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Determination of Pesticides in Marine Sediments from Dakar Coast (Senegal) by Gas Chromatography-Tandem Mass Spectrometry (GC/MSMS)

Dame Cissé, Birame Ndiaye*, Cheikh Tidiane Dione, Ibrahima Diagne, Maoudo Hane, Sitor Diouf, Mame Mor Dione, Seydou Ba, Ousmane Ka, Mamadou Sarr and Momar Ndiaye

Faculty of Science and Technology, Laboratory of Physical-Organic Chemistry andEnvironmental Analysis (LCPOAE)-Cheikh Anta Diop University/Dakar/Senegal

**Corresponding author*

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A B S T R A C T

Many aquatic products, especially coastal species consumed, are often in contact with sediments. Wastewater discharge channels often drain sediments that trap micropollutants. Sediments taken downstream of the Hann, Ngor and Soumbedioune drainage canals of the Dakar coast during the rainy season. These sediments are analysed by Gas Chromatography-Tandem Mass Spectrometry (GC/MSMS) finally to determine their degree of pesticide contamination. After separating the organic and water phases, we used QuECHERS and dSPE salts for extraction and purification. The analytes are separated by a temperature gradient of 55-330°C for 40 minutes. The results show that in these samples, 9 pesticides are present at levels higher than their respective quantitation limits. Atrazine, dichlorvos and terbutryne are more common in sediments. Their highest levels are 144.35, 284.462 and 509.998 µg, Kg^{-1}, respectively. Contamination is greater at Hann and triazines pesticides are more present in different sediments and this distribution is generally asymmetrical with p-values below the 5% threshold.

Introduction

The need for the best agricultural yields leads to the use of phytosanitary products including pesticides (Racha El-Osmani, 2014). However, behind these benefits, pesticides have negative impacts on the environment and the health of living things. These pesticides, classified as persistent organic pollutants (POPs) since the Stockholm Convention in 2011, have carcinogenic, neurological and reproductive effects (Luc Multigner, 2005). In the past, pesticides have caused 500 cases of poisoning in Senegal (Pesticide Action Network, 2000) (Cissé, 2000). The wastewaters and strormwaters of the Han, Ngor and Soumbedioune canals transported suspended matter which, by obstruction or downstream of the discharge, sediments and can accumulate several micropollutants

such as pesticides (Jean-Luc Loizeau *et al.,* 2016). These pesticides of various uses and anthropogenic origins can be found in the marine environment and contaminate aquatic organisms such as mussels, fish and many seafood. Through the food chain, pesticide levels can biomagnify and human health is threatened (Aubertot *et al.,* 2005; Agbohessi *et al.,* 2012). The purpose of this study is to determine the levels of pesticides in the surface sediments from the discharges of wastewater discharged on the bays of Hann, Soumbedioune and Ngor de Dakar finally to assess the contamination.

Materials and Methods

Sampling Points

Sediment samples are collected in Ngor, Hann and Soumbedioune during the rainy season between June and September 2018 (Figure 1). These selected sites face the Atlantic and there is a lot of activity there. They provide a signifycant source of fish products. Hann Bay (SD1), which covers 13 km, is home to fishing activities and several industries including textile factories, fish processing plants. The bay receives sewage from the East Canal. The beach of Soumbedioune (SD2) is a place of landing of fish products and receives the wastewater coming from the West channel in open air that crosses a good part of the communes of Dakar. Ngor (SD3), tourist site is also crossed by the canal which is open and almost transformed into a garbage dump.

Sample Preparation and Processing

Surface sediment samples are collected between June and September 2018 using a hand-held bucket at depths between 0 and 5 cm (UNEP, 2007). In the laboratory, after extracting the coarse materials, the samples are dried in the oven at 40°C until dry weight. The fine fraction with a diameter of less than 63 µm is used for micropollutants analysis (Ongley, 1996; Markus Zennegg, 2016). For the analysis of pesticides, acetonitrile (ACN) is used for the separation of the organic and aqueous phase. After, the extraction of organic pollutants is made possible thanks to the extraction salt is the QuECHERS composed of4g MgSO4; 1g NaCl; 1g trisodium de citrate dihydrate; 0,5 g disodium hydrogen-citrate sesquihydrate. This step is followed by purification with the help of salt purification with dSPE RESTEK salt consisting of1,2 g MgSO4+400mg PSA+400mg C18.Thus, the extracted solution is acidified and then extracted using a SPME PA fiber 85 µm at 60°C.

