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A Comparaison Of UV-V Is Absorption And PIF Method By Determination Of Deltamethrin Residues In Senegalese Natural Waters

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Abstract

A photo-induced fluorescence (PIF) method was developed for the determination of the pyrethrinoid deltamethrin pesticide. The photo-conversion under UV irradiation of the pesticide deltamethrin into strongly fluorescent photoproduct was performed in several media. The time of UV-irradiation and the binary mixture solvents (methanol: water, v/v) of PIF parameters were optimized. Analytical figure of merit for the PIF determination of deltamethrin was satisfactory, with rather wide linear dynamic range (LDR) values of one to two orders of magnitude, low limit of detection (LOD) and of limit quantification (LOQ) values of deltamethrin were respectively 3.07 and 10.25 ng/mL according in the binary mixture methanol: water (80:20, v/v). Relative standard deviation (RSD) value was 0.6%. PIF validated by comparing its analytical performance to those of a standard UV absorption spectrophotometric UV-Visible method. The optimized PIF method was applied to the quantitative analysis of deltamethrin pesticide in tap and well water samples collected in Senegalese agricultural area. Standard addition procedure prior to extraction steps in dichloromethane was applied and satisfactory mean recovery percentage values as 98.27% and 100.84% were to be found.

Keywords: *Photo-induced fluorescence; Deltamethrin; Pesticides; Quantitative analysis; Natural water samples.*

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I. Introduction

Pesticides are phytosanitary products used to protect crops from weeds and pests and thus to increase agricultural yields. In fact, the use of pesticides in agriculture does not only have beneficial effects but also harmful consequences for the environment and human health [1]. More generally, pesticides present environmental hazards that are manifested mainly by their slow degradation in nature and also their probably transformation into toxic by-products. Thus excessive or inappropriate use by farmers can lead to contamination or accumulation of these compounds or their residues in many environmental matrices such as soil, atmosphere, food, surface and groundwater and then indirectly cause more or less serious health problems in humans [2-7].

In this work we are interested with deltamethrin insecticide in the family of pyrethroïd widely used in the agricultural Niayes area in Senegal [4, 7]. Deltamethrin or (*S*)-cyano-3-phenoxybenzyl (1*R*, 3*R*)-3-(2, 2-dibromovinyl)-2, 2-dimethyl cyclopropane carboxylate (Figure 1) is a synthetic pyrethroïd and possesses high insecticidal activity. It is used to control many species of mites and insects like whiteflies, field crops, glass house crops, vegetables [8, 9]. Appreciable levels of pyrethroïd residues can occur in food commodities from crops, food, soils, sediments, surface and groundwater [8-12]. Pyrethroïd are now employed worldwide as insecticides in forestry agriculture. Their no persistence is essentially due to their high photodegradability [13, 14]. However, high toxicity to fish aquatic species, and honeybees was observed for several pyrethroïd [15-17]. The exposure of pyrethroïd to temperature and UV irradiation can produce reactions leading to other products that may be much more toxic than the original pesticide [18, 19]. In Senegal, where climatic conditions are harsh, pyrethroid can contaminate soils and waters due to their thermochemical instability and their toxicity.

Several analytical methods have been developed for the determination of deltamethrin based on the use of chromatographic techniques, which include high performance liquid chromatography with various detectors (UV, Mass and fluorescence) [20-23], gas chromatography with-electron capture detector (GC-ECD) [8, 11]. Other methods such as electrochemical methods [24], indirect redox spectrophotometric [9] and spectrophotometric based on the condensation of the hydrolyzed aldehyde group with anthranilic acid, 4-aminoantipyrine and 2-chlorophenyl hydrazine [10] were also used. These techniques required a large number of solvents and also some limitations in terms of high cost of instruments used in routine analysis. Direct spectrofluorimetric methods [25, 26], photochemically-induced fluorescence methods [18, 27-35] and thermochemically-induced fluorescence derivatization (TIFD) methods [36-38] are still one of the important techniques for the determination of pesticides because they are less expensive and easy to use. Recently, Mendy et al. [36-38] have developed a new, simple and sensitive TIFD method for determining of metolachlor and buprofezin in aqueous and cyclodextrins media. Their TIFD method is based on the thermolysis transformation of naturally non-fluorescent pesticides into fluorescent complex o-phthalaldehyde-thermoproduct(s) in water. Coly and al. [39] have developed a PIF method by used a cyclodextrin –enhanced intensity fluorescence to determine deltamethrin in water.

