, 1981; Trotter Kent and Wong, 1991; Haubruge, Amichot, 1998; Bretaud Saglio and Toutant, 2001; EPA., 2006; Otieno, Lalah, Virani, Jondiko, and Schramm, 2010; Health Canada, 2013; Salman, 2013; Santé Canada, 2013.

On the contrary, the results of the toxicity study on Fluometuron has shown that this product is not toxic, neither by oral ingestion, with a DL50 for the rat included between 6416-8900 mg/kg, (WSSA, 1994; Halladja, 2008), nor by dermal way with a DL50 superior to 200 mg/kg for the rat and superior to 100 mg/kg for the rabbit.

It is not a cholinesterase inhibitor and is practically non-toxic to fish, (Kidd and James, 1991; WSSA, 1994; Kamrin, 1997; Sullivan, 2000; Committee on Gulf War and Health, 2003; Halladja, 2008; Dikshith, 2011).

The study on the mobility of these two products revealed that: Fluometuron is fairly mobile in soil; however the porosity of the soil and the flow rate can affect this mobility, (Insensee, Nash, and Helling, 1990; Hayes et al., 2006; Cullum, 2009). For Carbofuran, its mobility may be high or very high, dependent on the type of soil (FAO/WHO, 2005).

In Senegal, a very sunny country, the Spectrophotometric method seemed to us more appropriate to study the photodegradation of Carbofuran and Fluometuron, and to determine their rate of infiltration into the soil in order to assess the risk of surface water and groundwater contamination.

2. Experimental

2.1 Reagents

To accomplish this work, two pesticides were used: Carbofuran (99.5%) and Fluometuron (98.5%) purchased from Cluseau Info (Prolabo) and three different solvents, among which two organic ones, methanol and acetonitrile purchased from various suppliers (Aldrich, Fluka, Fisher Scientific, Prolabo, Jansen and Merck) and distilled water obtained from LPA laboratory (Department of Chemistry UCAD)

2.2 Procedure

2.2.1 Preparation of the solutions

We first prepared stock solutions of the pesticides with concentrations of 10⁻³ M in whatever the solvent used. To study the effect of the pH, stock solutions of HCI and NaOH with concentration of 1N were prepared.

Working standard solutions were prepared from the stock solutions by appropriated dilutions before use. The flasks containing the stock solutions were covered with aluminum foil to protect them against light and stored in the refrigerator. 2.2.2 Kinetics of photodegradation

The kinetics was followed by studying the variation of the absorbance A (A = Log I/Io = ϵ cl) of the peak at the wavelength of maximum absorption of the pesticide as a function of irradiation time (t). For this, we recorded the spectrum of the pesticide before and after irradiation at the same area. Thus, we noticed that the peak of the maximum absorption wavelength decreased. We also noticed that this peak did not undergone significant displacement during this period of irradiation.

2.2.3 Determination of the half-life time

Let us consider the product B that absorbs light and gives photoproducts according to the following equation:

 $B + hv \rightarrow photoproducts.$

The photodegradation kinetics was followed by studying the changes in absorbance as function of irradiation time. We supposed that the photoproducts do not absorb in the same area. The variations depending on the kinetics of photodegradation:

- For zero order kinetics: $t_{1/2} = [B]_0/2K$; K is the rate constant, $t_{1/2}$ is the half life time of the reaction, it is proportional to the initial concentration $[B]_0$;
- For first order kinetics: $t_{1/2} = 0.693/K$, it is independent of the original concentration;

- For second order kinetics: $t_{1/2} = 1/[B]_0K$, it is inversely proportional to the initial concentration.

2.2.4 Determination of the Infiltration Rate

Sand dune of Keur Ndiaye LÔ in the Niayes (Senegal's major vegetable producing areas) was used for the determination of the infiltration rate.

The sand was washed 15 times per day with tap water during three days and three times the fourth day with distilled water. Then it was introduced into a PVC tube of 12 cm of diameter and 50 cm of height.

And by using a dropping funnel filled with water and equipped with a tap, we adjusted the water flow so that the time interval between two drops was 12 s.