Method of Analysis

The analysis of pesticides in sediments is carried out with gas chromatography coupled with a Mass Spectrometer in tandem mode (GC/MSMS). The separation of the compounds was carried out on a column *Optima XLBde 30 m × 0,25* mm internal diameter and a film thickness of 0.25 µm. The stationary phase consists of 5% Phényl/95% Diméthyl-polysiloxane. In the oven, a temperature gradient of 50 to 330°C with different slopes for 40 minutes is designed for the separation of analytes. At the end of the column, the compounds are fragmented into a temperature-heated 70eV electronic impact source. The determination of pesticides is done in scan mode then in MRM (Multiple Reaction Monitoring) mode. The scan mode (Figure 2) allows you to select the retention time and mass spectrum of each compound. Thus, we have the «fingerprint» of all compounds. MRM mode is used to perform quantitative analysis of trace elements already identified in scan mode. The retention times of the different molecules as well as the specific ions used for their quantification and identification (Table 1). The recovery rates of pesticides obtained range from 68.21 to 98.09%. The %RSD are low and remain below 20%. In GC/MSMS, the recovery rates obtained are generally in the 70-120% range.

Results and Discussion

Variation of pesticide levels in sediment

By integral of the obtained peaks, the levels of pesticides of the sediments in the various sites group in the following table.

Atrazine, dichlorvos and terbutryne have the highest levels in the sediments of the different sites. The mean terbutryne levels in Hann, Ngor and Soumbedioune are 509.998, 98.576 and 60.201 µg, Kg^{-1}respectively. In Hann, pesticide contamination of sediments is dominated by terbutryne, which is 77.43% of the total content (Table 2). Similarly, dichlorvos, a highly hazardous organophosphate pesticide (WHO class 1b) has high concentrations with an average of 65.493 in Hann, 89.934 in Soumbedioune and 284.462 $\mu g.Kg^{-1}$ (Mahé *et al.,* 2020). The high atrazine concentrations (147.35 $\mu g.Kg^{-1}$ at Soumbedioune) may be related by its broad

spectrum of use. It can also be enhanced by its persistence in soil (140-150 days) and water (180 days). Atrazine with proven acute toxicity kills invertebrate species at 0.2-7 mg.L^{-1} and fish at 5-15 mg.L^{-1} (Mahé *et al.,* 2020; Miquel, 2003). Acetochlor has relatively low levels. At Ngor, the variations in acetochlor are (0.555-0.690) µg. Kg^{-1} and Soumbedioune and Hann these variations are respectively (0.627-0.800) and (0.445-1.02) µg. Kg^{-1}. WHO Class II alachlor dicofol levels are also low at all three sites (Classification OMS recommandée, 2019). These DDT levels (0.0917, 0.1409 and 0.086 µg. Kg^{-1}) are very low compared to the levels found in the sediments of the Ouémé River of Benin (134-189 µg. Kg^{-1}) and in the sediments of the Magou River (489.5 µg. Kg^{-1}). Levels of DDE found during this campaign are also much lower than levels found on the Ouémé River (68-131 µg. Kg^{-1}) (Tachegnon Prudencio Agbohessi and Ibrahim Imorou Toko, 2012).