Since deltamethrin is not naturally fluorescent, we have developed in this present work a simple and sensitive photomochemically-induced fluorescence (PIF) method to transform it into fluorescent photomoproduct in mixture binary solvent methanol-eau, 80:20, and v/v. This analytical method have been applied to evaluate the deltamethrin residues in spiked tap and well waters collected in the Niayes area according to the standard addition procedure.

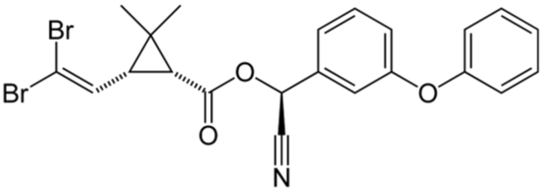

Figure 1: Chemical structure of deltamethrin.

II. Materials And Methods

Reagents

Technical-grade of deltamethrin (purity-99.8%) was obtained, from Sigma Aldrich. The HPLC grade solvents used in these experiment (methanol, ethanol, acetonitrile, dichloromethane and acetone were obtained from sigma Aldrich, ethyl acetate from scharlau. Ultra-pure water was used (mrc laboratory technologic).

Apparatus

A jasco UV-Vis spectrophotometric (model V- 570) with matched quartz cells and connected at computer with spectro-manager software was used for all spectral measurements.Rotatif evaporator (Bùchi Rotavapor R-124), paper filter diameter (0.48 µm), syringe filter PTFE membrane, diameter (150 mm) pore size 0.45 µm and a balance (sortorius , LA230S) were used. All fluorescence measurements were performed at room temperature on a Cary Eclipse spectrofluorimeter, interfaced with a microcomputer.

Procedure

Stock standard solutions preparation

A stock solution deltamethrin (10^{-3} mol/L) was prepared by dissolving accurately weighed in 50 mL in methanol. The stock solution was stored in a freezer at 8°C, through deltamethrin is generally believed to be stable at least 6 months at room temperature.

The working standard solutions with concentrations in the range ($3 – 19.10^{-5}$ mol/L) were prepared by appropriate dilution of the stock standard solution with the HPLC grade solvents used in a 5 mL volumetric flasks. Solutions were freshly prepared before use. These solutions were used for linearity investigation and for evaluation of precision, repeatability and accuracy of the method.

Water samples preparation

Tap and well water samples, receiving from run-off water from agricultural fields sprayed with delamethrin were collected. Water Samples were filtered through a syring filter PTFE membrane, diameter (150 mm) pore size (0.45 µm) in order to remove suspended solid water. Then, the water samples were fortified with known quantities of deltamethrin standard solution.

UV-Vis Absorption Analytical Measurement

The Liquid-Liquid Extraction (LLE) method was used for deltamethrin absorbance measurements. The fortified water samples (Tap and well) were transfered to a 125 mL separator funel buchi and shaked vigourously with the extraction solvent dichloromethane.

The process was repeated twice with (2x5 mL) with the same extraction solvent. The organic phase was passed through anhydrous sodium sulfate to remove traces of water contents and the extracts were concentrated by evaporation with a water bath at 40 °C. The dry extracts were reconstituted with acetonitrile and binary mixture acetonitrile: water (60:40, v/v) and analyzed by UV-Vis spectrophotometry. The absorbances were measured at the maximum wavelength to establish the calibration curves and compared to the standard calibrations curves in order to determine the deltamethrin concentration. The absorbance measurements of standard solutions allowed to determine the unknown concentrations of deltamethrin in water samples.

PIF Analytical Measurements

500-mL water samples were collected in 1-L amber glass bottles from well and tap waters in a agricultural Senegal's Niayes area. All samples were filtered through a quartz filter with a 0.45 µm pore size polytetrafluoroethylene (PTFE) membrane filter disc in order to eliminate the suspended organic matter, and stored at 4°C. We proceded to a solid phase extraction (SPE), using dichloromethane as extracting solvent. To obtain a water matrix free of organic contamination, the SPE C18 cartridge was preconditioned with 5 mL methanol followed by 5 mL ultrapure water; 50 mL water samples were passed through the cartridge before being enriched.

