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RESEARCH ARTICLE

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Assessment of surface and groundwater contamination by pesticides residues in a Sudano-Sahelian agricultural watershed (Korokoro, Mali)

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ABSTRACT

Surface and groundwater contamination by pesticides (organochlorine, organophosphates and pyrethroids) was studied in the Korokoro watershed (60.6 km², Mali) where cotton and cereals (sorghum, maize etc.) are the main crops. Nine and eleven investigative domestic wells were monitored and sampled respectively in 2010 and 2011 at the end of each cropping season. At lysimeters scale, surface and groundwater contamination processes by pesticides in the watershed have been experimented also in 2010 and 2011 during each rainy season. Cotton was sown in each lysimeter box and after growing all plants have been treated with the pesticide endosulfan which still used in cotton fields by watershed farmers although it has been banned worldwide. After each rainfall event in 2010 and 2011, runoff and infiltration samples were sampled in each lysimeter. All water samples collected in 2010 and 2011 from lysimeters and Korokoro watershed were analyzed by gas chromatography to quantify respectively endosulfan residues and the pesticides (profenofos, atrazine and cypermethrin) frequently used in cotton fields by the farmers. The results showed that all domestic wells water was mainly contaminated by organochlorine pesticides (dieldrin, 5.8 µg.L⁻¹ and endosulfan, 1.8 µg.L⁻¹). According to the experiments carried out in lysimeters, this contamination was mostly explained by runoff events in 2010 than 2011 ones (6.5 ± 2.9% against 0.1 ± 0.09% of exported matters) even if infiltration values in 2010 were less than those of 2011 (0.1 ± 0.09% against 0.2 ± 0.04% of exported matters). Other factors such as aeolian transport can also contribute to this contamination.

Keywords: domestic wells water, Korokoro watershed, lysimeter boxes, surface and groundwater contamination, pesticides residues.

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I. INTRODUCTION

In West Africa, the economy of most countries particularly that of Mali, is based on agriculture mainly cotton, which nowadays provides more than 65% of export earnings [1]. The development of cotton is linked to several factors including the intensive use of chemical inputs such as pesticides. In Mali, these chemicals products are frequently used in cotton areas of the "Compagnie Malienne du Développement des Textiles (CMDT)" and the "Office de la Haute Vallée du Niger (OHVN)". In these areas, 80% of pesticides used are insecticides (organochlorines, organophosphorus, etc.) [2]. However, in a context where the use of some of these chemical products is not controlled and/or banned for agricultural use (e.g.

Organochlorine pesticides) and also due to their toxicity, bioaccumulation potential and endocrine actions [3-8], their intensive use is not without consequences on farmers health and the environment [9]. Indeed, studies have revealed the contamination of soils, sediments, surface and groundwater by pesticide residues in West African countries such as Burkina Faso [10], Senegal [11], Côte d'Ivoire [12] and Mali [13]. The contamination of these environmental compartments is generally linked to the agricultural land use [14;15]. Indeed, contamination of surface and groundwater is respectively the consequence of soil one during phytosanitary treatments and runoff process from the treated areas and also during infiltration process of pesticides residues through soil profile [16;17]. In fact, field plowing promotes pesticide residues which

