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Study of the Degradation of Haloxyfop-Methyl in Natural Waters in Senegal under the Effect of Solar Radiation and Evaluation of Its Herbicidal Activity

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Abstract

The objective of our study is to examine the impact of solar radiation on haloxyfop-methyl in an aqueous environment and to determine whether it is responsible for herbicidal activity. To do this, we first prepared a 10^{-4} M concentration solution of haloxyfop-methyl in distilled water, well water (at a height of 0.5 m), well water (at a height of 1 m), runoff water and river water, then exposed it to sunlight at regular intervals. This study shows that haloxyfop-methyl is highly unstable when exposed to sunlight. The percentages of photodegradation in these various types of water are 98%, 94.3%, 93%, 94.2% and 58.6% respectively. In all the environments studied, its photodegradation follows first-order kinetics and the half-lives found are relatively short, ranging from 9.18 to 47.47 minutes. A correlation was observed between the photodegradation rate of this herbicide and the amount of dissolved organic matter in these various environments. Indeed, the photodegradation of this herbicide was slower in environments where organic matter was more abundant. The study on herbicidal activity revealed that the initial product was primarily responsible for this activity, not its photoproduct. The latter showed slow degradation, even after 18 days of exposure to sunlight.

Keywords

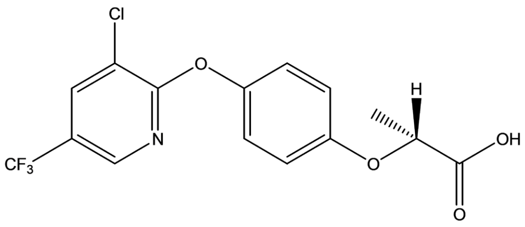
Photodegradation, Haloxyfop-Methyl, Natural Waters, Herbicidal Activity

1. Introduction

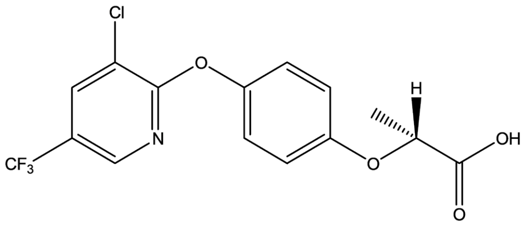
Pesticides, also known as plant protection products, are substances used to control or kill a group of pests in order to improve the quality and yield of crops [1]. Their use in agriculture gradually increased after the Second World War, becoming a widespread practice that has not only increased global food production but also protected plants throughout their development [2]. According to data provided by the FAO in 2018 and 2019, around 4 million tonnes of pesticides are consumed each year, with a significant increase in herbicides (56%), followed by fungicides (25%), insecticides (19%) and other types of pesticides, such as rodenticides and nematicides [3] [4]. Herbicides generally aim to eradicate weeds without affecting crop plants, in order to maximise the benefits of weed control. While pesticides offer many advantages and have unique physicochemical properties, they can also be toxic, mobile and capable of bioaccumulation [5]. They present a major risk in natural waters, as the various processes they undergo in the aquatic environment can lead to their transformation into more toxic substances. This transformation can take place biologically or by photodegradation.

Most studies on the photodegradation of pesticides have been carried out using ultraviolet irradiation sources such as mercury lamps or light-emitting diodes (LEDs). However, the use of these lamps requires the acquisition of expensive equipment and the wearing of protective equipment. This is why we have carried out a study into the photodegradation of haloxyfop-methyl (see diagram below) under the influence of natural sunlight.

