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Level of Pesticide Contamination of Pineapple Fruit in a Region of the Côte d'Ivoire

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Abstract

This study focused on the analysis of 90 pineapple fruit samples, carefully collected during two field campaigns in three areas of the Adiaké department. The aim was to identify and quantify the pesticide residues present in pineapple fruit from these areas. The samples were prepared and analysed using a Shimadzu high-performance liquid chromatography system. The results revealed that the pineapple fruit was contaminated by several pesticides. The calibration curves showed excellent linearity and good reproducibility, with average correlation coefficients ($R^2 \geq 0.996$). Substance recovery ranged from 60 to 115%. Twelve pesticide residues were identified, including glyphosate, aldicarb, profenofos, metazachlor, cypermethrin, deltamethrin, lambda-cyhalothrin, isoproturon, cyanazine, prometryn, monuron and buturon, with concentrations ranging from 0.002 to 0.01 mg/kg. Glyphosate had the highest concentration, at 0.01 mg/kg. However, it should be noted that the levels of pesticide residues present in pineapple fruit from the three localities remain below the standards set by the FAO/WHO. As a result, pineapple fruit from these areas could be considered compliant with international food safety standards.

Keywords

Contamination, Fruit, Pineapple, Pesticide, Standards, Food Safety, FAO/WHO

1. Introduction

The world's population is growing steadily and is expected to increase from 8 to 9.8 billion by 2050 [1]. Faced with this demographic challenge, the search for sus-

tainable solutions to meet the growing food needs of this population is becoming essential. Among the promising crops, pineapple stands out for its richness in vitamins and enzymes, as well as its commercial potential on the international market [2] [3]. Originally from South America, this tropical fruit is grown in many regions, including Côte d'Ivoire, which has long been a leader on the European market in Africa [4]. However, pineapple production in Côte d'Ivoire has fallen significantly, from 238,000 tonnes in 2000 to just 50,000 tonnes in 2022 (PCCET, 2022). To revitalise the sector, the Ivorian government introduced an agricultural policy in June 1993 aimed at modernizing farming practices, leading to increased use of agricultural inputs, particularly pesticides. Unfortunately, the intensive use of these products has harmful consequences for both the environment and human health [2]. Studies have highlighted the existence of inappropriate phytosanitary practices, such as non-compliance with prescribed doses, protection rules and hygiene instructions during treatments. In the short term, these inappropriate practices lead to skin and mucous membrane irritation, headaches and nausea, and in the long term to respiratory problems, reproductive disorders, endocrine diseases, cancer and sometimes death [2] [5]. This is the background to our study, which focuses on pineapple fruit from three localities in the south-east of Côte d'Ivoire, a region of high production. The aim is to identify and quantify the pesticide residues present in pineapple fruit from these areas in order to contribute to food safety.

2. Material and Methods

2.1. Material

Our study matrix consists of 90 samples of pineapple fruit, carefully collected in the surrounding areas of Samo, Assé and Toumanguié. The laboratory equipment used to detect pesticide residues consisted of: an OHAUS electronic balance for weighing the samples with a precision ranging from 1 mg to 0.1 g, a SEVERIN electric mixer for grinding the samples to be analysed, a Com-four stainless steel pineapple cutter, a SHIMADZU HPLC chain equipped with a SIL-20A automatic injector, a CTO-20A column heater, an Interchrom column (250 × 4.6) mm and 5 µm particle diameter. The solvents and reagents used include distilled water, methanol (purity 99.9%), sodium tetraborate (purity 99.5%), FMOC (9-fluorenylmethyl chloroformate) (purity 98%) and dichloromethane (purity 99.8%).