PIF analysis were performed by using standard addition procedure. The filtered water samples were spiked in 10-mL flasks with standard solution of deltamethrin (0.4 ng mL^{-1}). Then, five aliquots of 5-mL portions of these spiked water samples were introduced into five flasks, and increasing concentrations of the pesticide standard solution from 8 – 1100 ng mL^{-1} for deltamethrin, were added into four flasks, the first corresponding to the blank. Flasks were adjusted to the mark with the optimum mixture solvent 80:20, v/v methanol/water. The PIF signal was measured at the maximum emission wavelength after UV-irradiation at the optimum time. In all cases, the PIF intensity measurements were corrected for the background signal using the appropriate blanks. PIF signals were carried out in triplicate and expressed as mean values to optimize the analytical results.

III. Results And Discussions

Spectrophotometric absorption UV-Visible method

The absorption spectra of deltamethrin in acetonitrile, methanol, ethyl acetate and ethanol are presented in figure 2. For all these organic solvents the same large band of absorption was obtained at the maximum wavelength of 278 nm. These bands characterize the delocalization of the π electrons of the aromatic nucleus allowing π - π* transition. The molar extinction coefficients vary between 1140 and 2958.1 L mol^{-1} cm^{-1} in organic solvents and 2201.4 - 3273.9 L moL^{-1}cm^{-1} in the binary mixtures acetonitrile: water (v/v).

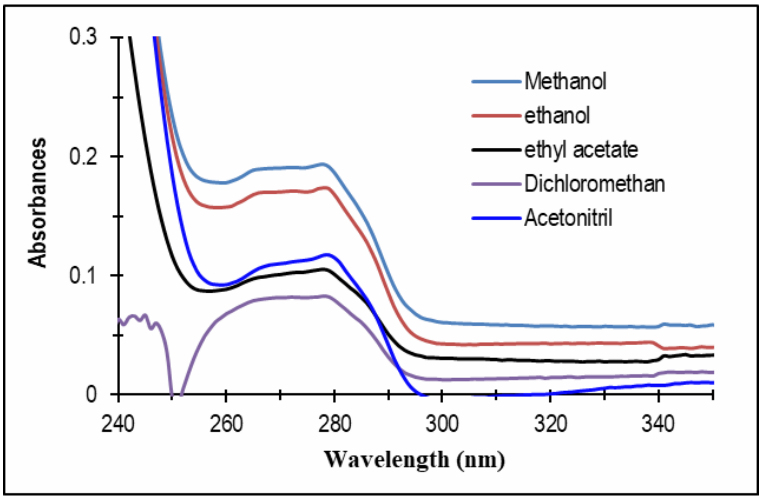

Figure 2: Absorption spectra of deltamethrin (25 µg/mL) in organic solvents.

In the aim to select the optimal analytical, the spectrophotometric method for deltamethrin determination was optimized by comparing the limits of detection values in various medium. As shown in table 1, the calibration curves appeared with large linear dynamic range (LDR) and correlation coefficient values (r^2) very close to unity, which indicate a good linearity of the spectrophotometric analytical curves.

The limits of detection (LD) values were calculated on the basis of deltamethrin concentration giving a signal-to-noise ratio (S/N) of 3 (IUPAC criterion). The LD significantly varied with the organic solvents and the binary mixture acetonitrile: water (v/v) respectively, ranging from values of 0.06 – 0.42 µg mL^{-1} and 0.05-0.58 µg mL^{-1}. The limits of quantification (LQ) values were between 0.19-1.35 µg mL^{-1} and 1.17-1.9 µg mL^{-1}. The relative standard deviation (RSD) values less than 2% indicates a good repeatability of measurements.

Table 1: Statistical data of deltamethrin calibration curves in various medium.