The aim of this study is to examine the impact of solar radiation on the herbicide haloxyfop-methyl in various natural waters in the Niayes agricultural zone in Senegal, which is 180 km long and between 5 and 10 km wide, from Dakar to Saint-Louis [5]. We will then determine whether the herbicidal activity is mainly due to the parent molecule or its photoproduct. Haloxyfop-methyl or Gallant is a herbicide from the family of aromatic aryloxyphenoxypropionic acids, used as a pre- and post-emergence herbicide, with known toxicity and teratogenic effects in mammals [6]. Haloxyfop-methyl has been shown to be a potent inhibitor of acetyl coenzyme A carboxylase (ACCase), a key enzyme in fatty acid biosynthesis, obtained from tolerant species [7]. It provokes certain biochemical responses in different plants, leading to a block in fatty acid synthesis and the death of target plants [8]. It can be used to eradicate grassy weeds with a long period of effectiveness, but is ineffective against Cyperaceous plants [9]. Exposure of this product to sunlight in natural waters can lead to its transformation, which could give rise to photoproducts. Haloxyfop-methyl, a pro-herbicide, is demethylated into haloxyfop-R (also known as haloxyfop-P due to its ability to rotate polar light in the plane). If a haloxyfop-S enantiomer is present, for example from haloxyfop-S-methyl precursors, it is rapidly converted to haloxyfop-R in plants [6]. However, the degradation of haloxyfop-methyl in natural waters and sediments leads to rapid production of haloxyfop-R (up to 99% of the initial concentration) by hydrolysis, whether the system is irradiated or dark. But in an irradiated system, two additional metabolites exceeding 10% AR were produced: 4-trifluoromethyl-5-aminopentanol (DE-535 TAP at 17.8%) and the pyridinol isomer DE-535 (at 16.2% AR). In natural water at pH 8.5, DE-535 furan reached a maximum of 6.8% after 1.8 days [10].



Chemical structure of haloxyfop-methyl



Structure of haloxyfop-R

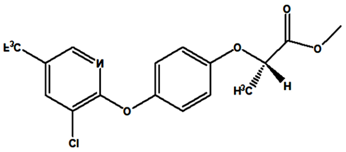
2. Experiences

2.1. Products and Solvents

To carry out this work, we used various natural waters well water (h = 0.5 m), well water (h = 1 m), distilled water, river water and run-off water) and haloxyfop-methyl (98.5%). Haloxyfop-methyl was obtained from sigma-Aldrich (PESTANAL*, analytical standard, Germany). All solutions were protected from light with aluminium foil and stored in a refrigerator.

The chemical properties of haloxyfop-methyl are shown in **Table 1**.

Table 1. Chemical properties of haloxyfop-methyl.

Product	Formula	Molar mass (g/mol)	WS (20˚C)[a] (mg/L)	MP (˚C)[b]	BP (˚C)[c]	Structure
Haloxyfop-Méthyl	$C_{16}H_{13}ClF_3NO_4$	375,7	9,1	−12,4	390,8	

[a] Solubility in water (20˚C) [10], [b] Melting point [10], [c] Bolting point [11].

2.2. Equipment

In this work, we used: a UV-Visible spectrophotometer (GENESYS 30), a pesticide sprayer and a solarimeter for measuring solar energy. Beakers, test tubes, vats, a thermometer, pipettes, micropipettes (50 μL and 1000 μL), an electric balance and filter paper were also used.

2.3. Experimental Procedures

2.3.1. Preparation of Solutions

To carry out this study, a stock solution of pesticide at a concentration of 10^{-3} M was prepared in each of the solvents used. From these stock solutions, daughter solutions of the desired concentration were obtained by successive dilutions. The vials containing the stock solutions were covered with aluminium foil to protect them from light and stored in the refrigerator.

2.3.2. Preparing Water Samples

Natural water samples from the Niayes agricultural zone in Senegal were collected in 1.5 L glass bottles according to standard procedure. The samples were filtered using a syringe filter with a PTFE membrane with a diameter of 25 mm and a pore size of 0.2 µm (Sigma-Aldrich), in order to remove any suspended organic matter.

2.3.3. Determination of Herbicidal Activity

To carry out this work, we used two 2 m^2 grass beds on clay soil. One was exposed to sunlight with an average energy of 1270 W/m^2, while the other was shielded from the light. On each plane, we sprinkled the grass with a pesticide solution according to the recommendations on the bottle: 500 mL of haloxyfop-methyl solution should be diluted in 100 L of water to treat half a hectare of grass.

After each spraying, we sprayed with water every day until the 18th day. We then observed the effect of our irradiated and non-irradiated products on the grasses every other day until day 18.

Abbreviations and acronyms

LED: light-emitting diodes

ACCase: acetyl coenzyme A carboxylase

WS: Solubility in water

MP: Melting point

BP: Bolting point

PTFE: Polytetrafluoroethylene

pH: Potential of hydrogen

TOC: total organic carbon

TC: total carbon

IC: inorganic carbon

λ: Wavelength

K: photodegradation rate constant

PP: Percentage of photodegradation

r^2: correlation coefficient

UV: ultaviolet

FAO: Food and Agriculture Organization of the United Nations

Tirr: irradiation time

$t_{1/2}$: half-life of the photodegradation reaction

Equations

First order kinetic equation: $\ln\frac{A_0}{A} = f\left(t\right)$, $\ln\frac{A_0}{A} = kt$.