2.2. Methods

We carried out two sampling campaigns at each site (Assé, Toumanguié and Samo) between September 2024 and June 2025, coinciding with the main harvests in the region. Each site comprises three plots varying in size from 1 to 2 hectares. The plots were chosen so as to ensure a balanced distribution between the three sites, in order to take into account a diversity of cultivation practices and soil and climate conditions. From each plot, 10 commercially ripe fruits were taken at random to ensure adequate spatial representativeness. This represents 30 samples per

site, for a total of 90 samples. All the fruit harvested belonged to the Sweet or MD2 cultivar, which accounts for a large proportion of regional production. This choice ensures varietal homogeneity in the analysis, while faithfully reflecting the realities of commercial production. The borders of the sampling sites were dominated by the presence of oil palm plantations, rubber cultivation, market gardening, and cassava. The samples are packed in plastic bags and labeled. The whole set is kept in a cooler and then transported to the National Agricultural Development Support Laboratory (LANADA), where they were temporarily stored at −5˚C. Preparation and analyses were carried out in the days following the sampling.

2.2.1. Molecules Dosage Procedure (Except Glyphosate)

(a). Extraction of Molecules Dosage Procedure

First, 50 g of each sample was taken from a clean, dry jar, after peeling the fruit using a stainless-steel peeler. Next, 30 mL of distilled water was added, and the mixture was homogenized using a SEVERIN electric blender. 100 mL of dichloromethane was added to the crushed material, which was then stirred for 24 hours using an IKA orbital shaker. After this stage, the contents of the jar were centrifuged and filtered through Wattman paper into a round-bottomed flask. The filtrate was then evaporated to a dry residue using a BUCHI R-250 rotary evaporator at 40˚C for 20 minutes. Finally, the residue was recovered by dissolving in 5 mL of methanol.

(b). Purification of Molecules Dosage Procedure

This phase facilitated the recovery of the molecules of interest. A vacuum pump connected to a previously activated C18 column enabled us to filter the recovered residues a second time with methanol in a tube. A mixture of 3 mL methanol and 2 mL distilled water was used to activate the C18 column. The contents of the tube were then transferred to a vial, ready to be injected into the chromatography system for analysis.

(c). Identification and Quantification of Molecules Dosage Procedure

Using a SHIMADZU High Performance Liquid Chromatography (HPLC) system coupled with an Ultra-Violet detector, we extracted pesticide residues. Equipped with a SIL-20A automatic injector, a CTO-20A column heater, an Interchrom column (250 × 4.6) mm and 5 µm particle diameter, the HPLC system has a mobile phase composed of water and acetonitrile in a 75:25 (v/v) ratio. The VP-ODS shimpack reverse phase column forms the stationary phase. The flow rate of the eluent through the column is maintained at 1 mL/min. The volume of sample injected is 20 microlitres. The system's operating period ranges from 0 to 15 minutes. The pump used is a WATERS 600 gradient pump at a pressure of 13 MPa. With a wavelength of 254 nm, the UV detector was used to record the peaks, the different surfaces of which were created using a microprocessor assisted by SHIMADZU software. The identification of a compound is based on its retention time, which is the time required for the compound to emerge from the column. The compounds present in the sample were quantified by comparing the area of the compound peak with that obtained from the standard solution.

2.2.2. Glyphosate Dosing Method

(a). Extraction of Glyphosate Dosing Method

15 g of the pineapple fruit sample grindings were mixed with 100 mL of distilled water. This mixture was then vigorously homogenised using an IKA orbital shaker for 30 minutes. After this stage, the mixture was filtered through a Wattman N˚114 filter paper, placed in a round-bottomed flask. The filtrate was evaporated to dryness in a BUCHI R-250 rotavapor at 40˚C for 15 min. The residues were recovered with 5 mL of distilled water.

(b). Purification of Molecules Dosage Procedure

We took 1 mL of the recovered residue, then added 1 mL of FMOC and 1 mL of sodium tetraborate to a 50 mL Falcon tube. The mixture was then vortexed to ensure complete homogenisation and placed on a shaker for 30 minutes, taking care to protect it from light to avoid degradation. After shaking, we centrifuged the mixture to separate the phases, and recovered the supernatant, which was transferred to vials for identification and quantification by HPLC.