Solvents	LD (µg/mL)	LQ (µg/mL)	RSD (%)	R^2	ε (L mol^{-1} cm^{-1})
Acetonitrile	0.06	0.19	0.3	0.999	2301.6
Methanol	0.09	0.31	0.1	0.995	2958.1
Dichloromethan	0.40	1.35	0.8	0.995	1381.6
Ethyl acetate	0.23	0.78	0.2	0.993	1140.0
Ethanol	0.15	0.51	0.1	0.995	2462.6
Acetonitril:water (60:40, v/v)	0.06	0.19	0.1	0.999	3111.8

The difference between the values of LD in acetonitrile and the other organic solvents, and the binary mixture acetonitrile: water (60:40, v/v) of the other binary mixture demonstrated that the great analytical interest of utilizing acetonitrile and the binary mixture acetonitrile: water (60:40, v/v).The LDs values of 0.06 µg mL^{-1} obtained for acetonitrile and acetonitrile: water (60:40, v/v) by UV-Spectrophotometric method was judged very low. Therefore, this low value suggested that UV-Spectrophotometry can be considered as a convenient and sensitive method for determination of deltamethrin residues in samples Tap and well water. Figure 3 and 4 summarise all results.

Analytical Application to fortified Tap and well water samples

The method developed has been applied for the determination of deltamethrin in samples water.

The averages recoveries after standard addition at different concentration of deltamethrin prepared in binary mixture acetonitrile: water (60:40, v/v) in the fortified samples tap and well water were calculated respectively to be 91.40 and 92.03 %. Any traces of deltamethrin were not observed in the blank and blind samples. The calibration standard and addition curves were paralleled, therefore any interferences were not observed. Table 3 and 4, figures (3) summaries all results.

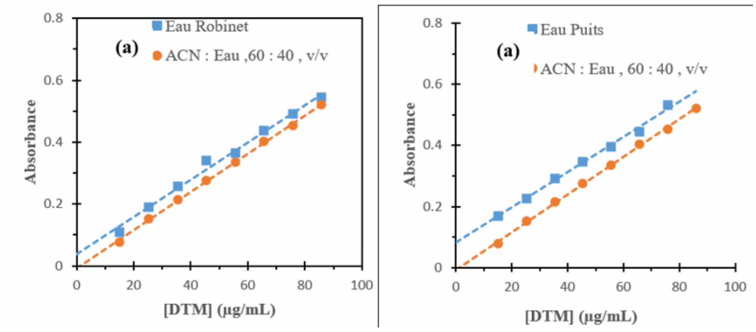

Figure 3: Calibration (●) and standard addition (■) curves of deltamethrin in tap (a) and well (b) water samples in optimum condition binary mixture (acetonitrile : water, 60 :40, v/v)

Table 2: Recovery percentages of deltamethrin with the addition standard method by Liquid-Liquid Extraction from Tap and well water.

Type of sample	Added (ng/mL)	Found (ng/mL)	Recovery (%)	Mean Recovery (%)	RSD (%)
Tap Water	6.00	4.72	78.68	92.03	2.36
	8.00	7.19	115.35		
	10.00	9.22	92.28		
	12.00	10.86	90.55		
	14.00	12.38	88.45		
	16.00	14.39	89.94		
	20.00	16.90	84.54		

Well Water	6.00	5.69	94.84	91.95	1.57
	8.00	7.23	90.38		
	10.00	9.38	93.87		
	12.00	11.40	95.02		
	14.00	12.92	92.34		
	16.00	14.55	90.98		
	20.00	17.35	86.77		

Photochemical Induced Fluorescence method (PIF)

The aim of this study was to develop a rapid, selective and sensitive PIF method for determining deltamethrin residues in Senegalese natural waters, due to their use in vegetable and fruit market gardening in Senegal's Niayes area. Indeed, the PIF study was performed in optimum binary water/methanol 20/80, v/v mixtures after optimization. The PIF spectral properties of this pesticide is reported in Table 5. Figure 5 shows the excitation/emission matrix and excitation and emission spectra of deltamethrin PIF in the binary mixture water/methanol, 20/80, v/v after UV-irradiation. As can be seen in figure 5b, the PIF excitation spectrum presented three peaks at 220, 250 and 290 nm, on the other hand, emission spectrum was rather well resolved and presented only one single, wide band. However the PIF excitation and emission maximum wavelength being located respectively at 250 and 344 nm in the binary mixture water: methanol, 20:80, v/v. No important shift of the PIF excitation and emission maximum wavelengths wasn't noted with the binary mixtures, but the PIF signal intensity and irradiation time significantly varied according versus the concentrations to the medium (Figure 2).