Half-life time equation: $t_{1/2} = \frac{\ln 2}{k}$.

Equation for percentage of photodegradation: $PP = \left(\frac{A_0 - A}{A_0}\right)\times 100$.

3. Results and Discussions

3.1. Determination of Organic Matter Content and pH in Natural Water Samples

We used several types of water samples (distilled water, well water (h = 0.5 m), run-off water, well water (h = 1 m) and river water). For each sample, we determined the amount of dissolved organic matter and the initial pH. To determine the total organic carbon (TOC) concentrations, we first determined the total carbon concentrations (TC) and then the inorganic carbon concentrations (IC). The difference (TC - IC) gives us the TOC. The experiment shows that the values found vary with the sample. All the results are shown in **Table 2**.

Table 2. Analytical parameters by type of water sample.

Eaux	TC (mg/L)	IC (mg/L)	TOC (mg/L)	pH_i
Well (0.5 m)	50,75	46,5	4,25	8,1
Distilled water	20,52	20,48	0,04	6,85
Runoff	105,40	93,94	11,46	7,6
Well water (1 m)	98,64	50,08	48, 56	7,8
River water	138,54	56,95	81,59	8,3

TC = total carbon; IC = inorganic carbon; TOC = total organic carbon (TC - IC); pH_i = pH of natural water.

This table shows that dissolved organic matter concentrations vary with the matrix independently of pH. TOC values are relatively higher in river water and well water 1 m deep, followed by run-off water and well water (0.5 m deep) and finally distilled water. Photodegradation reactions will be more or less rapid in environments with higher concentrations of dissolved organic matter. This is because dissolved organic matter can accelerate or delay photodegradation reactions.

3.2. Effect of Irradiation Time on the Electronic Absorption Spectrum of Haloxyfop-Methyl in Various Natural Waters

Figure 1 shows the electronic absorption spectra of haloxyfop-methyl in samples of well water (0.5 m) and river water over the irradiation period. The spectrum of this non-irradiated product shows a band located between 325 and 525 nm, with a peak at around 380 nm. The same result was obtained in the other types of water, showing that haloxyfop-methyl absorbs in the same zone whatever the aqueous matrix.

As irradiation progresses, a decrease in peak intensity is observed in all the cases studied, indicating that haloxyfop-methyl is unstable under the effect of solar radiation in natural waters. This result is in good agreement with the literature. Indeed, studies have shown that photodegradation of haloxyfop-methyl (esters) rapidly gives different photoproducts such as haloxyfop-R (acid), pyridinol, pyridinone and phenol [6] [10].

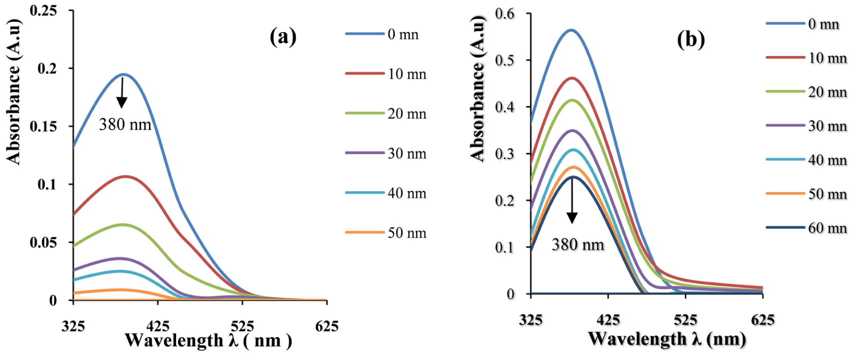


Figure 1. Changes in the absorption spectra of haloxyfop-methyl (10^{-4} M) in well water (0.5 m) (a) and river water (b) as a function of irradiation time.

To better assess the impact of solar radiation on the degradation of haloxyfop-methyl in the different aqueous matrices, we determined the percentage of photodegradation of this herbicide in each aqueous matrix and the half-life times.

3.3. Characteristic Photodegradation Kinetics of Haloxyfop-Methyl

Figures 2(a)-(b) show the curves $\ln\frac{A_0}{A} = f(t)$, respectively for well water (depth 0.5 m) and river water. We obtained lines with a positive slope and a correlation coefficient close to unity, indicating kinetics of order 1. The same result was obtained in all the other cases studied.