Study of global contamination

Assessment of the overall pesticide content shows that sediment contamination was higher at Ngor in June (1052.571 µg.Kg^{-1}). In Hann, sediment collected in July is more loaded with 1470.739 µg.Kg-1. For Soumbedioune, sediments from September are more contaminated 465.257 µg.Kg^{-1} and there is a slight variation in overall concentrations between July (341.947 µg.Kg^{-1}) and August (382.069 µg.Kg^{-1}) (Figure 3). As in Dakar, other pesticides such as Chlorpyriphos, Imazalil, Ethion and Diazinon are also present in sediments of the Guadalquivir River Basin (Spain) and are noted with overall concentrations of 13.6 ng.g^{-1} in 2010 and 10.9 ng.g^{-1} in 2011 (Ana Masiá *et al.,* 2013). Given all these levels of pesticides in the various sediments and all the factors that influence their presence in the aquatic environment, the coastal marine environment can be very likely the receptacle (Nathalie Coquille, 2017). The contamination rates of the three triazines in Hann, Soumbedioune and Ngor sediments are 87.41, 64.40 and 38.73% respectively. These triazines, particularly atrazine, have been widely used in combination with about 20 other herbicides since 2001. With these levels in addition to their water solubility, soil adsorption coefficient, vapour pressure and field half-life, these triazines will have a high potential for water contamination (Fan, 2014). Moreover, these levels are worrying and in addition to being persistent in all environmental compartments triazines are classified as possible carcinogens for humans (Karasali and Maragou, 2016). The levels of the other chemical families (organophosphate, organophosphate, pyrethroid, etc.) also have significant levels in the sediments with levels of 11.75, 33.52 and 59.86%, respectively in Hann, Soumbedioune and Ngor (Figure 4). These pesticides, particularly organophosphates, need to be monitored because their acute poisoning is responsible for high mortality in developing countries with high agricultural potential (Thabet *et al.,* 2009). Levels of OCPs are lower among chemical families found in sediments.

Statistical Study: Multivariate Analysis of Variance (MANOVAS)

Multivariate analysis of variance by SPSS is used for an overview of contaminant profiles, pesticide distributions or correlations in the sediment studied.

The Shapiro-Wilk normality test shows that our analyzed samples come from sediments that have normal distributions for acetochlor and dicofol. The levels of these two pesticides are above the 5% threshold and are respectively 0.198 and 0.342. So the assumption of a normal distribution is true for these two elements. Tests by Shapiro-Wilk and Kolmogorov-Smirnov (for a large sample) show that contamination of different sediments by other pesticides with p-values between 0.000 and 0.029 less than 5% follow an asymmetric distribution (Table 3). With this distribution it may be thought that the origin of the contamination of the sediments by acetochlor and dicofol can be diffuse and continuous pollution. The results of the Levene equality test show that with the exception of atrazine, the p-values of pesticides are below the 5% alpha threshold. So for these pesticides (alachlor, acetochlor, etc.), we reject the hypothesis of equality of variance for the sediments of the different sites. For atrazine, the variance is the same for sediments from its sites (Table 4).

At the end of the analyses, plant protection pollution is observed, clearly dominated by terbutryne, dichlorvos and atrazine. Terbutryne ranges from (166.9-1394.2) to 509.998 µg.kg^{-1} average (Hann). Levels of dichlorvos range from 1.689 to 601.01 µg.Kg^{-1} and the highest mean is 284.462 µg.Kg^{-1} (Ngor). Atrazine, the third most common pesticide, has levels of 318.40 µg.kg^{-1} (Soumbedioune) with an average of 147.35 µg.Kg^{-1}. These three pollutants account for more than 90% of the contamination (96.36% in Hann, 90.16% in Soumbedioune and 94.29% in Ngor). This pollution is also estimated in total pesticide contamination of up to 1470,739 µg/Kg in July in Hann.

Table.1 Characteristics of pesticides

Nature	Compounds	Parent ion	Son ions	tr (min)	LQ
Pesticide	alachlore	160	132-130-117-145	15,46	0,05625
	acétochlore	146	131-130-118-91	15,17	0,0624
	atrazine	215	200-173-215-138	13,01	0,06501
	dicofol	139	75-111	17,62	0,04629
	deltaméthrine	181	151-152	31,74	0,01386
	dichlorvos	109	79-109	11,22	0,23775
	irgarol	182	109-139-125-182	18,2	0,03522
	malathion	127	92-89-101-109-77	16,37	0,01278
	terbutryne	185	170-185	16,07	0,05625
Internal Stallion	Diphénylamine-d6	175	144-172	11,46	-
	trifuraline-d14	267	209-163	11,5	-
	pendimethaline-d5	255	164-143	17,68	-

Table.2 Pesticide levels (µg.Kg^{-1}) in sediment by dry weight.

