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Monitoring of Spatial and Temporal Distribution of Fenitrothion Residues in Soils and Sediments in Sahel Using Carbon Fiber Microelectrode

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Abstract

Fenitrothion (FNT) is an organophosphate insecticide used to control locust infestations. Its most stable and toxic metabolite is 3-methyl-4-nitrophenol (MNP). Several formulations of pesticides based on FNT were used in Sahel to control locust infestations. However, the concentration levels and persistence of FNT residues in soil and sediments remain unknown due to its high analysis cost, which is generally carried out using chromatographic methods. Previous works showed that electrochemical methods can be used to monitor FNT using carbon fiber microelectrodes. The main objective of this work is to determine the concentration levels and persistence of FNT residues in soils and sediments samples in the Sahel using carbon fiber microelectrodes during an agricultural campaign. The obtained results showed on the one hand that the concentration level of FNT residues varies from 6.56 to 346.50 µg/kg in soils samples and from 6.56 to 331.75 µg/kg in sediments and on the other hand that FNT did not persist beyond three months. These results showed that FNT did not persist from one agricultural season to the next and confirmed the rapid dissipation of FNT in Sahel ecosystems.

Keywords

Fenitrothion, 3-Methyl-4-Nitrophenol, Carbon Fiber Microelectrode, Soil, Sediments, Sahel

1. Introduction

The use of synthetic pesticides in modern agriculture is an essential practice, widely used worldwide to increase yields and ensure food security [1]. Among pesticides, organophosphate (OPs) compounds form the large group of chemicals that used over the past 60 years for protecting crops, livestock, human health and as warfare agents [2].

Fenitrothion (FNT), or O,O-dimethyl-O-(4-nitro-m-tolyl) phosphorothioate, is an insecticide from the organophosphate pesticide. It degrades in the environmental matrices through biotic and abiotic processes to produce metabolites [3] [4] (**Figure 1**). Photolysis and hydrolysis are the main pathways of its abiotic degradation. Indeed, under natural conditions, the photodegradation of FNT is slow particularly in the absence of moisture [5]. However, it becomes important in the presence of humidity [5]. The half-life of FNT degradation by photolysis is estimated to 85 days on sandy loam soil exposed to sunlight [5]. The main metabolites of FNT are fenitrooxon and MNP [6] [7] which is the most persistent and toxic of the metabolites of FNT. Ito *et al.* reported that FNT hydrolysis occurs faster under anaerobic conditions (flooded environments) [8]. The half-life of FNT biodegradation ranges from 4.4 to 53.7 days in non-flooded soils and to 3.9 and 10.9 days in flooded soils. Miyamoto carried out a study on the persistence of FNT residues in four (04) soils in Japan under flooded and non-flooded conditions [9]. The results show that submerging the soil considerably accelerates the rate of degradation of FNT residues with an estimated half-life of 4 days in the soils of Moriyama and Katano under flooded conditions against more than 20 days for the soil in non-flooded conditions. A half-life of 24 hours under environmental conditions has been reported by Sekizawa *et al.* [10].

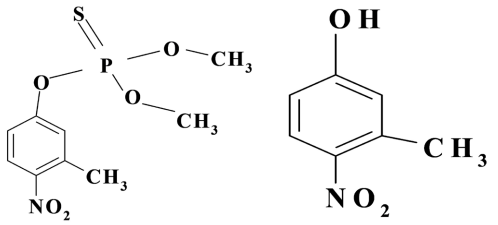

Figure 1. Structure of fenitrothion (a) and 3-methyl-4-nitrophenol (b) [3].

In addition, the formation of metabolites differs depending on the soil water regime. Indeed, in non-flooded soils, MNP and CO_2 are the main degradation

products of FNT. In contrast, in flooded soils, amino-FNT is the major metabolite of FNT.

Other research on the persistence of FNT in five (5) tropical soils of India, carried out by Adhya *et al.*, 1987, showed that the degradation of the residues of FNT takes place faster in flooded soils than in non-soils [11]. The half-life periods reported for five (5) soils were estimated to be 3.9 to 10.9 days under flooded conditions versus 4 to 97 days for the same soils in non-flooded conditions. Under aerobic conditions, FNT degrades biologically with an estimated half-life of 2 days in sandy loam soil. The main non-volatile degradation product of this pesticide is MNP, the concentration of which can reach 20% of the initial amount applied in 3 days.

Biodegradation of FNT in the soil takes place slowly in a neutral environment and is nevertheless the main route of degradation. The half-life of FNT is estimated to be 4.4 years in soil pH 6.2 [8].

Like other organophosphate compounds, FNT is toxic [5] [12]. Its metabolites, such as fenitrooxon and MNP are known for their potential for bioaccumulation in food chains [13].

The northern of Burkina Faso, as other regions of Sahel, is a favorite area for locusts. Known for their genetic diversity, their ability to move, the extreme density of their swarms and their voracity for plants, locusts pose a formidable threat to agriculture in many countries. The fight against these insects represents a major issue, in particular for food security in the Sahel countries. Chemical control has so far remained the most effective means against these plant pests [13] [14].

As part of the integrated fight against the locust plague in the Sahel and in order to protect the environment, the Sahelian Pesticide Committee (CSP) has used several pesticide formulations of which the active molecule is FNT [15]. Thus, large quantities of FNT are used in the Sahel during periods of great upsurges in invasions and could cause much damage to ecosystems.

However, the concentration levels and persistence of FNT and MNP residues in soil and sediments in relation to the fight against locust invasion in the Sahel are not known.

Previous work related to the use of FNT in the fight against locust invasion in the Sahel, already reported, concerned mainly the study of the impact of this pesticide on the dynamics of desert locust population [14]-[16].