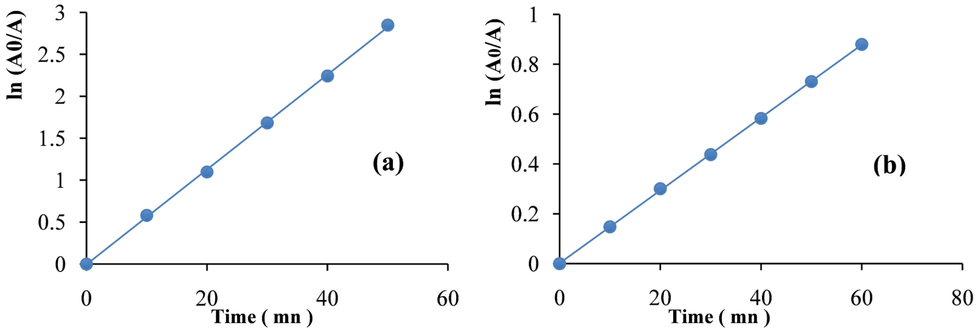


Figure 2. First-order kinetics of haloxyfop-methyl (10^{-4} M) in well water (0.5 m) (a) and river water (b).

From these lines, we determined the rate constants and deduced the half-lives, using the following relationships: $\ln\frac{A_0}{A} = kt$, $t_{1/2} = \frac{\ln 2}{k}$.

Where the rate constant k corresponds to the slope of the straight line.

To better assess the effect of solar radiation on our product in the various matrices studied, we also determined the percentage of photodegradation for a duration of 60 minutes using the formula: $\left(\frac{A_0 - A}{A_0}\right)\times 100$.

In this calculation, A represents the absorbance at an irradiation time equal to 60 minutes (tirr = 60 min) and A0 represents the absorbance at tirr = 0 min.

All the results for the photodegradation kinetics of our product are shown in **Table 3**.

Table 3. Kinetic parameters for the photodegradation of haloxyfop-methyl.

Matrix	K (min^{-1})	$t_{1/2}{}^{(min)}$	Order	PP (%)	r^2
Well water (0.5 m)	$(4,6 \pm 0.08) \times 10^{-2}$	12,24	1	94,3	0,999
River water	$(1,4 \pm 0.03) \times 10^{-2}$	47,47	1	58,6	0,999
Runoff water	$(4,7 \pm 0.08) \times 10^{-2}$	14,7	1	94,2	0,999
Well water (1 m)	$(7,1 \pm 0.1) \times 10^{-2}$	9,79	1	93	0,998
Distilled water	$(7,5 \pm 0.1) \times 10^{-2}$	9,18	1	98	0,999

k = photodegradation rate constant (min^{-1}); $t_{1/2}$ = half-life of the photodegradation reaction (min); PP = Percentage of photodegradation; r^2 = correlation coefficient.

This table shows that the photodegradation of our product obeys first-order kinetics whatever the matrix studied. The same result was obtained by Khonté *et al.* [12] when they studied the photodegradation of the herbicide rimsulfuron in natural waters. Similar kinetics were also found by D.D. Thiaré *et al.* [13] when they studied the photodegradation of the fungicide Benomyl in distilled water, seawater, well water and river water. Similar kinetics were also found by D. Sarr *et al.* [14] when they studied the photodegradation of the herbicide fluometuron in water using UV and fluorescence. This shows that the photodegradation of pesticides in aqueous media generally follows kinetics of order 1.