Pesticides	Hann (SD1)		Soumbedioune (SD2)		Ngor (SD3)	
	Min-Max	Moy ± DS	Min-Max	Moy ± DS	Min-Max	Moy ± DS
alachlore	0,585-3,317	1,285±1,355	0,770-0,918	0,834± 0,062	0,664-0,882	0,764±0,102
atrazine	19,465-116,12	59,229±43,07	35,010-318,40	147,35± 129,92	13,227-230,19	91,926±101,397
acétochlore	0,445-1,02	0,621±0,268	0,627-0,800	0,697± 0,074	0,555-0,690	0,613±0,058
Dicofol	1,001-1,13	1,052±0,055	1,353-1,637	1,473± 0,119	1,160-1,528	1,346±0 ,176
deltraméthrine	1,179-15,008	4,677±6,887	1,489-1,849	1,635± 0,158	1,317-1,737	1,534±0,210
dichlorvos	24,113-139,65	65,493±51,31	18,844-124,95	89,934±48,490	1,689-601,01	284,462±300,923
irgarol	7,486-12,111	9,348±2,225	1,354-10,504	7,946± 4,408	1,245-12,308	6,572±5,887
malathion	4,034-11,62	6,965±3,287	4,470-32,478	19,897±11,571	2,455-34,55	17,975±16,64
terbutryne	166,9-1394,2	510,0±590,64	13,548-128,15	60,201±50,338	7,775-305,93	98,576±141,292

Table.3 Normality Test

Pesticides	Kolmogorov-Smirnov			Shapiro-Wilk		
	Statistique	ddl	Signification	Statistique	ddl	Signification
alachlore	0,439	12	0,000	0,466	12	0,000
atrazine	0,211	12	0,146	0,842	12	0,029
acétochlore	0,214	12	0,134	0,908	12	0,198
dicofol	0,171	12	0,200	0,926	12	0,342
deltraméthrine	0,495	12	0,000	0,378	12	0,000
dichlorvos	0,348	12	0,000	0,697	12	0,001
irgarol	0,243	12	0,048	0,817	12	0,015
malathion	0,250	12	0,037	0,840	12	0,028
terbutryne	0,331	12	0,001	0,567	12	0,000

Table.4 Levene Variant Equality Test

	alachlore	atrazine	acétochlore	dicofol	deltraméthrine	dichlorvos	irgarol	malathion	terbutryne
Levene index	7,875	2,191	4,291	5,007	8,482	44,303	5,454	6,924	5,981
ddl1	2	2	2	2	2	2	2	2	2
ddl2	9	9	9	9	9	9	9	9	9
Sig.	0,011	0,168	0,049	0,035	0,008	0	0,028	0,015	0,022

Figure.1 Sampling Points

Figure.2 Chromatogram of a standard solution in scan mode.