To measure the infiltration rate, we poured the pesticide solution on the sand surface. Then we poured the water from the dropping funnel through the tube, which was collected every 15 min in several beakers which were numbered: 1, 2, 3, 4, etc.. ...

The analysis of the samples collected from each beaker, allowed us to determine the presence of the pesticides. By knowing the time of appearance of each fraction of pesticide and the distance run, the infiltration rate in this soil type was deduced by the relation V = d/t.

2.3 Apparatus

To realize the photodegradation of our products we used the following materials:

- Thermo Spectronic Helios Gamma UV-Vis Spectrophotometer connected to a computer;
- Photochemical reactor comprising an irradiation source (Oriel model 6137) equipped with a mercury lamp of 200 watt power, supply box (Oriel model 8500), magnetic stirrer, quartz cell (path Optical 1 cm) placed 45 cm from the lamp and protective cage;
- PVC tube of 12 cm in diameter and 50 cm in height, filled with sand for the determination of infiltration rates;
- Dropping funnel filled with water used as eluent;
- Precision balance (precision 10⁻⁴ g, Sartorius model);
- Vials, bottles, beakers, pipettes, micropipettes of volume 2μ l, 200 μ .L, from Pipetman brand and a micropipette of volume 20μ L, from Gilson brand).

3. Results and Discussion

3.1 Electronic absorption spectra of Fluometuron in various solvents

The spectra of unirradiated Fluometuron were followed in methanol (Fig. 1) and in acetonitrile (Fig. 2) used as solvent. They showed two $\pi \rightarrow \pi^*$ transition bands located respectively between 200 and 220 nm, and between 220 and 260 nm. In water the spectrum revealed a single band situated between 220 and 260 nm.



Fig.1: Absorption Spectra of Fluometuron in Methanol; C = 10^{-4} M; $\lambda = 243$ (nm)



Fig.2: Absorption Spectra of Fluometuron in Acetonitrile; C = 10⁻⁵ M

The peak at 243 nm was followed in both organic solvents. It was noticed that the intensity of this peak decreased with the irradiation time, indicating that this product is light sensitive. We also noticed in these solvents the appearance of a third band whose intensity increased with the irradiation time. In water, the absorption spectrum of unirradiated Fluometuron showed a single π - π * transition band located between 220 and 260 nm (Fig.3). It was observed a small decrease of the peak intensity after intensive irradiation, showing that the degradation of this product in water under light effect is low.



Fig.3: Absorption Spectra of Fluometuron in Water; $C = 10^{-4} M$

3.2 Electronic Absorption Spectra of Carbofuran in Various Solvents

The electronic absorption spectrum of unirradiated Carbofuran in methanol and in acetonitrile showed a single $\pi \rightarrow \pi^*$ transition band located between 200 and 240 nm (Fig. 4).





Under the effect of irradiation, we observed a decrease of the peak intensity at 206.5 nm, indicating that this pesticide degrades under the effect of light in these solvents.

In water, the photodegradation of Carbofuran considerably depends on the pH. The electronic spectra of the unirradiated product respectively at pH 2, 4 and 7 showed a $\pi \rightarrow \pi^*$ transition band, located between 190 and 210 nm. After irradiation at the same values of pH, a quasi-stability of the peak was observed, indicating that Carbofuran is not very light sensitive at acidic and neutral pH. On the other hand at the values of pH 10.5 and 12.2 (Fig. 5), the electronic spectrum of unirradiated Carbofuran showed two $\pi \rightarrow \pi^*$ transition bands, situated respectively between 190 nm and 225 nm and between 225 nm and 250 nm. Gradually, while the irradiation took place, it was observed a decrease of the peak intensity at 200 nm for pH = 10.5 and at 241.5 nm for pH = 12.2. These results indicate that Carbofuran undergoes degradation under alkaline conditions.



Fig.5a: Absorption Spectra of Carbofuran in Water at pH = 10.5; C = 4.10⁻⁵ M



Fig.5b: Absorption Spectra of Carbofuran in Water at pH = 12.2; C = 4.10^{-5} M

3.3 Photodegradation Kinetics of Fluometuron and Carbofuran

In order to determine the half-life time of the two pesticides and the constant rate of their photodegradation, the relation Ln ($A_0/A = f$ (t) was plotted for Fluometuron in different solvents used while for Carbofuran in the same solvents, the following relation A = f (t) was plotted; where A_0 and A are the absorbance at t = 0 and t $\neq 0$ respectively.