The small number of works on FNT carried out in Sahel is partly justified by the high cost of monitoring pesticide residues which is generally done by the chromatographic method, coupled with sensitive and selective detectors such as Nitrogen Phosphorus Detector (NPD), Mass Spectrometry (SM) detectors [17] [18].

However, in view of the constraints linked to the use of chromatographic methods for monitoring organophosphorus molecules (long analysis time, high consumption of organic solvents, loss of residues by degradation during chemical treatment of the sample, the cost high equipment), many fast and inexpensive reliable alternative methods have been developed. It is in this context that electro-

chemical methods associated with specific and sensitive electrodes are increasingly used in the monitoring of organophosphorus pesticides in various environmental matrices [19] [20].

Previous works [21]-[23] showed that carbon fiber microelectrodes can be used to monitor organophosphorus compounds containing the nitro (-NO_2) group in environmental matrices.

The objective of this work is to monitor the spatial and temporal evolution of FNT residues in the Sahel using carbon fiber microelectrodes which are sensitive, efficient and low-cost electrochemical tools.

2. Materials and Methods

2.1. Reagents and Materials

Carbon fibers (12 µm diameter, 5 mm length) were purchased from Cytec Engineered Materials (West Paterson, NJ, USA) and used for carbon fiber microelectrodes elaboration.

Standards of fenitrothion (97%), 3-méthyl-4-nitrophenol (98%), Potassium ferrocyanide ($K_4Fe(CN)_6$), and K_2HPO_4 (99%), sulfuric acid (98%), Sodium hydroxide (98%), were purchased from Sigma-Aldrich. KH_2PO_4 (98%) was purchased from FLUKA.

Absolute ethanol (99.9%) and acetonitrile (99.9%) were purchased from AnalaR NORMAPUR.

All solutions were prepared with deionized water (pH 7.2, conductivity < 0.1 µS/cm and DOC < 0.1 mg/L).

2.2. Apparatus

A helical steel auger with screw-on tools was used for soil sampling.

A RETSCH brand analytical sieve with a 75 µm mesh, certified to ISO 3310/1, is used for sieving soil and sediment samples. An OHAUS brand analytical balance with a precision of 10^{-4} g was used for weighing samples; A CU-5000 IEC brand centrifuge was used to separate the liquid and solid phases after extraction; A BüCHI brand R-200; B-490 rotary evaporator is used for concentrating extracts. A PalmSens potentiostat (PalmSens 3 Instrument, Netherlands) was used for electrochemical analysis by using carbon fiber microelectrodes as working electrodes.

An Agilent Technologies chromatograph equipped with an Agilent J&W HP-5 column and Nitrogen Phosphorus Detector was used to confirm the concentration of FNT in soil.

A CS744 Analyzer, LECO brand was used to perform TOC analysis in soils.

2.3. Location of Study Area

The Sahel region of Burkina Faso is located between the 200 and 600 mm isohyets and has two seasons [24]: a rainy season from June-July to September-October and a dry season from October to May. The Sahel zone is characterized by a highly variable tropical climate in space and time with high temperatures between 33˚C

and 36˚C throughout the year. Since warmer air can hold more water vapour, evapotranspiration in Sahel is high and affects the water supply and the amount of surface water available for agriculture [25].

All selected sampling sites were treated with FNT formulations during the previous agricultural season.

Sampling sites are shown in **Figure 2**.