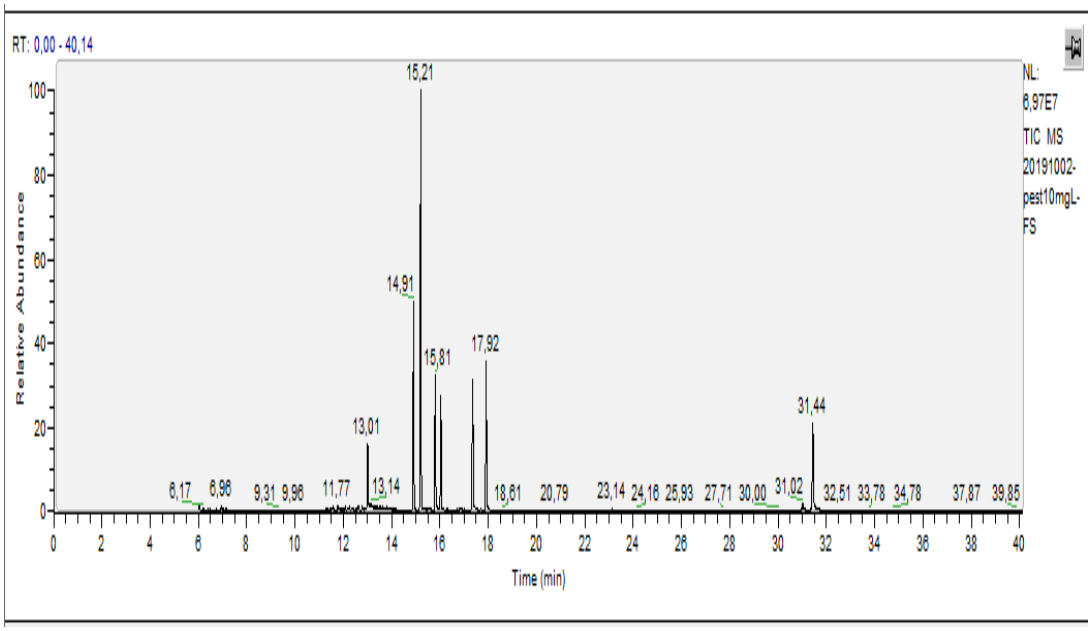


Figure.3 Total levels of pesticides

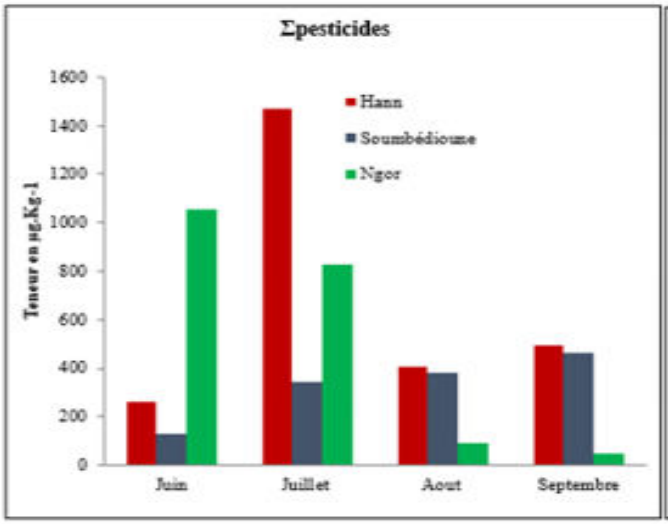
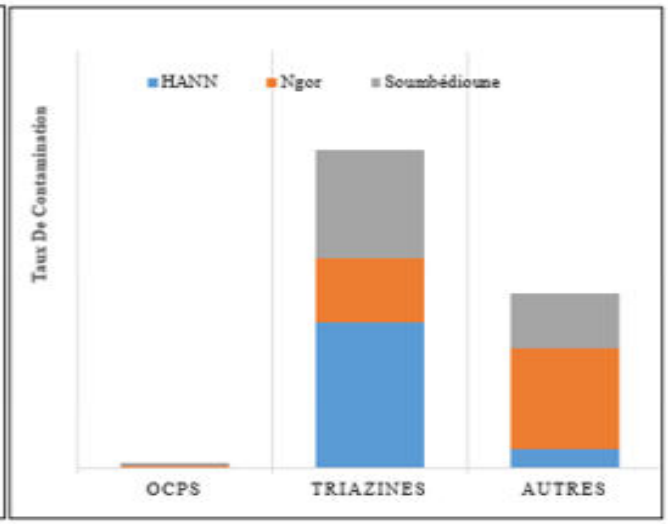


Figure.4 Quantification of pesticides by family



During this season, herbicide pesticides are dominant in the surface sediments of Hann (86.71%) and Soumbedioune (63.36%). Through the normality test, with the exception of acetochlor and dicofol, sediment contamination by pesticides follows an asymmetrical distribution. Regular channel cleaning can reduce or stop a chain of contamination of the marine environment via sediments.

Author Contributions

Dame Cisse: Investigation, formal analysis, writing—original draft. Birame Ndiaye: Validation, methodology, writing—reviewing. Cheikh Tidiane Dione:—Formal analysis, writing—review and editing. Ibrahima Diagne: Investigation, writing—reviewing. Maoudo Hane: Resources, investigation writing—reviewing. Sitor Diouf: Validation, formal analysis, writing—reviewing. Mame Mor Dione: Conceptualization, methodology, data curation, supervision, writing—reviewing the final version of the manuscript. Seydou Ba: Investigation, formal analysis, writing—original draft. Ousmane Ka: Validation, methodology, writing—reviewing. Mamadou Sarr:—Formal analysis, writing—review and editing. Momar Ndiaye: Investigation, writing—reviewing

Data Availability

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethical Approval Not applicable.

Consent to Participate Not applicable.

Consent to Publish Not applicable.

Conflict of Interest The authors declare no competing interests.

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