3.3.1 Photodegradation Kinetics of Fluometuron in the Used Solvents

In the figure 6 is represented the kinetics of photodegradation of Fluometuron in the various solvents used. It is observed that the photodegradation of this product obeys first order kinetics. The plotting of the equation Ln ($A_0/A = f(t)$) is linear with a correlation coefficient close to the unity.



Fig.6: Photodegradation Kinetics of Fluometuron in the Used Solvents: a) Water; $C = 10^{-4} M$

 λ = 241 nm; b) methanol; C = 10^{-4} M, λ = 243 nm; c) acetonitrile; C = 10^{-5} M, λ = 243.5 nm.

The compiled results are displayed in the table I.

Solvents	С		ε(mol ⁻¹ .	K (min ⁻	t _{1/2}	order	R	(R^2)
	(mol/L)	λ(nm)	L.cm ⁻¹)	¹)	(min)			
Water	10 ⁻⁴	241	13200	4.2x10 ⁻⁴	1675	1	0.993	0.9860
Methanol	10-4	243	15900	2.7x10 ⁻³	259	1	0.99	0.9801
Acetonitrile	10 ⁻⁵	243.5	48600	1.1x10 ⁻²	69	1	0.99	0.9801

Table I: Kinetics Parameters of Fluometuron Photodegradation

 $\boldsymbol{\epsilon}$: molar extinction coefficient ; R : correlation coefficient

The analysis of this table shows that: For all solvents used, this pesticide absorbs in the same area. The constant rate of photodegradation is higher in acetonitrile and lower in water; this means that, the half-life time is lower in acetonitrile and higher in water.

So, photodegradation of Fluometuron will therefore be slower in water and this can lead to surface waters contamination.

3.3.2 Photodegradation Kinetics of the Carbofuran in the Used Solvents

Carbofuran photodegradation was zero order kinetics in all the solvents used, and by plotting the relation A = f(t) we obtained in all cases a linear curve with coefficient of correlation close to the unity (Fig. 7 and Fig.7b).

Fig.7: Photodegradation Kinetics of Carbofuran in Various Solvents: a) Methanol; b) Acetonitrile



 $C = 10^{-4}$ M; $\lambda_{ex} = 206.5$ nm All other kinetics parameters are compiled in Tables II and III.

Solvents	C(mol/L)	λ (nm)	ε(mol 1.L.cm-1)	K (mol.L- 1.min-1)	t1/2 (min)	order	R	(R ²)
Methanol	10-4	206.5	19490	3.2.10-7	156	0	0.990	0.9801
Acetonitrile	10-4	206	18340	1.6.10-6	32	0	0.988	0.9761

Table II: Parameters of Carbofuran Photodegradation

 ϵ : molar extinction coefficient ; R^2 : correlation coefficient

Table III: Characteristics of Carbofuran Photodegradation Kinetics in Water

pН	λ (nm)	K (mol.L-1.min-1)	t1/2(min)	order	R	(R^2)
2.4	275.5	stable	-	-	-	-
3.7	275	stable	-	-	-	-
7	274.5	stable	-	-	-	-
10.5	200	1.968.10 ⁻⁸	1016.26	0	0.986	0.9722
11.9	242	4.10 ⁻⁸	500	0	0.996	0.9880
12.2	241.5	5.2.10 ⁻⁸	384.6	0	0.988	0.9761

R² : correlation coefficient

As for Fluometuron, we noticed that the half-life time of Carbofuran was higher in water, as consequences it undergoes a slower rate of photodegradation. Thus, in the case of very slow microbial decomposition, both pesticides can contaminate surface waters.

3.4 Measurements of the Infiltration rates of Fluometuron and Carbofuran

3.4.1 Permeability of the Soil

The study of the permeability of the sand used was done by passing a constant flow of water through the PVC tube filled with sand and by collecting the filtrated fractions of water every 30 minutes in beakers over a period of 6 hours.