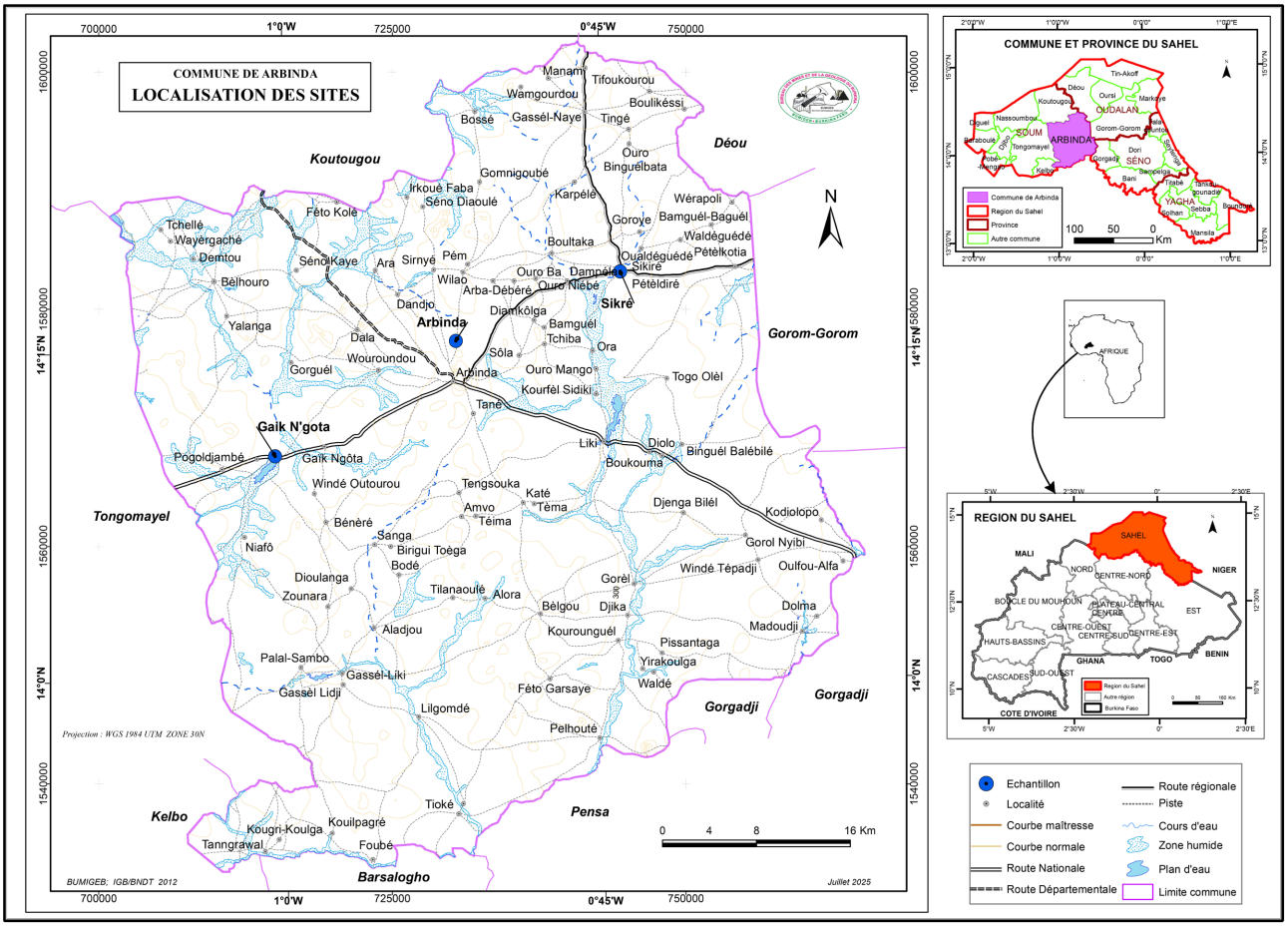

Figure 2. Sampling sites location.

2.4. Pesticides Application Procedure

Fenical 450 UL (FNT in UL formulation) was applied using a MicroUlva device. At each treated site, the insecticide was applied in one day. With a treatment of one (1) liter per hectare, the actual dose of 450 g of FNT per hectare was applied in coverage following lines spaced 7 meters apart at a forward speed of 1 m/s in accordance with FAO guidelines for the control of the desert locust [26].

2.5. Soils and Sediments Sampling Strategy

Soil and sediment samples were collected at T0 (immediately before pesticide application), at T1 (1 hour after pesticide application), and then at T7, T15, and T90 seven, fifteen, and ninety days after pesticide application, respectively. The sam-

ples were taken from a depth of 0 - 20 cm using an auger and following the method described by Mathieu and Pieltain [27] (**Figure 3**).

Sampling at each site was carried out in the morning.

Due to practical difficulties encountered related to the wet conditions at the samples, a mixture of 13 subsamples weighing approximately two (2) kg was immediately collected from the sites, placed in a labeled bag and stored in a cooler before being transported to the laboratory. After drying and sieving the mixture of 13 subsamples, six (6) composite samples are taken using a riffle splitter for analysis, including three (3) by SWV and three (3) for confirmation by GC-NPD.

Samples were dried under ventilation and away from sunlight for 48 hours then sieved to 75 microns and pulps stored by freezing to −20˚C [28].

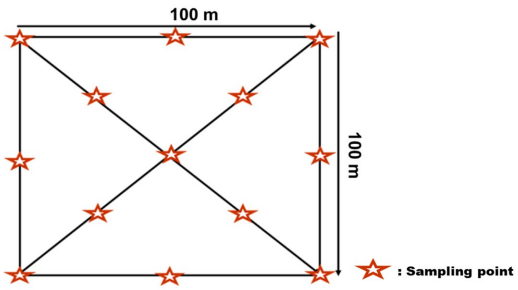

Figure 3. Sampling points repartition.

Samples were immediately taken before the insecticide was applied in order to obtain control samples (T0) that provide information on the initial content of FNT residues or its main metabolite in the soil. These control samples were used for the physico-chemical characterization of the soils.

To consider the temporal evolution of pesticide residues, soils and sediments samples were taken at each site at the following times:

- T1, one hour after pesticides application;
- T7, one week after pesticides application;
- T15, two weeks after pesticides application;
- T90, three months after pesticides application, to verify whether FNT has completely dissipated in the sites.

Samples sites location and nature of matrix sampled are given in **Table 1**.

Table 1. Nature of the matrix sampled by sampling location.

Location	Samples	Samples label and sampling location coordinates
Sikré	Soil	N 1583209 W 0744399
Sikré	Sediments	N 1583256 W 0744500
Arbinda	Soil	N 1577359 W 0730419
Gaïk N'goata	Sediments	N 1567625 W 0715034

2.6. Samples Characterization

The samples taken from each site prior to treatment were subjected to particle size

analysis. The method used was that described by Devis and Freitas (1984) [29]. One hundred grams of the sample were dried and separated by vibration on a series of superimposed sieves with mesh sizes ranging from 2 μm to 2 mm. The contents of each sieve were then weighed and the fraction of the sample collected was reported as a percentage of the total sample quantity.

Total Organic Carbon (TOC) analysis in soil was performed using digestion and combustion method.

2.7. Extraction and Clean up of Pesticide Residues

A 20 g of soil sample with a predetermined moisture content was placed in a 250 mL Erlenmeyer flask. After adding 20 mL of acetonitrile, the mixture was stirred for one hour before being centrifuged at 500 rpm for 15 min. At the end of centrifugation, two phases separate: the solid phase consisting of the soil and a liquid phase or aliquot, represented by the extracts. The aliquot was collected, filtered and evaporated to dryness in a rotary evaporator at approximately 50˚C. The dried residues are then solubilized with a minimum amount of absolute ethanol and transferred to a 50 mL flask. The flask is then filled to volume with phosphate buffer at pH 7.2.