are both adsorbed on fine soil particles and accumulated in the surface horizon (0-20cm) to rise to soil surface or not and during significant rainfall events these pesticide residues are transported by runoff and/or infiltration processes. Thus, through these runoff and/or infiltration processes, surface and groundwater can be contaminated by the agricultural use of pesticides. However, this contamination will depend on both the physico-chemical properties of pesticides family used, soil conditions (moisture content, microorganism levels, etc.) and also the climatic conditions of the environment concerned (temperature, dry or rainy season moment etc.). However, even if the agricultural use of pesticides is done under regulatory conditions, the risk of surface and groundwater contamination through runoff and/or infiltration processes is still a greater factor. Indeed, the U.S. Environmental Protection Agency (EPA) has previously reported that normal agricultural use has led to the presence of at least 46 pesticides residues in groundwater and 76 in surface waterbodies, the most frequently detected were atrazine (herbicide) and endosulfan. (organochlorine pesticide) [18;19]. Generally, water resources particularly groundwater ones are largely used for human consumption but nowadays they are frequently contaminated by pesticides residues due to their potential persistence and active substances transport through soil profile [17]. Indeed, various pesticides residues were detected in groundwater during the last 30 years in Europe [20-22], United States [23-25] and Africa [26;12]. The occurrence of pesticides residues in water resources is in general linked to their potential accumulation in environmental matrices. In addition, it has been reported that many pesticides (e.g. organochlorine pesticides) can persist for long periods in an ecosystem and continuous to be detected in surface waters 20 years after their use had been banned [27]. Therefore, it is necessary to know the fate of pesticides residues brought into surface waters (river, lake etc.) and/or groundwater by runoff and/or infiltration processes. Unfortunately, this aspect is not sufficiently developed in Sahelian countries particularly in the context of Mali because studies of water resources contamination are very limited [28]. Hence, the need for more scientific research in order to contribute for appropriate decision-making. This research project is enrolled in this framework and aims to improve knowledge of surface and groundwater contamination processes by pesticides used in cotton cropping areas at Korokoro watershed scale (Mali).

II. MATERIAL AND METHOD

II.1 Study area

Korokoro Watershed (60.6 km²) is located in Koulikoro region (Mali) between 12°42'N and 12°50'N, 7°22'W and 7°28'W. It is also located on the right bank of the Niger River and situate at 70 km from the capitol of Mali (Bamako) (**Fig.1**). It is occupied by 13 agricultural organizations among which 4 (Kodalabougou, Chonikoro, Sido and Fiéna) were followed from 2009 to 2011 for their greater involvement (>20 years) in pesticides use in the cotton production. The climate of this watershed is characterized by a long dry season (November to May) and short rainy season (June to October). Annual rainfall is variable according years (from 700 to 800 mm). Temperatures undergo little fluctuation however they are high in March and May (36°C to 40°C) and low in December to February (below 20°C). The soil compartment is dominated by three mainly soils, (i) Entisol (78.3%, of the watershed surface), (ii) Alfisol (18.7%) and (iii) Inceptisol (0.1%). Cotton fields that are subjected in this study are mainly located on Alfisol. Crops (cotton, sorghum, maize, millet etc.) occupy 25% of the watershed area [29]. Among these, cotton is mainly produced for the global market and requires always intensive use of chemical inputs such as insecticides (organochlorines, organophosphates and pyrethroids). These chemical products are used by farmers in order to control insect pests (i.e. *Helicoverpa armigera* and *Aphis gossypii*) and also obtaining better agricultural yields.

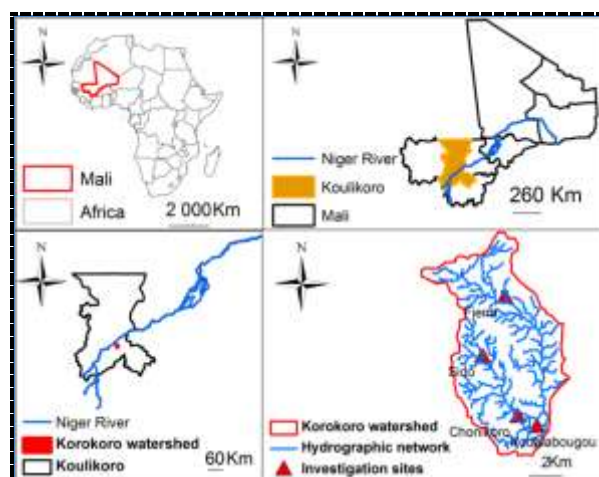


Fig.1: Location of Korokoro watershed and the four investigative sites

II.2 Pesticides used in the agricultural Korokoro watershed

In order to assess surface and groundwater contamination by pesticides residues of Korokoro watershed, it has been necessary to identify pesticides really used in cotton production at local scale. Thus, surveys have been conducted from 2009

to 2011 in 4 main villages or hamlets (Kodalabougou, Chonikoro, Fiena and Sido) where the cotton is produced for several decades. Generally, the surveys were conducted in investigative sites using questionnaires addressed either individually or to groups as appropriate in order to obtain significant details about pesticide formulations, doses, frequency of phytosanitary treatment, period of application etc. These surveys were later supplemented by informations from the "Compagnie Malienne de Développement de Textiles (CMDT)" and also from "Office de la Haute Vallée du Niger (OHVN)".