(c). Identification and Quantification of Glyphosate Dosing Method

Pesticide residues were identified and quantified using the same procedure as for the molecules studied previously.

Calibration and Quantification

The selection of pesticides of interest was based on a field campaign conducted among pineapple fruit growers in this region, as well as interviews with sellers of plant protection products for pineapple cultivation. The pesticides selected included four pyrethroids: cypermethrin, permethrin, deltamethrin and lambda-cyhalothrin; ten ureas: fenuron, monuron, isoproturon, diuron, linuron, chlortoluron, monolinuron, methabenzthiazuron, metoxuron and buturon; nine triazines: desisopropylatrazine, desethylatrazine, simazine, cyanazine, atrazine, propazine, terbuthylazine, prometryne and terbutryne; three triazinones: metamitron, hexazinone and metribuzin; six organochlorines (OCPs): metazachlor, aldrin, diazinon, dimethoate, profenofos and ethoprophos; five carbamates: aldicarb, carbosulfan, carbaryl, dithiocarbamate and chlorpropham; and three organophosphates (OPs): parathion-methyl, chlorfenvinphos and parathion-ethyl. For each pesticide of interest, a calibration curve was established using reference standards supplied by Dr. Ehrenstorfer, with a purity greater than or equal to 98%. Five separate concentrations were prepared by successive dilutions in methanol or acetonitrile, depending on the solubility of each compound. Each of the standard solutions was injected into the HPLC chromatographic system, and the areas of the peaks were measured and plotted against the corresponding concentrations. Linear regression was applied to each curve to calculate the correlation coefficient R^2. These curves were used to identify the characteristic retention times of the active substances on the chromatograms of extracts from pineapple samples, injected under the same analytical conditions. The areas of the peaks, for both the standards and the samples, were used to quantify the various active ingredients present in the extracts. In addition, fortified control samples and duplicates were analysed to check the accuracy and

repeatability of the measurements. Concentrations were determined using the following formula:

$$C = \left(s_1 \cdot C_e \cdot v_2 \cdot v_F \cdot F\right) / \left(s_e \cdot M_e \cdot v_1\right) \quad (1)$$

C : concentration of active ingredient (mg/kg)
S_1 : area of sample peak
S_e : area of standard peak
C_e : concentration of standard (mg /L)
V_1 : volume to be purified (L)
V_2 : volume after purification (L)
V_F : final volume (L)
M_e : sample mass (kg)
F : dilution factor

Statistical analysis was carried out to assess pesticide concentrations in the various samples examined. Pesticide levels were compared with regulatory maximum limits (MRLs) to assess the quality of the ripe pineapple fruit samples. Validation of the analytical method involved a performance evaluation, taking into account parameters such as the calibration curve, linearity, limit of detection and limit of quantification, to ensure reliable results. The calibration curves showed excellent linearity and good reproducibility, with average correlation coefficients ($R^2 \geq 0.996$). Recovery of the substances varied from 60 to 115%. The limits of detection (LOD) were 0.002 - 0.004 mg/kg; 0.001 - 0.006 mg/kg; 0.002 - 0.005 mg/kg; 0.003 - 0.006 mg/kg and 0.002 - 0.007 mg/kg respectively for pyrethroids and triazines, ureas; carbamates and triazinones; organophosphates, organochlorines and organophosphates. The limits of quantification (LOQ) were 0.004 - 0.008 mg/kg; 0.002 - 0.007 mg/kg; 0.003 - 0.008 mg/kg; 0.002 - 0.006 mg/kg for pyrethroids, triazines, ureas, carbamates and organophosphates respectively. All the analytical parameters (recovery rate, correlation coefficient, limit of detection and limit of quantification) are shown in Table 1.

Table 1. Analytical parameters (recovery rate, correlation coefficient, limit of detection and limit of quantification).