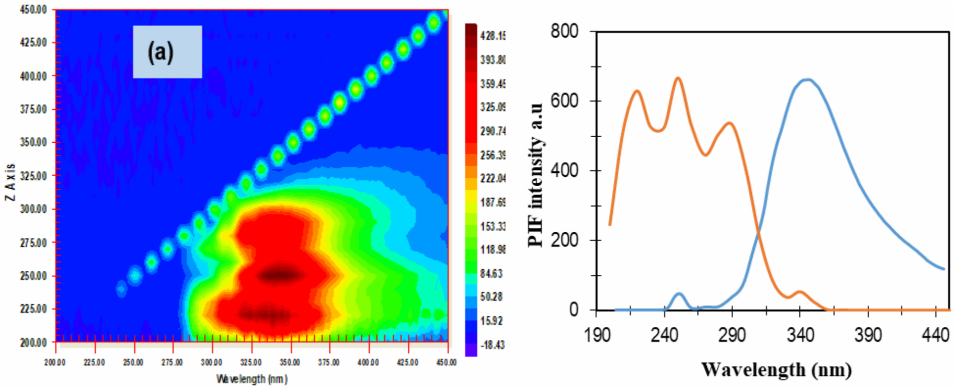

Figure 5: Excitation-emission matrix (a) and fluorescence spectra (b) of deltamethrin (1µg/mL) after 1 min UV-irradiation in methanol: water (80:20, v/v).

The effect of the percentage of water in the methanol-water binary mixture was studied on the PIF intensity of delthamethrin at the optimum condition of irradiation time (t_{irr}^{opt} = 1min). The intensity of the PIF quickly increases with the percentage of water (from 10 to 20%), reaching a maximum value for 20% water (80% methanol), and then gradually decreases as the percentage of water increases from 20 to 80%. (Fig. 6). Similarly, the effect of the percentage of water on the PIF intensity in binary alcohol-water and dimethylsulfoxide (DMSO)-water (70-50%) mixtures was investigated by several authors in the determination of pyrethrinoid residues, including fenvalerate, cypermethrin and bifenthrin in formulations and natural waters. [40, 41].

The results showed that the variation of water polarity in binary mixtures with water content plays an important role in increasing the PIF signal and in the optimization of the PIF method.

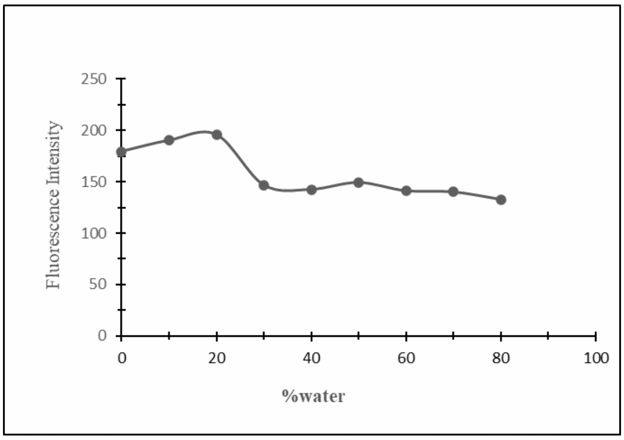

Figure 6: Effect of the variation of water percentage on the PIF signal of deltamethrin (1µg.mL^{-1}) in water-methanol mixtures at optimum irradiation time (t_{irr}opt= 1min).

Photolysis kinetic study

The evolution of the photodegradation reaction of deltamethrin is followed as a function of the variation of the PIF signal, and the maximum excitation and emission wavelengths of the pesticide, as a function of the irradiation time, at room temperature and in the studied media were recorded. The PIF intensity curve of deltamethrin as a function of irradiation time (t_{irr}) is characterised by a steady increase in PIF intensity. The latter reached the maximum value at the optimal irradiation time (t_{irr}^{opt}), followed by a progressive decrease of the signal. The t_{irr}^{opt} value varied from 1 to 10 minutes in the water: methanol mixture (20:80 v/v) (fig11) and according to other settings table (1). The increase of the PIF signal on the first part of the curve corresponds to the photochemical formation of fluorescent photoproduct(s), and the second part indicates their progressive degradation into non- or weakly fluorescent compound(s). We did not search for the structure of the fluorescence photoproduct, which should be very similar to some recent studies on the hydrolysis reaction and biodegradation (3-phenoxybenzaldehyde) by bacteria [41]. The excitation and emission spectrum after the optimal condition is shown in (Figure 7).