The solution thus obtained was then transferred to the electrochemical cell for analysis.

The extraction involved samples from each site, replicated twice to assess the variability in the analytical data.

2.8. Analysis

Stock solutions (1 $g{\cdot}L^{-1}$) of FNT and MNP were prepared by dissolving the equivalent mass of FNT and MNP in absolute ethanol. Working solutions 10 mg/L of FNT and MNP are then prepared. 0.1 M of PBS (pH 6.2) was used as supporting electrolyte.

All electrochemical experiments were performed in square wave voltammetry (SWV). Measurements were performed using an electrochemical analyzer PalmSens (PalmSens Instrument, Netherlands) connected to a personal computer using Ivium PC and PSLite software. A three-electrode configuration was employed consisting of a carbon fiber microelectrode as working electrode (CFME) (diameter Φ = 12 μm, an SCE electrode as the reference electrode, and a platinum wire as the auxiliary electrode. The carbon electrode surface was renewed by a homemade electrochemical treatment. Indeed, the carbon fiber microelectrodes were first pretreated electrochemically in a mixture of sulphuric acid H_2SO_4 (0.5 M)/Ethanol (50/50 w/w) followed by a treatment in 0.1 M Phosphate Buffered Saline (PBS) pH 6.2 using following conditions: potential scanning rate: 100 $mV{\cdot}s^{-1}$ in the potential range −1.3 to 1.3 V/SCE during 20 cycles. A quality control of the CFME cleanness with visual and electrochemical tests was used. Electrochemical experiments were carried out in a 50 mL glass voltammetric cell at room temperature.

All measurements were carried out under ambient conditions. The appropriate solutions were transferred into the electrochemical cell. The optimization parameters are the following: Scanning was performed from −1.8 to +1 V versus SCE with a step potential of 10 mV, amplitude of 60 mV, and a frequency of 60 Hz.

Before each experiment, the solutions were deaerated by bubbling nitrogen, and the electrochemical cell was kept under a nitrogen atmosphere throughout the experiments.

Analytical curves were carried out in 0.1 M PBS at pH 7.2 and at variable concentrations of FNT. using the standard addition method.

For example, an appropriate volume of the FNT standard solution was added to the PBS. The mixture was then simultaneously stirred and bubbled for ten (10) minutes. The SWV voltammogram corresponding to the electrochemical response of FNT is then recorded ten seconds after having stopped the agitation and the bubbling of the solution.

Voltametric measurements were carried out using a *PalmSens Instrument PC233 Potentiostat* (*Netherlands*) controlled using *PalmSensVs1.60 software*. Data management was controlled by *PSLITE 1.7.3 software*.

3. Results and Discussion

3.1. Samples Characterization

The results of characteristic parameters of tudied soils are summarized in Table 2, showing that the studied soils are acidic. Indeed, the ferruginous soils found in Sikré are poor, with a very acidic pH (4.7), and low levels of nutrients and exchangeable bases [30].

As for the sandy-loam soils found at Arbinda, they generally have a slightly acidic to neutral pH, often optimal between 5.5 and 7.0. The sandy-loam soils found at Gaïk N'Goata are known for their significantly lower pH and higher nutrient content [31].

The acidic nature of all these soils could accelerate the acid hydrolysis of the FNT [8]-[10].

Table 2. Characteristics of soils and sediments samples.

Sites	Nature of sample	Geographic coordinates	Sample characteristics	% organic matter	% carbon matter
Sikré	Soil	N 1583209 W0744399	Sandy loamy soil	0.79	0.46
	Sediments	N 1583256 W0744500			
Arbinda	Soil	N 1577359 W0730419	Ferruginous soil, slightly leached	0.55	0.39
Gaïk-N'Goata	Sediments	N 1567625 W0715034	Loamy soil	1.69	0.98

3.2. Calibration Curve Establishment by Square Wave Voltammetry Using Carbon Fiber Microelectrode

The calibration curve was established by Square wave voltammetry using carbon fiber microelectrode as working electrode in PBS 0.1 M pH 7.2 at frequency = 60 Hz, amplitude 60 mV, increments 10 mV and the reference electrode is ECS. The carbon fiber microelectrode is poised at a potential of −1.8 V to +1 V vers ECS and the FNT concentration varies from 10 - 200 µg/L. The obtained result is shown in Figure 4.

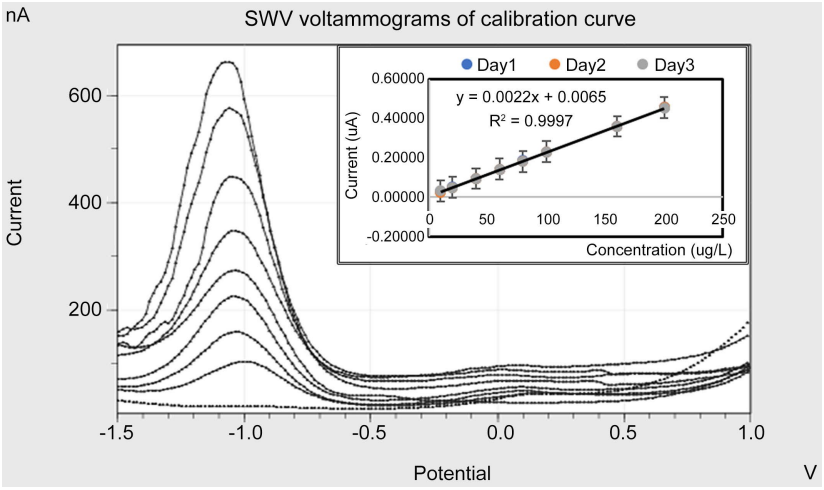

Figure 4. SWV voltammograms of calibration curve, FNT concentration range: 10 - 200 µg/L.