Molecules of interest	LOD (mg/kg)	LOQ (mg/kg)	R^2	Recovery (%)
Cypermethrin	0.002 - 0.003	0.005 - 0.008	0.997	98
Permethrin	0.002 - 0.003	nd	0.996	110
Deltamethrin	0.002 - 0.004	0.004 - 0.008	0.990	88
Lambda-cyhalothrin	0.003 - 0.004	0.005 - 0.008	0.996	114
Fenuron	0.001 - 0.003	nd	0.998	89
Monuron	0.003 - 0.005	0.003 - 0.008	0.997	88
Isoproturon	0.003 - 0.005	0.004 - 0.008	0.995	110
Diuron	0.003 - 0.005	nd	0.996	114

Continued

Chlortoluron	0.002 - 0.005	nd	0.997	88
Monolinronuron	0.003 - 0.005	nd	0.996	100
Methabenzthiazuron	0.002 - 0.005	nd	0.995	79
Metoxuron	0.003 - 0.005	nd	0.998	94
Buturon	0.003 - 0.005	0.003 - 0.009	0.996	86
Aldicarb	0.001 - 0.003	0.002 - 0.004	0.997	78
Chlorpropham	0.003 - 0.004	nd	0.998	105
Dithiocarbamate	nd	nd	0.995	95
Carbaryl	nd	nd	0.997	105
Carbosulfan	nd	nd	0.998	89
Parathion-methyl	0.003 - 0.007	nd	0.996	101
Chlorfenvinphos	0.002 - 0.007	nd	0.998	95
Profenofos	0.002 - 0.003	0.003 - 0.005	0.997	87
Ethoprophos	0.002 - 0.003	nd	0.997	98
Dimethoate	0.003 - 0.007	nd	0.998	95
Diazinon	0.003 - 0.007	nd	0.996	107
Aldrin	0.003 - 0.007	nd	0.997	95
Metazachlor	0.003 - 0.006	0.004 - 0.007	0.998	89
Metribuzin	0.002 - 0.004	nd	0.996	78
Hexazinone	nd	nd	0.998	95
Desethylatrazine	0.002 - 0.004	nd	0.996	106
Simazine	0.002 - 0.004	nd	0.998	108
Cyanazine	0.002 - 0.004	0.002 - 0.007	0.997	60
Atrazine	0.002 - 0.004	nd	0.997	96
Propazine	0.002 - 0.004	0.002 - 0.004	0.998	65
Desisopropylatratzine	0.002 - 0.004	nd	0.997	85
Linuron	0.002 - 0.005	nd	0.998	115
Prometryn	0.002 - 0.005	0.006 - 0.009	0.996	99
Terbutryn	0.002 - 0.005	nd	0.998	96
Metamitrone	0.002 - 0.005	nd	0.998	106

3. Results and Discussion

Analysis of the pineapple fruit samples revealed the presence of pesticide residues, characterised by specific retention times. These retention times were carefully compared with those of the analytical standards, making it possible to identify the various pesticides present in the samples. The values are shown in Table 2.

Table 2. Pesticide residues detected during the identification phase.

Molecules detected	TR (min)			
	Standards	Asse	Toumanguie	Samo
Glyphosate	4.2	4.3	4.2	4.2
Aldicarb	11.5	12	11.5	11.5
Profenofos	3.2	3.2	3.2	3.2
Metazachlor	3.2	3.2	3.2	3.3
Cypermethrin	2.6	2.6	2.6	2.6
Deltamethrin	4.2	4.1	4.1	4.2
Lambda-cyhalothrin	2.6	2.7	2.6	2.6
Isoproturon	14.5	14	14	14.1
Cyanazine	9	8.9	8.9	8.9
Prometrin	11	11.5	11.5	11.5
Monuron	9.5	9.2	9.2	9.2
Buturon	9.6	9.5	9.5	9.5

3.1. Average Pesticide Residue Levels in Pineapple Fruit from the Different Study Areas

Figure 1 illustrates the average concentrations of pesticide residues detected in pineapple fruit from Assé, Toumanguié and Samo respectively.