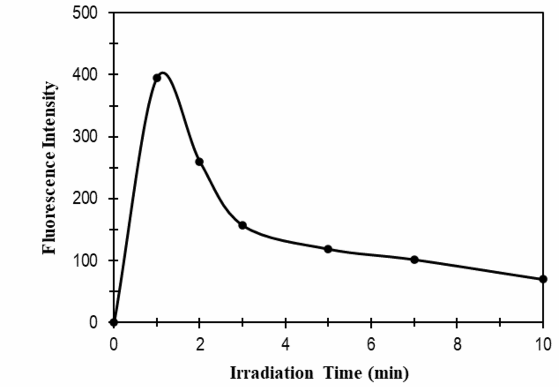

Figure 7: Effect of the UV irradiation time of deltamethrin (1µg/mL) on fluorescence intensity in the binary mixture methanol: water (80:20, v/v).

Analytical Figure of Merit

To assess the analytical value of the PIF method, we established the analytical performance values of deltamethrin in the different media studied, which are reported in Table 1. For the validation of the PIF method, the analytical performance of the pesticide deltamethrin was also determined by the UV-VIS absorption spectrophotometric method (Table 1).

The linearity of the PIF intensity and absorbance versus concentration curves [I = f(c) and A = f(c)] were obtained by the PIF method and by UV-VIS absorption spectrophotometry, used as a reference method. The dynamic linearity range (LDR) values were spread over more than two orders of magnitude, with correlation coefficients (r^2) very close to unity, indicating excellent linearity of the analytical curves for both methods (Figure 1). The Relative Standard Deviation (RSD) values for the PIF method were 0.6% and ranged from 0.09% to 0.7% for the UV-VIS absorption spectrophotometry of deltamethrin depending on the medium studied. The latter have shown that the reproducibility of the measurements of the PIF method is practically more satisfactory than that of UV-VIS absorption spectrophotometry.

The limits of detection (LOD) and quantification (LOQ) were calculated on the basis of an analyte concentration giving a signal-to-noise ratio (S/N) of 3 and 10 respectively (IUPAC criteria). A lower LOD value of 3.1 ng mL^{-1} was obtained with the PIF method, while the corresponding limit of quantification (LOQ) was 10.3 ng mL^{-1} depending on the medium after 1 min of irradiation. A LOD value of about 33 ng mL^{-1} in methanol was also reported by Coly and Aaron [42] for the PIF determination of deltamethrin in technical formulations, in a combined study with other aromatic insecticides. The LOD and LOQ values obtained by UV-VIS absorption spectrophotometry were significantly higher than those determined by the PIF method (Table 3).

This confirmed that the PIF method was much more sensitive than the UV-VIS absorption spectrophotometry method, and that the latter was chosen as the analytical method for the determination of deltamethrin residues in the environment. Therefore, the validation of PIF by UV-VIS absorption spectrophotometry can be considered satisfactory. In addition to this, it is relevant to note that the lowest LOD and LOQ values were obtained by PIF in the 20/80,v/v water/methanol mixture for deltamethrin, which explains the already mentioned selection of this medium for future analytical studies of the pesticide deltamethrin by PIF.

Table 3: PIF and UV-VIS absorption spectral properties and analytical figures of merit of deltamethrin in organics solvent

	Solvents	λabs (nm)	LOD (ng/mL)	LOQ (ng/mL)	r^2	RSD (%)
UV-VIS absorption	ACN/Water (60 :40, v/v)	278	60	190	0.999	0.1
Method PIF	MetOH/Water (80 :20, v/v)	λex/λem(nm)	LOD (ng/mL)	LOQ (ng/mL)	r^2	RSD (%)
		250/350	3.07	10.25	0.997	0.6

Analytical Applications

We examined the application of the PIF method to the quantitative analysis of tap and well water samples collected from an agricultural area in Senegal. We used the UV-VIS absorption spectrophotometric method to validate the analytical application of the PIF method. The water samples were all fortified and analysed as described in the experimental section. We tried to quantify deltamethrin in these water samples using the standard addition procedure with solid-liquid extraction, using dichloromethan as extraction solvent, in order to eliminate possible interfering species in the water samples.