It appears clearly that this calibration curve varied linearly upon FNT concentration increase with linear regression model, demonstrated excellent linearity across the 10 - 200 µg/L and was represented by the linear equation: Ip (µA) = 0.0022C (µg·mL^{-1}) + 0.0065 with R^2 = 0.9997 (Figure 4).

The assessment of calibration function showed that the optimal line of best fit minimizes the sum of squared residuals, indicating that at least one predictor variable has a non-zero coefficient.

That assumes that linear regression is suitable model.

3.3. Analytical Method Performance Validation

The validation of the reported electrochemical method for the determination of FNT in soil was conducted comprehensively in accordance with EURACHEM/CITAC guidelines and ISO/IEC 17025 requirements, ensuring its suitability for quantitative analysis. The method underwent rigorous evaluation for key performance parameters, including ruggedness (Table 3 and Table 4), specificity/selectivity (Table 5), matrix effects (Figure 5 and Table 4), linearity, accuracy (Table 6), precision (Table 7), limits of detection (LOD) and quantification (LOQ) (Table 8), and measurement uncertainty (Table 9).

Specificity of the method was evaluated by assessing potential interferents. As shown on Table 3, only MNP showed significant interference with the signal of FNT.

The interference of MNP on FNT response could be explained by the fact that the nitro group of MNP is reduced at −1.08 V (vs. ECS), close to the reduction potential of the nitro group of FNT (−1.02 V). This interference of MNP on FNT response has already been reported [32] [33].

Table 3. Parameters evaluated in the ruggedness test.

Parameter		*Value*		
Mass of assay	A	20 g	a	40 g
pH of phosphate buffer	B	5	b	9
Time of shaking	C	1 hour	c	2 hours
Increment of analytical method	D	5 mV	d	50 mV
Amplitude of analytical method	E	20 mV	e	120 mV
Frequency of analytical method	F	5 Hz	f	20 Hz
Volume of extraction solvent	G	20 ml	g	40 ml

Table 4. Evaluation of results from a ruggedness study of the analytical process.

Parameter	*Mean of results at normal value*	*Mean of results at alternative value*	*Difference*	*Significant effect at 95% confidence interval*
A	$\frac{(s+t+u+v)}{4}=53.193$	$\frac{(w+x+y+z)}{4}=53.233$	$\Delta_A=0.040$	-
B	$\frac{(s+t+w+x)}{4}=54.833$	$\frac{(u+v+y+z)}{4}=51.593$	$\Delta_B=3.240$	+
C	$\frac{(s+u+w+y)}{4}=53.160$	$\frac{(t+v+x+z)}{4}=53.265$	$\Delta_C=0.105$	-
D	$\frac{(s+t+y+z)}{4}=54.805$	$\frac{(u+v+w+x)}{4}=51.620$	$\Delta_D=3.185$	+
E	$\frac{(s+u+x+z)}{4}=52.143$	$\frac{(t+v+w+y)}{4}=55.350$	$\Delta_E=3.208$	+
F	$\frac{(s+v+w+z)}{4}=55.593$	$\frac{(t+u+x+y)}{4}=50.833$	$\Delta_F=4.760$	+
G	$\frac{(s+v+x+y)}{4}=52.440$	$\frac{(t+u+w+z)}{4}=53.985$	$\Delta_F=1.545$	-

Table 5. Effect of interferents on detection of FNT.

Interfering substance	Interferent concentration (µg/L)	Measured current response (%)	RSD (%) (n = 3)
Na^+	50	99.5	1.73
K^+	50	99.7	1.02
NO_3^-	50	99.2	1.86
SO_4^{2-}	50	99.2	1.06
4-nitrophenol	50	95.8	0.97
4-aminophenol	50	94.8	1.13
Profenofos	50	98.4	1.35
Dimethoate	50	99.8	1.26
3-methyl-4-nitrophenol	50	89.6	0.93

Table 6. Statistical assessment of method accuracy.

Parameters	Calculated values	Theoretical values	Null hypothesis/conditions of validity
Cochran's test	$C_{calc.} = 0.4046$	0.684 C (0.05; 5; 3)	The treatments are equally effective at 95% confidence interval/$C_{calc.} < C_{crit}$
Comparative test of homogeneity of variances Levene's test	0.4123	3.478 F (0.05; 4; 10)	The variance is equal across groups at 95% confidence interval./$F_{calc.} < F_{crit}$
Validity of average recovery rate			Average recovery rate range of 70% - 110% at 95% confidence interval.
Confidence limits (%)	89.12 - 94.76 (RSD range from 1.19% to 1.47%)		

Matrix effects were ruled out through the analysis of multiple blank soil samples (Figure 5), supported by consistent recovery rates (89.12% to 94.76%) (Table 4).