Figure 1 shows that the pesticide residues found in pineapple fruit from Assé, Toumanguié and Samo were insecticides and herbicides. The families of pesticides found are in agreement with the results of work by Gouli Bi and Konan [6]. The majority of insecticides belong to the pyrethroid family. Of these molecules, isoproturon (0.008 $mg{\cdot}kg^{-1}$) has the highest concentration, while aldicarb (0.002 $mg{\cdot}kg^{-1}$) and cypermethrin (0.002 $mg{\cdot}kg^{-1}$) have identical lower concentrations in Assé pineapple fruit. In pineapple fruits from Toumanguié, we found identical and the lowest concentrations for profenofos and monuron (0.003 $mg{\cdot}kg^{-1}$). Metazachlor (0.008 $mg{\cdot}kg^{-1}$) had the highest concentration, followed by isoproturon and prometryn at identical concentrations (0.007 $mg{\cdot}kg^{-1}$). Samo pineapple fruit samples, on the other hand, showed a maximum concentration for glyphosate (0.01 $mg{\cdot}kg^{-1}$) followed by metazachlor, isoproturon and prometryn at identical concentrations (0.007 $mg{\cdot}kg^{-1}$). Aldicarb and monuron (0.003 $mg{\cdot}kg^{-1}$) have the lowest levels. The irrational use of pesticides and a lack of awareness among producers could explain the presence of these substances in pineapple fruit [7]. A study carried out in the Mono and Couffo departments of Benin revealed the presence of the same chemical families of pesticides in peppers (Capsicum frutescens), although these substances are present in varying proportions [8] [9]. Pesticides are in great demand around the world because of their effectiveness in eradicating weeds and their affordability [10] [11].