II.3 Experiments of surface and groundwater contamination by pesticides residues of Korokoro watershed in lysimeter boxes

Three lysimeter boxes (3m x 1m x 1m) were installed (June 16, 2009) on University campus of Bamako in order to study surface and groundwater contamination by pesticides residues in Korokoro watershed (Mali). The pedological material of the lysimeters is from an alfisol of the watershed where cotton is cultivated since many decades. Each box has two aluminum containers for respectively runoff and infiltration water collection. This experimental apparatus was completed with a rain gauge. In order to verify the initial soils contamination by other pesticides residues, two tests were performed on each lysimeter box before experimentations. The tests were based on rain simulation for two events in each lysimeter box respectively on July 4 and 7, 2009. After, only runoff water samples were collected and analyzed with a gas chromatograph. The results have not shown any contamination. Thus, during each raining season, the cotton has been grown in lysimeters in 2010 and 2011 and treated with pesticide solution. All the phytosanitary treatments have been performed with the pesticide endosulfan although its agricultural use has been banned worldwide. However, it has been chosen in this study for contamination tests because it still used by some Malian farmers as Korokoro watershed ones in cotton production [30]. Cotton seeds were so sown in each lysimeter box respectively on July 10, 2010 and on August 6, 2011. After growing, cotton plants have been treated with respectively two endosulfan applications (500 mg of applied matters) and one application (1,000 mg of applied matters). However, all agriculture practices (phytosanitary treatment, fertilizers application etc.) were referred to official recommendations in Malian cotton production area.

II.4 Protocol of water sampling in lysimeters and Korokoro watershed

In lysimeter boxes, surface and groundwater were sampled after each rainy season (from July to August) in 2010 and 2011 in order to assess their

contamination level by endosulfan residues. Water samples were collected always in amber glass bottles after each rain event which had caused runoff and/or infiltration. Rainfall amount was also measured as runoff and infiltration volumes after each event. The measurements have started on August 28, 2010 and August 24, 2011 after endosulfan application on cotton plants in each lysimeter box during 8 and 5 rainfall events respectively in 2010 and 2011. However, during the sampling and laboratory storage, some water samples were lost.

At Korokoro watershed scale, watercourses near the cotton fields are not generally permanent so the sampling was mainly based on groundwater i.e. domestic wells water in 2010 and 2011. Water samples were collected to study their contamination level by pesticides which were used frequently by farmers in cotton production (endosulfan, cypermethrin, atrazine and profenofos). The samples were taken in January 13, 2010 and September 20, 2011 respectively in 9 and 11 domestic wells according to their distribution in the watershed. These domestic wells supply "drinking water" to local inhabitant and are generally situated in the downstream areas of cotton fields. Amber glass bottles previously decontaminated were used to collect water samples. All bottles were transported in a cooler with cooling pads [12]. All sampling investigative sites were georeferenced with a Garmin GPS 72 (Fig. 2). In laboratory and in order to ensure the stability of researched pesticides residues, water samples were stored in a refrigerator at 4°C before their analysis.



Fig.2: Investigative sites and domestic wells water distribution in Korokoro watershed (Mali)

II.5 Analysis of pesticides residues in water samples (lysimeters and Korokoro watershed)

Water samples of lysimeters and Korokoro watershed ones were subjected respectively to endosulfan residues and the pesticides which were

following in the watershed during the cotton production (endosulfan, cypermethrin, atrazine and profenofos) in 2010 and 2011.

II.5.1 Extraction and purification of active ingredient

The technic of three liquid-liquid extractions were performed on each 500mL water sample from domestic wells, runoff and infiltration of lysimeters. During each extraction, 10% of hexane has been used as extraction solvent [31]. Anhydrous sodium sulphate (Na_2SO_4) was also added in each sample before filtered on filter paper. Then, all filtrates were concentrated on a rotary evaporator (Büchi Rotavapor) to 1 mL and purified on nonpolar cartridges which were previously conditioned with 5mL of hexane/diethyl ether (40:60, v/v) and 5 mL of hexane followed by sample deposit. Elution in each cartridge was carried out with 5 mL of hexane/diethyl ether in proportions respectively of 80:20 and 40:60 (v/v) then all extracts were concentrated again to 0.5 mL and completed to 1 mL with hexane then transferred into vial and analyzed with gas chromatographs.