This table also shows that the photodegradation rate constants for haloxyfop-methyl are relatively high, corresponding to relatively low half-lives. The half-lives found range from 9.18 to 47.47 minutes. This result shows that haloxyfop-methyl is very sensitive to light in natural waters, compared with other pesticides subjected to the action of solar radiation or other more powerful sources of energy, such as ultraviolet radiation. In studying the photodegradation of the herbicide rimsulfuron under the effect of solar radiation in natural waters, Khonté *et al.* [12] found half-lives in excess of 144 minutes, even in the presence of a photosensitiser. In another study conducted on the degradation of two endosulfan isomers (a-endosulfan and b-endosulfan) under the effect of solar radiation in borehole water, S. K. Traoré *et al.* [15] observed a certain stability. The half-lives found were 770 hours and 407 hours respectively. Other authors such as D. Sarr *et al.* [16] and D. D. Thiaré *et al.* [13] and Med Lemine Ould *et al.* [17], studying respectively the degradation of fluometuron, benomyl and metalaxyl by UV radiation in aqueous media, found half-lives 35 times higher for fluometuron, between 2.75 and 26. 85 times higher in the case of benomyl irradiated in various natural waters and 7.6 times higher in the case of metalaxyl compared with haloxyfop-methyl when the latter was irradiated under solar radiation in river water, where its degradation was slower. This shows that haloxyfop-methyl is highly unstable under the effect of solar radiation in natural waters. However, the quantity transformed varies according to the matrix studied. It is relatively higher in distilled water, but lower in river water. In all the cases studied, we note that the percentage of photodegradation of this product is greater than that obtained by S. Halladja *et al.* [18], when they studied the photodegradation of fluometuron under the effect of solar radiation in water. The results of their study showed that less than 30% of this product had disappeared after 6.7 days. A correlation was observed between the rate of disappearance of this product and the quantity of dissolved organic matter. In distilled water, where the quantity of organic matter was lower, degradation was faster. On the other hand, in river water, where the quantity of organic matter was higher, degradation was slower. The slow photodegradation observed in river water could therefore be attributed to the high presence of dissolved organic matter. This phenomenon could be explained, as in the case of the photolysis of Carbofuran [19], Diuron [17] and metalaxyl [20] in the presence of dissolved organic matter, by the fact that the dissolved organic matter fixes the molecules of the product and absorbs much of the UV radiation. This could slow down the photodegradation reaction. This result obtained in the case of our product is different from that obtained by Regi Mathew and colleagues [21] when they studied the photodegradation of the herbicide metolachlor in water in the presence of dissolved organic matter. In this study, the rapid photodegradation of this herbicide in the presence of dissolved organic matter could be explained by the presence of free radicals produced by the dissolved organic matter, which would be capable of initiating the photodegradation reaction of the herbicide molecules. This difference in behaviour noted in the two studies shows that dissolved organic matter plays a dual role:

- They can either absorb UV-visible radiation before it reaches the pesticide molecules, thereby slowing down or even inhibiting the photodegradation reaction;
- They can also produce free radicals capable of initiating the photodegradation reaction in areas where the pesticide does not absorb directly, thereby accelerating the pesticide's photodegradation reaction. This dual behaviour of organic matter could depend not only on the structure of the pesticide and the origin of the aqueous matrix, but also on the other constituents of the soil.

3.4. Evaluation of the Herbicidal Activity of Haloxyfop-Methyl

Two 2 m^2 grass beds were used to assess this herbicidal activity.

Using the protocol described in 2.3.3, we observed yellowing of the grasses exposed to the sun from the sixth day after application of the haloxyfop-methyl solution (**Figure 3**). It was not until the sixteenth day that the grass was completely dried out (**Figures 3(B)-(D)**). In the case of the plane kept away from light, the herbicidal action was more obvious. In fact, the majority of grasses were dried out in less than four days and total drying was observed from the sixth day of application of our product (**Figure 3(B)**). These results show that the herbicidal activity can be attributed to the product or its photoproduct (haloxyfop-R), which also