By plotting the relation Vol. $_{water} = f(t)$, the curve obtained is displayed in the figure 8 below.

The good linearity of the curve showed that the infiltration of water into the sand was uniform and indicates that this sand has a well-balanced texture. So our products had a constant infiltration rate into the sand.



Fig.8: Sand of Keur Ndiaye LÔ; Test of Permeability with Distilled Water

3.4.2 Fluorimetric Characteristics of the Unidentified Organic Compound Present in the sand when Excited at 274 nm

The analytical study of the samples of solution collected after infiltration through the sand column showed that it contained a fluorescent product. By exciting the samples with the same wavelength as Carbofuran (at 274 nm), we noticed an emission peak at 430 nm (Fig. 9), different from those of Carbofuran (which was at 307 nm). By washing the sand several times per day, after one week it is noticed, the disappearance of this peak located at 430 nm, indicating that this unidentified organic compound was soluble in water (Fig. 9)



Fig.9: Fluorescence Emission Spectra of the Unidentified Organic Compound Present in the sand: a) before Washing; b) After Intensive Washing

3.4.3 Measurement of Infiltration Rate of Carbofuran

The solution samples were collected after infiltration through the column of PVC during the first 30 minutes, and then every 15 minutes. By exciting the samples at 274 nm, it was noticed the formation of an emission band situated at 307 nm corresponding to that of Carbofuran. When we plotted the relationship between If and f (t) (where If represents the fluorescence intensity of the Carbofuran and t the time of infiltration), the time-intensity curve obtained was higher than 45 min. This indicated that the infiltration time of Carbofuran in this kind of sand was 45 min. When the sand height was 40 cm, the infiltration rate of Carbofuran was 0.88 cm /min. (This experiment was repeated three times).

Fig.10: Carbofuran Intensity of Fluorescence Versus Time of Infiltration

3.4.4 Measurement of the Infiltration rate of Fluometuron

At first we determined the fluorimetric characteristics of the unidentified organic compound present in the sand when excited at the same wavelength as those used to excite Fluometuron. It was obtained before and after 15 min of irradiation (optimal irradiation time of Fluometuron), the formation of an emission peak of low intensity at 410 nm, different from the emission wavelength of the Fluometuron (which was at 435 nm). The small decrease in the signal intensity of this sand product after 15 min of irradiation showed that it was stable under light conditions. By comparing on one hand the emission wavelength of the unidentified organic compound of the sand (410 nm) with those of the Fluometuron (435 nm) and on the other hand the intensity of their fluorescence signal [12.57 for the unidentified organic compound of the sand and 153.7 for the solution of Fluometuron (10⁻⁵ M) and not yet introduced into the sand column], it was deducted that the fluorescence signal of Fluometuron had not been affected by the unidentified organic compound. This result allowed us to detect without any ambiguity the presence of Fluometuron transport through the sand column.

Fig.11: Emission Spectra of the Unidentified Organic Compound Present in the sand: 1) before Irradiation; 2) after 70 min of Irradiation

By irradiating during 15 min the samples of the solution collected after one hour of infiltration, at 304 nm excitation, it was found an emission band identical to that of Fluometuron (Fig.13), indicating the presence of this one.

Fig.12: Emission Spectrum of the Unidentified Organic Compound Present in the sand, Collected after 70 min of Irradiation

By plotting the following relationship $I_f = f(t)$, we obtained a curve whose peak intensity was higher at time t = 1 hour, corresponding to the infiltration time of Fluometuron and indicating an infiltration rate of 0.66 cm/min.

Conclusion

This results of the study showed that Carbofuran and Fluometuron are unstable under the effect of UV irradiation. Thus in Senegal, a hot and sunny country, the exposure of these two pesticides to sunlight could lead to their photodegradation.

The study of the effect of solvents on the photodegradation rate of these products showed that the half-life time was higher in water. So contamination of surface waters could take place when the microbial decomposition is slow. The study on the infiltration rate of these two products has shown that they infiltrate quickly into the soil studied. As result of this, the groundwater contamination by these products can become more important than those of the surface waters. Thus in agriculture it is required efficient use of these products in order to reduce or eliminate the risk of surface or groundwater contamination

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