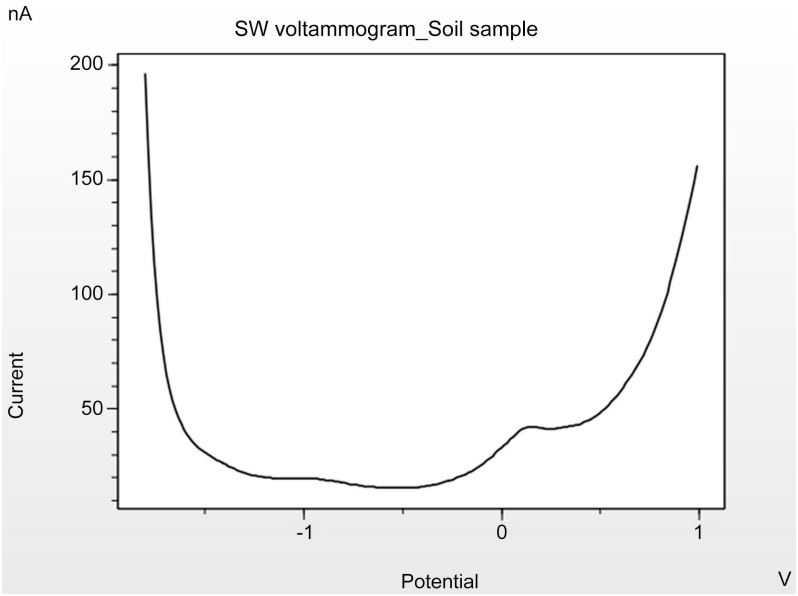

Figure 5. SWV voltammogram of soil blank sample; pH = 7.2; frequency = 10 Hz, amplitude 100 mV, increments 20 mV, Reference electrode: ECS.

The accuracy of the method was established through recovery studies at multiple spiked levels, yielding recoveries between 89.12% and 94.76% with low relative standard deviations (Table 6).

Recovery (%) was calculated using the following formula:

$$\text{Recovery rate}\left(\%\right)=\frac{C_{soil}\left(\mu g/kg\right)\times 100}{C_{theor}\left(\mu g/kg\right)}$$

FNT residue levels in spiked soil were assessed using the following formula:

$$C_{soil}\left(\mu g/kg\right)=C_{cell}\left(\mu g/L\right)\times\frac{V_{solvent}\left(L\right)}{m_{soil}\left(kg\right)}$$

C_{soil}: concentration of FNT residues in soil;

C_{cell} (µg/L): concentration of residues in the cell, evaluated using a calibration

curve;

C_{theor}: The theoretical value expected from spiked soil.

m_{soil}: mass of soil used for the analysis.

$$C_{theor}\left(\mu g/kg\right)=\frac{C_{stand}\left(\mu g/L\right)\times V_{stand}\left(L\right)}{m_{soil}\left(kg\right)}$$

V_{stand}: volume of the FNT standard solution used to spike the soil.

Table 7. Statistical analysis of precision data.

Parameters	*Calculated values*	*Theoretical values*	*Null hypothesis/conditions of validity*
Cochran's test	$C_{calc.}$ = 0.3986	0.617 C (0.05; 3:10)	The treatments are equally effective at 95% confidence interval/$C_{calc.} < C_{crit}$
Within group standard deviation (S_r)	0.0021 (N = 24)	-	S_r is less than Repeatability Limits (95% of confidence interval).
between-group standard deviation (S_b)	0	-	
Intermediate precision standard deviation (S_I)	0.0021 (N = 24)	-	There is no significant difference in the analytical results obtained under different conditions.
Reproductibility standard deviation (S_R)	1.043 (n = 8)	-	S_R is less than Reproducibility Limits (95% of confidence interval).
Repeatability Limits (*95% of confidence interval*)	0.0069 (n = 8)	t_{crit} = 2.365 (α = 0.05; ν = 7)	-
Reproductibility Limits (*95% of confidence interval*)	3.488 (n = 8)	t_{crit} = 2.365 (α = 0.05; ν = 7)	-
Validity of Intermediate precision			
Fisher test	0.621	2.657 F (0.05; 7; 16)	There is no significant difference between the groups/$F_{calc} < F_{crit}$
Comparative test of homogeneity of variances			
Levene's test	0.472	2.657 F (0.05; 7; 16)	The variance is equal across groups at 95% confidence interval/$F_{calc.} < F_{crit}$
Within-day precision	1.404%	-	The precision is equal across groups at 95%/No significant difference between within-day precision and between-day precision
Between-day precision	1.579%	-	

Table 8. Experimental data for LOD and LOQ assessment.

N° Sample	*Current* (μA)	*Concentration* ($\mu g/kg$)	*LOD* ($\mu g/kg$)	*RSD* (%)	*LOQ* ($\mu g/kg$)
1	0.0114	18.98			
2	0.0137	21.52			
3	0.0115	19.14			
4	0.0134	21.19			
5	0.0097	17.11	**6.56** **(n = 10)**	**9.87**	**19.87** **(n = 10)**
6	0.0100	17.41			
7	0.0105	18.05			
8	0.0141	22.02			
9	0.0146	22.61			
10	0.0152	23.22			

Table 9. Sources of uncertainties and standard uncertainty estimate.