II.5.2 Chromatographic analysis

Organochlorine pesticides (endosulfan, DDT etc.) and atrazine were analyzed with a gas chromatograph equipped with a micro electron capture detector (GC- μ ECD) but profenofos and cypermethrin were analyzed with a gas chromatograph with a nitrogen phosphorus detector (GC-NPD). Capillaries columns which were used for analyses are HP-5 (length 30 m, 0.320 mm in diameter and 0.25 μm of film thickness) and DB-1701 for confirmation. Carrier gas (nitrogen) was high purity (99.8%). The injection was performed in splitless mode and the injection volume was set at 1 μL . Temperature program was established as follows, oven initial temperature was set at 80°C for 2 min, 80°C to 150°C to 25°C mn^{-1} , 150°C to 200°C to 3°C mn^{-1} , 200°C to 280°C to 8°C mn^{-1} and at 280°C for 10 minutes. Temperatures of injector and detector were respectively set at 250°C and 300°C. All analyzed pesticides were identified and quantified by external calibration method. Linearity ($r^2 > 0.996$) was established for each compound with five points of standards solutions (from 0.0125 $\mu\text{g.mL}^{-1}$ to 0.125 $\mu\text{g.mL}^{-1}$). Detection and quantification limits (LOD, LOQ) were calculated according to the standard deviation, slope, dilution factor and the test volume for each pesticide standard.

II.6 Quantification of endosulfan residues in runoff and infiltration at lysimeters scale

In this study, experiments carried out in lysimeters aimed highlighting the contamination risk of surface and groundwater of Korokro watershed by

the use of endosulfan in cotton production. During the experiments in 2010 (September, from 1 to 19, 2010), 8 rainfall events were recorded, 307 mm of rain and 9 significant water samples (6 of runoff and 3 of infiltration) were measured. In 2011 (from August 24 to September 13), 5 rainfall events were recorded, 110 mm of rain and 7 significant water samples (3 of runoff and 4 of infiltration) were also measured. All significant water samples measured in 2010 and 2011 were analyzed with a gas chromatograph in order to express endosulfan concentrations in runoff, infiltration and its exported quantities by rainfall events. At the end of each rainy season in 2010 and 2011, a mass balance was established and in order to optimize it, endosulfan concentrations and matter quantities exported in the lost water samples (during the sampling and the laboratory storage) were estimated [32;33]. During these experiments, it has supposed that endosulfan was degraded over time, so, in runoff case, its concentrations were calculated in 2010 and 2011 according to kinetic equation of first order:

$$C(T) = C_1 * e^{-KT} \quad [1]$$

with $C(T)$ = endosulfan concentration as a function of time (T), C_1 = concentration measured at the first sampling day, $K = \frac{\log(2)}{DT50}$ is the proportionality coefficient and $DT50 = 86$ days, endosulfan half-life [34].

In infiltration case, concentrations were expressed according to the mass conservation law. Thus, exported quantities of endosulfan residues (μg) in runoff ($\sum Q_{\text{ruis}}$) and infiltration ($\sum Q_{\text{inf}}$) were calculated and reported to applied quantities (Q_{appl}). Exportation processes by runoff or hydraulic erosion are calculated by multiplying each calculated concentration (C_i) by runoff volume V_i ($n = 9$ and 8 respectively in 2010 and 2011):

$$\sum Q_{\text{ruis}} = \sum_{i=1}^n C_i * V_i \quad [2]$$

As well as, quantities (μg) of exported matter by infiltration are calculated also by multiplying each calculated concentration (C'_i) by infiltration volume V'_i ($m = 18$ and 6 respectively in 2010 and 2011):