Study of spiked water samples

Using the PIF method, the standard addition procedure was applied to tap water and natural well water collected from the Niayes area of Senegal, which were spiked with a standard mixture of deltamethrin methanol/water 80/20, v/v (concentration 0.4 µg/mL). Afterwards, as described in the experimental section, an increasing concentration of deltamethrin standard solution was added, and the PIF measurements were performed.

The standard addition curve was linear and the slope was found to be very close to that measured for the calibration curve, demonstrating the absence of significant interference from any compounds present in the water samples. Satisfactory recoveries were obtained (Figure 8) at different deltamethrin concentrations for the standard addition procedure in the PIF method, indicating the efficiency of the extraction procedure. Recovery percentages ranged from about 87.00% to 114.00% in well water, and from about 76.70% to 116.25% in tap water for deltamethrin for the PIF method used (Table 4).

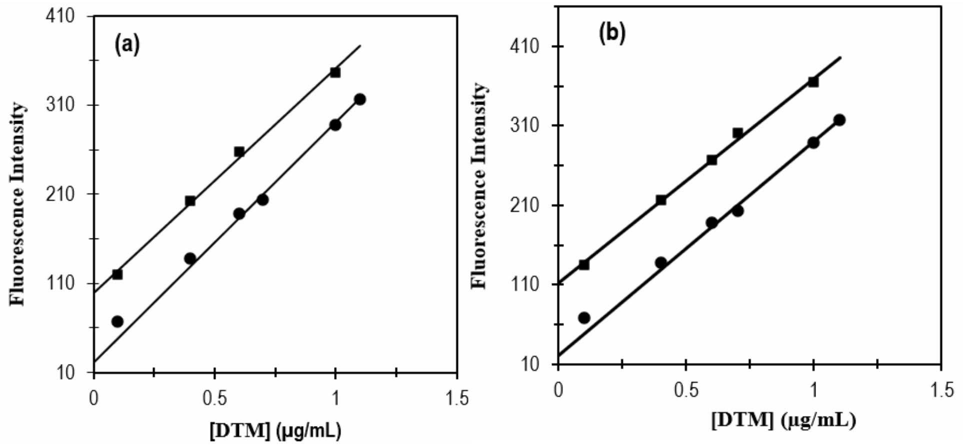

Figure 8: Calibration (●) and standard addition (■) curves of deltamethrin in tap (a) and well (b) water samples in optimum condition by method PIF.

Table 4: Analytical applications of the PIF method to the determination of deltamethrin, and evaluation of recovery values in spiked in tap and well waters Senegalese area obtained by the standard addition procedures.

Type of sample	Added (μg/mL)	Found (μg/mL)	Recovery (%)	Mean Recovery (%)	RSD (%)
Tap Water	0.10	0.10	103.82	100.84	1.03
	0.35	0.38	107.47		
	0.45	0.52	116.25		
	1.52	1.16	76.75		
	2.53	2.09	82.84		
Well Water	0.10	0.11	112.00	98.27	2.27
	0.35	0.40	114.00		
	0.45	0.48	106.00		
	1.52	1.16	76.70		
	2.53	2.09	87.00		

IV. Conclusion

In this work we have developed a very simple, inexpensive, sensitive and accurate PIF method for the determination of deltamethrin residues in natural water samples from Senegal. We demonstrated the analytical suitability of the PIF, which was optimized in a 20/80, v/v water/methanol mixture. The analytical figures of merit confirmed the good sensitivity and accuracy of the PIF for deltamethrin. The applicability of the PIF method to the determination of deltamethrin pesticide residues in fortified natural water samples at the ng mL^{-1} level, with satisfactory recovery values, was also demonstrated. In addition, the PIF method was satisfactorily validated through UV-VIS absorption spectrophotometry, used as a reference method. Therefore, the results of this study confirm the analytical value of the PIF method for the quantitative analysis of deltamethrin residues in environmental water samples. Studies are currently underway in our laboratories to combine PIF detection with a flow injection system to improve the speed of analysis for the routine determination of residues of this pesticide, deltamethrin, in natural water samples.

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