Parameters	*Sources of uncertainty*	*Uncertainty components*	*Estimate (X)*	*Uncertainty*	*Uncertainty contribution u(x)*	*Relative Standard uncertainty u(x)/x*
Weighing (m_{soil})	Balance routine use Balance daily drift	Standard deviation of balance calibration	20 g	0.064 g	$0.064\,g/\sqrt{3}$	0.00185
$V_{solvent}$	Pipette of 20 mL	Pipette calibration uncertainty	20 mL	0.03 mL	$0.03\,mL/\sqrt{3}$	0.00087
	coefficient of volume expansion of acetonitrile	Volume of acetonitrile expansion	20 mL	$20 \times 4\ ^{\circ}C \times 1 \times 10^{-3}{}^{\circ}C^{-1}$	$80\times10^{-3}\,ml/\sqrt{3}$	0.00023
C_{cell}	Micropipette of 50 uL	Micropipette calibration uncertainty	100 µL	0.50%	$0.005\,\mu L/\sqrt{3}$	0.00003
	volumetric flask of 50 mL	volumetric flask calibration uncertainty	50 mL	0.06 mL	$0.06\,mL/\sqrt{3}$	0.00069
	coefficient of volume expansion of the water in analysis conditons (24˚C)	Volume of water expansion	50 mL	$50 \times 4\ ^{\circ}C \times 2.1 \times 10^{-3}{}^{\circ}C^{-1}$	$42\times10^{-3}/\sqrt{3}$	0.00001
	Agitation and centrifugation Volume of the aliquot Concentration of residues Recuperation with absolute ethanol	Mean recovery + Run to run variation in recovery	100	1.218	0.01218	0.00012
	Filling to 50 mL	volumetric flask calibration uncertainty	50 mL	0.05 mL	$0.05\,mL/\sqrt{3}$	0.00058
	Calibration curve	Relative standard deviation of slope and intercept	100	Racine $((Sa \times 100/a)^2 + (Sb \times 100/b)^2)$	0.2550	0.00255
	pH Increment Amplitude Frquency Microelectrode area Temperature viscosity of supporting electrolyte Diameter of the microelectrode Diffusion coefficient of species	Repeatability and reproductibility relative standards deviation	100	Racine $((Sr \times 100/S_{totale})^2 + (S_R \times 100/Mean)^2)$	0.01940	0.00019
Purity and stability of reagents	FNT (97%)	Purity of reagent	100	3%	$0.03/\sqrt{3}$	0.01732
	KH_2PO4 (98%)		100	2%	$0.02/\sqrt{3}$	0.01155
	K_2HPO4 (99%)		100	1%	$0.01/\sqrt{3}$	0.00577
	Acetonitrile (99.9%)		100	0.1%	$0.001/\sqrt{3}$	0.00058
	Absolute ethanol (99.9%)		100	0.1%	$0.001/\sqrt{3}$	0.00058
	Stability of stock solution	Stability of reagent relative standard deviation	100	$SI*100/S_{total}$	0.2187	0.00219

3.4. Analysis of Samples

Samples were analyzed by means of calibration curve presented in Figure 4.

The results of the electrochemical analysis are presented in Figure 5 and Figure 6.

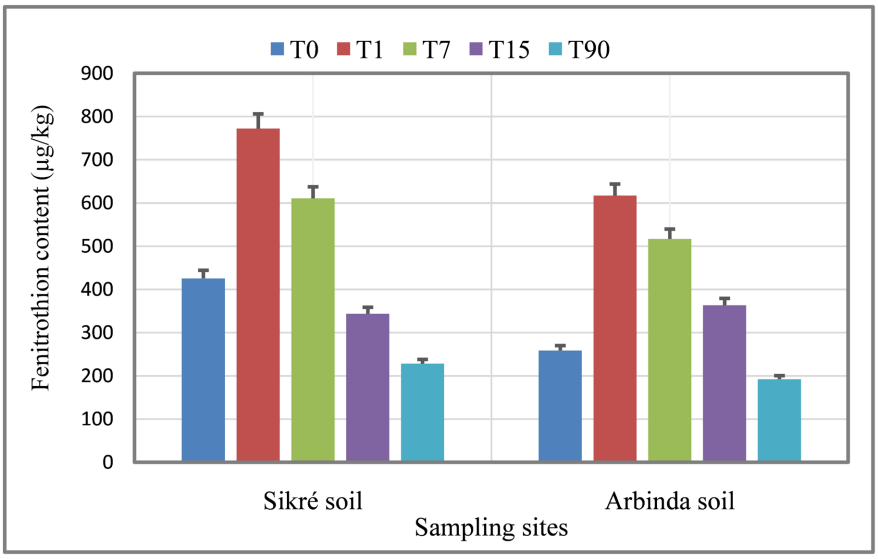

Figure 6. Temporal variation of FNT residues content in soils at Sikré and Arbinda.

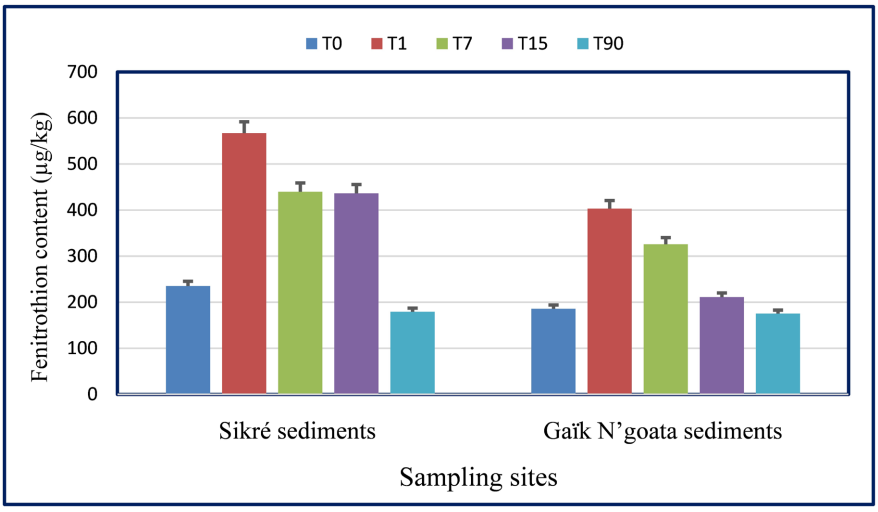

Figure 7. Temporal variation of FNT residues content in sediments at Sikré and Gaik N'goata.

The FNT residues content found in soil and sediment samples, are respectively illustred in Figure 6 and Figure 7 as histograms representing, temporal evolution of FT residues content.

These histograms show that all soil and sediment samples collected before and after treatment (T0, T1, T7, T15, and T90) contain electroactive residues that are reduced on the surface of the carbon fiber microelectrode. The highest concentrations of these residues were recorded in both soil and sediment samples collected

from all sites at T1 (one hour after treatment).