$$\sum Q_{\text{inf}} = \sum_{i=1}^m C'_i * V'_i \quad [3]$$

III. RESULTS AND DISCUSSION

III.1 Pesticides used in the agricultural Korokoro watershed

The surveys conducted in Korokoro watershed from 2009 to 2011 have shown that farmers used mainly insecticides (table 1). The insecticides which were frequently used in cotton production are profenofos, cypermethrin and endosulfan. Among this group, endosulfan is the pesticide which agricultural use has been banned worldwide due to its human toxicity and environmental persistence [35]. However, in the Korokoro watershed, these pesticides are distributed

either by the local cotton company (i.e. CMDT or OHVN) or purchased through the informal sector. Generally, cypermethrin and profenofos are applied in liquid emulsifiable form or soluble concentrates but endosulfan is applied only in emulsifiable concentrates. During the cotton production, the phytosanitary treatments are performed from July to September of each cropping season. However, the treatments frequency and the application rate are also set by the farmer following the degree of parasitic infestation on cotton plants. Table 1 lists the pesticides that are used in the agricultural Korokoro watershed.

Table 1: Main pesticide formulations used in Korokoro watershed from 2009 to 2011

Commercial specialty	Use	Active ingredients	Chemical family
Atrafor 500 SC	Herbicide	Atrazine* (500 gL ⁻¹)	Triazine
Cytoforce 288 EC	Insecticide	Cypermethrin	Pyrethroid
		Monochrotophos	Organophosphate
Mistral 450 DP	Fongicide	Endosulfan	Organochlorine
		Chlorothalonil	Pyrethroid
Gazelle C 88 EC	Insecticide	Cypermethrin (72 gL ⁻¹)	Pyrethroid
		Acetamiprid*(16 gL ⁻¹)	Neonicotinoid
Thiofanex 500 EC	Insecticide	Endosulfan* (500 gL ⁻¹)	Organochlorine
Ténor 500 SC	Insecticide	Profenofos (500 gL ⁻¹)	Organophosphate
Cotogard 500 SC	Herbicide	Fluometuron	Phenylurea
		Prometryne	Triazine
Phaser	Insecticide	Endosulfan	Organochlorine
Emir 88 EC	Insecticide	Cypermethrin (72 gL ⁻¹)	Pyrethroid
		Acetamiprid (16 gL ⁻¹)	Neonicotinoid
Cyferfos 336	Insecticide	Cypermethrin (136 gL ⁻¹)	Pyrethroid
		Methamidophos (200 gL ⁻¹)	Organophosphate
Endosulfan 500 EC	Insecticide	Endosulfan *(500 gL ⁻¹)	Organochlorine
Malathion	Insecticide	Malathion	Organophosphate
Attakan C 344 SC	Insecticide	Cypermethrin* (144 gL ⁻¹)	Pyrethroid
		Imidacloprid (200 gL ⁻¹)	Neonicotinoid
Nomax 150 SC	Insecticide	Cypermethrin* (75 gL ⁻¹)	Pyrethroid
		Teflubenzuron (75 gL ⁻¹)	Benzoylurea
Calife 500 EC	Insecticide	Profenofos* (500 gL ⁻¹)	Organophosphate

* Monitoring pesticides in the agricultural Korokoro watershed

III.2 Occurrence of endosulfan residues in runoff and infiltration at lysimeter boxes

The experiments carried out in 2010 showed that endosulfan residues were more transported by runoff (6.5±2.9% of exported matter) than infiltration (0.1±0.09% of exported matter). It means that endosulfan exportation by water is rather than related to runoff than infiltration process. Therefore, the use of endosulfan by certain farmers in cotton production can contaminate watercourses at Korokoro watershed scale during significant rainfall events. Indeed, in

lysimeter boxes, after the first and second endosulfan application on cotton plants (August 28, 2010 and September 11, 2010), significant rainfalls and higher concentrations of endosulfan residues have been respectively occurred and measured in runoff samples than infiltration ones. These higher concentrations can be due to the fact that after application on cotton plants, endosulfan can reach the soil and be adsorbed on fine soil particles and according to these significant rainfall events, be more transported by runoff process. In addition, during

significant rainfall events, the raindrops can also impact the soil aggregates and favor slaking crusts formation which consequently reduce soil infiltration capacity and the roughness. Thus, soil surface becomes smooth and impermeable [36-38]. This phenomenon can thus limit infiltration and favor runoff. This can more explain the occurrence of higher concentrations of endosulfan residues in runoff samples than infiltration ones.