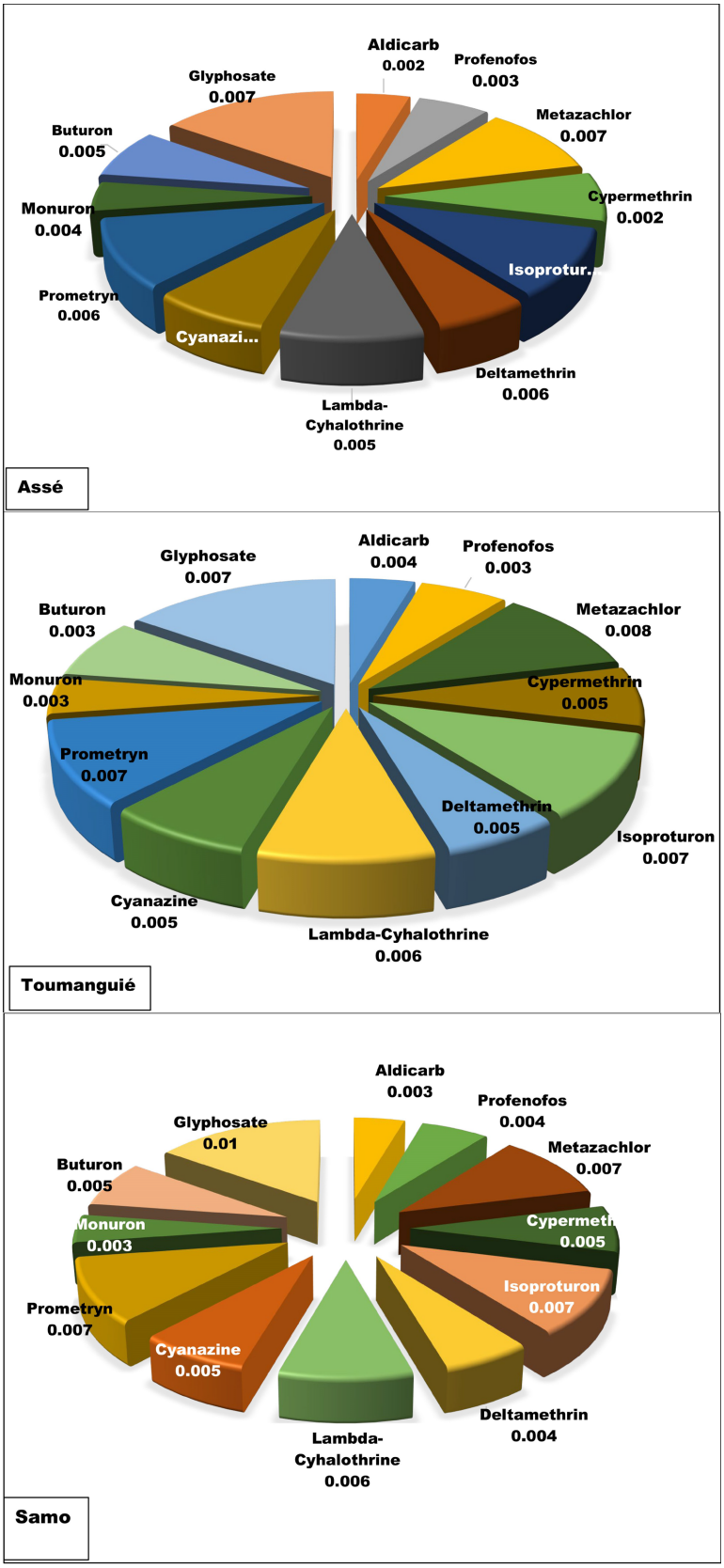

Figure 1. Average concentrations of pesticide residues detected in pineapple fruit from Assé, Toumanguié and Samo respectively.

3.2. Comparison of Pesticide Residue Levels in Pineapple Fruit from Different Study Areas with Codex Alimentarius Standards

Analysis of Table 3 shows that glyphosate (0.01 mg/kg), metazachlor (0.008 mg/kg), isoproturon (0.008 mg/kg) and prometryn (0.008 mg/kg) had the highest concentrations in the samples. Known under the trade name Roundup, glyphosate is a herbicide whose role is to replace ploughing [12]-[14]. The active ingredient in many herbicides, glyphosate is widely used in agriculture around the world thanks to its multiple functions and affordable cost [15] [16]. Metazachlor is the common ISO name for 2-chloro-N-(pyrazol-1-ylmethyl) acet-2',6'-xylidide (IUPAC). It is used for foliar spraying on trees and shrubs against weeds [17]. This would explain its high concentration in the samples. The other substances identified showed lower concentrations. These residue levels could indicate that farmers have received adequate training in cultivation techniques [18] [19].

Table 3. compares the average concentrations of pesticide residues in pineapple fruit from Assé, Toumanguié and Samo with Codex Alimentarius standards.

Molecules detected	Average content (mg/kg)				FAO/OMS	
	Asse (mg/kg)	Toumanguie (mg/kg)	Samo (mg/kg)	LMR (mg/kg)	Organisations	Years
Glyphosate	0.007	0.007	0.01	0.05	Codex Alimentarius	2016
Aldicarb	0.002	0.004	0.003	0.07	Codex Alimentarius	2011
Profenofos	0.003	0.003	0.004	0.07	Codex Alimentarius	2016
Metazachlor	0.007	0.008	0.007	0.02	European Union	2016
Cypermethrin	0.002	0.005	0.005	0.7	Codex Alimentarius	2009
Deltamethrin	0.006	0.005	0.004	0.03	Codex Alimentarius	2016
Lambda-Cyhalothrine	0.005	0.006	0.006	0.03	Codex Alimentarius	2016
Isoproturon	0.008	0.007	0.007	0.01	European Union	2019
Cyanazine	0.005	0.004	0.002	0.05	Codex Alimentarius	2016
Prometryn	0.008	0.008	0.007	0.1	Codex Alimentarius	2016
Monuron	0.004	0.005	0.003	0.05	European Union	2016
Buturon	0.005	ND	0.003	0.05	European Union	2016

ND = no found.