Furthermore, the spatiotemporal evolution of residues in the soil is similar to that in the sediments. Specifically, an increase in residues was observed when moving from T0 (taken before spraying) to T1, and a decrease in content is observed from T1 to T90.

Moreover, the concentration levels of residues observed in samples taken at T90 suggest that the residues found in samples taken at T0 are not FNT residues. In other words, the residues found in the samples taken at T0 and T90 are electroactive residues that interfere with the FNT residues on the surface of the carbon fiber microelectrode.

Indeed, the FNT molecule is hydrolyzable, photodegradable, and biodegradable [4] [5]. The half-life of FNT reported in the literature is estimated to be between 4.4 and 53.7 days [8]. Under Sahelian conditions, the half-life of FNT has been estimated to be 24 hours [10], 36 hours [34], and 6 days [35]. The sites were treated in October, at the end of the rainy season in the Sahel. At the time of sampling, the humidity and light conditions were ideal for accelerating the volatilization as well as biotic and abiotic degradation of FNT in the Sahel region.

Table 10. Results of the comparative analysis of FNT by GC-NPD.

Sites	Nature of sample	Sampling period	SVW results (µg/kg) (n = 3)	GC-NPD results (µg/kg) (n = 3)	SWV corrected values (µg/kg)
Sikré	Soil	T0	425.75 (RSD = 3.05%)	<0.2 (RSD = 0.98%)	<6.56
		T1	772.25 (RSD = 2.63%)	312 (RSD = 1.23%)	346.5
		T90	228.25 (RSD = 3.13%)	<0.2 (RSD = 1.17%)	<6.56
Arbinda	Soil	T0	258.75 (RSD = 2.72%)	<0.2 (RSD = 0.93%)	<6.56
		T1	617 (RSD = 1.97%)	343.87 (RSD = 1.43%)	273.13
		T90	192.34 (RSD = 2.66%)	<0.2 (RSD = 2.11%)	<6.56
Sikré	Sediments	T0	235.25 (RSD = 2.97%)	<0.2 (RSD = 2.32%)	<6.56
		T1	567 (RSD = 1.81%)	309.69 (RSD = 2.25%)	331.75
		T90	179.5 (RSD = 3.11%)	<0.2 (RSD = 1.19%)	<6.56
Gaik N'goata	Sediments	T0	186 (RSD = 2.84%)	<0.2 (RSD = 1.09%)	<6.56
		T1	403.25 (RSD = 2.94%)	201.18 (RSD = 1.92%)	217.25
		T90	175.25 (RSD = 3.01%)	<0.2 (RSD = 2.56%)	<6.56

Considering the slight difference in redox potential of the nitro group of MNP compared to that of FNT, the interference of MNP to FNT response (**Table 5**),

could be responsible of cathodic peak observed in the voltammograms of samples taken at T0 and T90.

To confirm this assumption, soil samples taken at T0, T1 and T90 were analyzed by GC-NPD (**Table 10**).

The results of the chromatographic analysis (**Table 10**) confirmed that the samples taken at T0 and T90 do not contain any FNT residues. However, in the sample taken at T1, the FNT residue content was estimated to 312 µg/kg. These results differ from those obtained by the electrochemical method.

Given the slight variation in residue levels observed at times T0 and T90 in all matrices, on the one hand, and the results of the GC-NPD confirmation, on the other, a correction was applied to the results of the samples collected at times T0, T1, and T90, according to the formula below.

$$C_{\text{corrected}}(t) = C_{\text{SWV}}(t) - C_{\text{SWV}}(t_0)$$

where:

$C_{\text{corrected}}(t)$: Corrected concentration at t;

$C_{\text{SWV}}(t)$: Concentration at t found by SWV;

$C_{\text{SWV}}(t_0)$: Concentration at t_0 found by SWV.

This correction which applies to all matrices, accounts for the highest background interference, considers the small variation in the concentration of interferents over time.

The corrected results show that the residues content in the samples taken at T0 and T90 is less than <6.56 µg/kg, which is the detection limit of our method. Furthermore, in the sample taken at T1, the residue content is approximately 346.5 µg/kg. This content is within the same range as that obtained by the chromatographic method (312 µg/kg).

These results confirm that the residues found in samples taken at T0 and T90 are not FNT residues. Although FNT is weakly volatile, studies reported in the literature [36] [37] show that volatilization is one of the main pathways for the dissipation of FNT residues in the Sahel. Low in arid environments, it is thought to be particularly significant during the hot and humid period. In contrast, MNP, formed under aerobic conditions, is known for its chemical stability [38], its low mobility [39] in soil, and its low volatility [38] under natural conditions. It degrades poorly in arid environments [38] [40]. This physico-chemical property of MNP could explain its persistence in our sampling area, which is indeed an arid area.

4. Conclusions

A quantitative electrochemical method validated according to single laboratory approach was used for monitoring spatial and temporal distribution of FNT residues in Sahel during an agricultural campaign. The results showed that FNT residues levels were in the range of <6.56 µg/kg to 346 µg/kg in soil and from <6.56 µg/kg to 331.75 µg/kg in sediments.

Results also showed a rapid disappearance of FNT residues in the first two weeks after treatment and suggest that volatilization is the primary mechanism for the disappearance of FNT residues in the Sahel. Analysis of the temporal evolution showed that FNT molecules disappear completely after three months in the studied area. This rapid dissipation of FNT residues, particularly under Sahelian environmental conditions, is considered an advantage of using this pesticide.

Nevertheless, this level of FNT residue content should not be overlooked given the harmful nature of the FNT molecule.

The analytical method used has the advantage of analyzing FNT and significantly reducing the number of chemical sample processing steps and the consumption of organic solvents.

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Conflicts of Interest

The authors declare no conflict of interest.

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