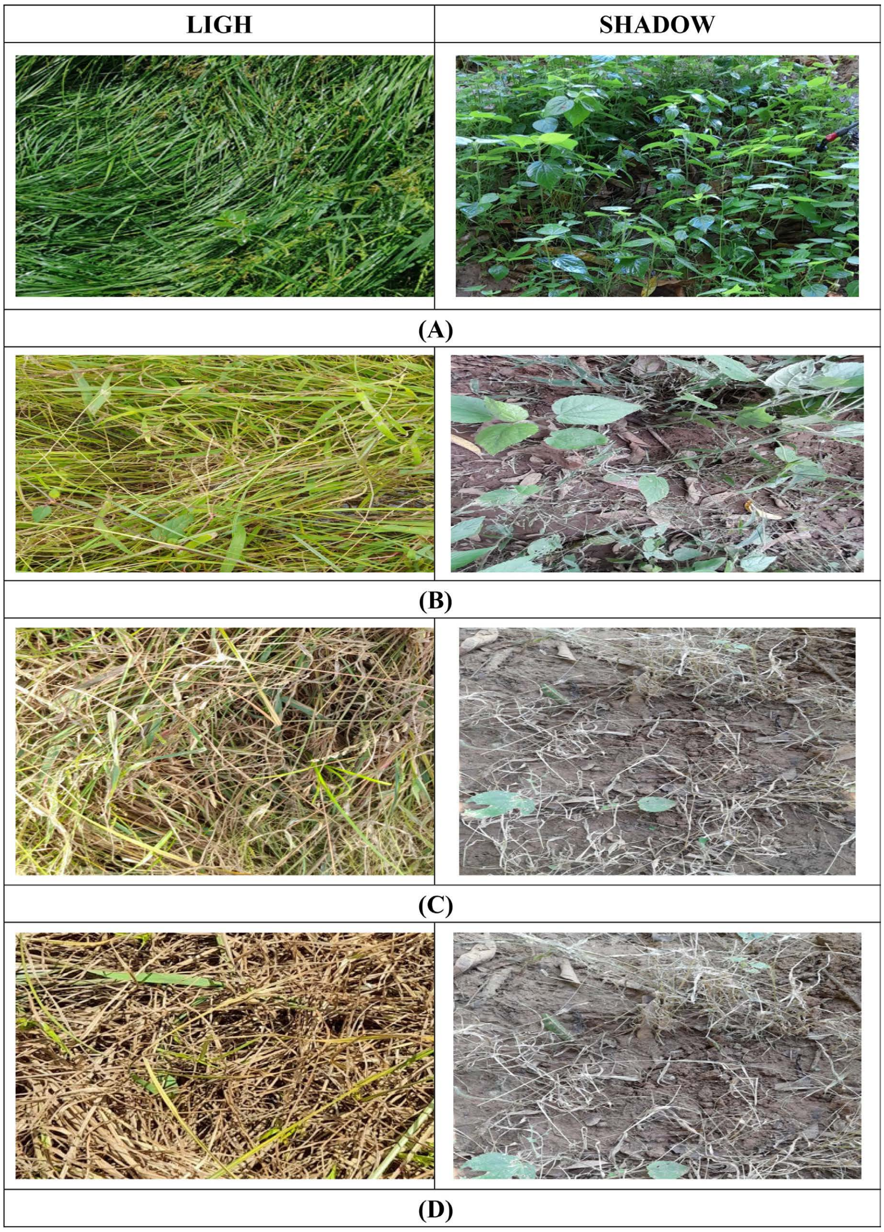


Figure 3. (A) Grass condition on day 1 of haloxyfop-R-methyl application; (B) Grass condition on day 6 of haloxyfop-R-methyl application; (C) Grass condition on day 14 of haloxyfop-R-methyl application; (D) Grass condition on day 18 of haloxyfop-R-methyl application.

has her bicidal activity ([6]). However, the herbicidal activity observed was greater without irradiation. This activity could therefore come mainly from the initial product. Several authors, including Buhler *et al.* [22] and Poiger *et al.* [23], have demonstrated that haloxyfop-methyl has greater herbicidal activity and greater efficacy than certain herbicides in the same family, such as fluozifop-buthyl or its metabolite haloxyfop-R. The results of our study are therefore consistent with these observations. These same authors also showed that the herbicidal activity of a compound or its degradation product can depend not only on the nature of the plant studied, but also on the sources of degradation, which can lead to the formation of different degradation products.

4. Conclusions

The results of this study show that in natural waters, haloxyfop-methyl is notoriously unstable under the effect of solar radiation. Consequently, the probability of contamination of natural waters is low, but this may not be the case for its various photoproducts such as haloxyfop-R.

Studies on herbicidal activity have shown that it is more intense in the absence of light than in the presence of light. In this case, it is possible that the herbicidal activity is present in the product itself and in the photoproducts. The results of this study will provide a reliable basis for monitoring the photodegradation of herbicides used in the Niayes agricultural zone in Senegal. In addition, the results will facilitate the safe use of herbicides in environmental matrices. The aim is to ensure optimum use of pesticides by farmers, who are often unaware of the impact of solar radiation on these substances. This will ensure the best possible health and safety conditions.

Contributions of Authors

Souleymani, A. wrote the original manuscript and analysed and interpreted the results. Diop, Y., is a doctoral student. He carried out the sampling and participated in the work. Yatte, B.S. is a doctoral student. He participated in the work. Mané, A., is a doctoral student. He also participated in the sampling. Sarr, D. participated in the design, analysis, and interpretation of the study results. Thiaré, D.D., participated in the analysis and interpretation of the results. Sall, C. participated in the experimental protocol and the work was carried out in his laboratory. Tine, A. contributed to the design of the study. Coly, A. is the principal supervisor of this work.

Conflicts of Interest

The authors declare no conflicts of interest.

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