3.3. Comparison of Residue Levels by Production Site

Table 4 shows significantly higher mean values in Toumanguié and Assé, where mean concentrations are slightly comparable. The Samo area, on the other hand, has relatively low average concentrations. The differences observed can be attributed to distinct cultivation practices and varying levels of phytosanitary training among growers. The variability, as shown by the standard deviations, remains moderate, indicating a degree of homogeneity in practices within each locality.

Table 4. Shows the minimum (Min), maximum (Max) and average (Avg) concentrations, as well as the standard deviations (SD) for the main pesticides detected in the three zones.

Molecules	Asse (mg/kg)				Toumanguie (mg/kg)				Samo (mg/kg)			
	Min	Max	Moy	ET	Min	Max	Moy	ET	Min	Max	Moy	ET
Glyphosate	0.002	0.007	0.004	0,0025	0.005	0.009	0.007	0.0020	0.009	0.014	0.012	0.0025
Aldicarbe	0.002	0.005	0.003	0.0015	0.003	0.006	0.004	0.0015	0.002	0.004	0.003	0.0010
Profénofos	0.003	0.006	0.004	0.0015	0.002	0.007	0.004	0.0025	0.003	0.005	0.004	0.0010
Metazachlor	0.007	0.008	0.007	0.0005	0.007	0.009	0.008	0.0010	0.006	0.008	0.007	0.0010
Cypermethrin	0.006	0.009	0.007	0.0015	0.004	0.007	0.005	0.0015	0.003	0.008	0.005	0. 0025
Deltaméthrine	0.005	0.007	0.006	0.0010	0.005	0.006	0.005	0.0005	0.003	0.006	0.004	0.0015
Lambda-cyhalothrine	0.005	0.006	0.005	0.0005	0.005	0.007	0.006	0.0010	0.005	0.007	0.006	0.0010
Isoproturon	0.007	0.008	0.007	0.0005	0.005	0.009	0.007	0.0020	0.006	0.008	0.007	0.0010
Cyanazine	0.004	0.006	0.005	0.0010	0.003	0.005	0.004	0.0010	0.002	0.003	0.002	0.0005
Prometryn	0.006	0.009	0.007	0.0015	0.007	0.009	0.008	0.0010	0.006	0.008	0.007	0.0010
Monuron	0.002	0.006	0.004	0.0020	0.003	0.006	0.004	0.0015	0.002	0.005	0.003	0.0015
Buturon	0.004	0.006	0.005	0.0010	0	0	0	0	0.002	0.004	0.003	0.0010

4. Conclusion

Chromatographic analysis identified and quantified the presence of twelve pesticide residues in pineapple fruit from three localities (Assé, Samo and Toumanguié), including glyphosate, aldicarb, profenofos, metazachlor, cypermethrin, deltamethrin, lamb-da-cyhalothrin, isoproturon, cyanazine, prometryn, monuron and buturon. Of these substances, glyphosate has the highest level, reaching 0.01 mg/kg. In addition, meta-zachlor, isoproturon and prometryn had identical levels of 0.008 mg/kg. It should be noted that the concentrations detected in the samples analysed remain below the maximum residue limits established by the FAO/WHO. The persistence and accumulation of certain pesticide residues may, in the long term, constitute a danger for both human health and the environment, if the use of these substances continues to intensify. Hence the need for increased monitoring and good, sustainable farming practices.

Consent for Publication

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Conflicts of Interest

The authors declare no conflicts of interest.

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Abbreviations

IUPAC	International Union of Pure and Applied Chemistry
FAO	Food and Agriculture Organization
FMOC	9-fluorenylmethoxycarbonyl
HPLC	High Performance Liquid Chromatography
MRL	Maximum Residue Limit
TR	Retention Time
WHO	World Health Organization
PCCET	Competitive value Chains for employment and Economic Transformation Project