During the experiments conducted in 2011, low rainfall events (110 mm against 307 mm in 2010) have been recorded so that endosulfan residues were lowly measured in runoff ($0.1 \pm 0.09\%$ of exported matter) and infiltration ($0.2 \pm 0.04\%$ of exported matter). These low rainfall events have thus reduced runoff and infiltration processes so that low concentrations of endosulfan residues have been measured. However, the concentrations obtained from infiltration process mean that endosulfan is mobile in soil and this mobility can be more favored during significant rainfall events and this can contaminate groundwater. Indeed, at Korokoro watershed scale, the use of this pesticide by certain farmers in cotton production can be a potential contamination risk of domestic wells water by infiltration process. However, in a context of Sahelian climate particularly in Mali, the results of this study are different from those obtained in regions of temperate climates or Mediterranean but they nevertheless remain comparable to similar and previous scientific works carried out [39;40].

III.3 Contamination of domestic wells water by pesticides residues in Korokoro watershed

During the cropping season in 2010, pesticides organochlorines have mainly been detected in 9 investigative domestic wells water. The concentrations measured of these pesticides are shown in Fig.3 except those for profenofos and cypermethrin which although they were frequently used in cotton production by the farmers of Korokoro watershed. The results show also that domestic wells water contamination is particularly due to ancient (i.e. aldrin, dieldrin, lindane, DDT and its metabolites) and recent (i.e. endosulfan) use of organochlorine pesticides. Higher concentrations have been measured for dieldrin ($5.8 \mu\text{g.L}^{-1}$) and endosulfan ($1.8 \mu\text{g.L}^{-1}$). Generally, all the concentrations are higher than the guideline values 0.1 and $0.5 \mu\text{g.L}^{-1}$, recommended by the Council of European Union Directive (98/83/CE, 1998) respectively for each active ingredient separate and total active ingredients analyzed except aldrin and dieldrin for which the guideline value was set at $0.03 \mu\text{g.L}^{-1}$.

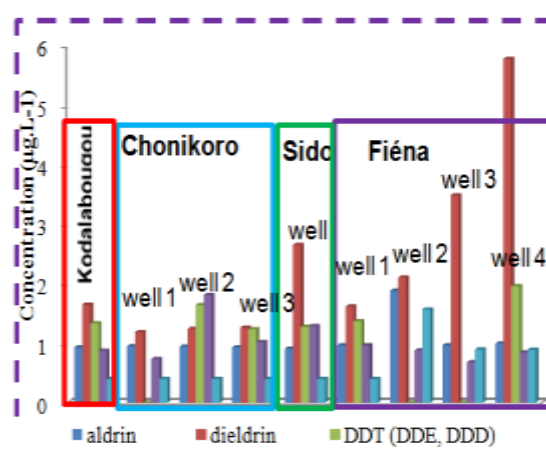


Fig. 3: Distribution of organochlorine pesticides in 9 investigative domestic wells of Korokoro watershed (January, 13, 2010).

As well as, during the cropping season in 2011, all water samples of domestic wells investigated (11 in total) have been contaminate by ancient and recent use of organochlorine pesticides as described above. However, profenofos and cypermethrin still none concern in these domestic wells water contamination although they have been applied on cotton plants by the farmers of Korokoro watershed. These two pesticides appear to be non-persistent in water than organochlorine pesticides. Indeed, organochlorine pesticides are known for their human and environmental toxicity and also potential persistence in air, soil and water compartments [35]. Higher concentrations have been measured for dieldrin ($0.8 \mu\text{g.L}^{-1}$) and aldrin ($0.2 \mu\text{g.L}^{-1}$). Generally, all the concentrations measured in 2011 are very low compared to those obtained in 2010 and this may be related to soil, climatic conditions, degree of parasitic infestation and the nature of the chemicals products used. The occurrence of organochlorine pesticides in 11 domestic wells water of Korokoro watershed is mentioned in Fig. 4 below.

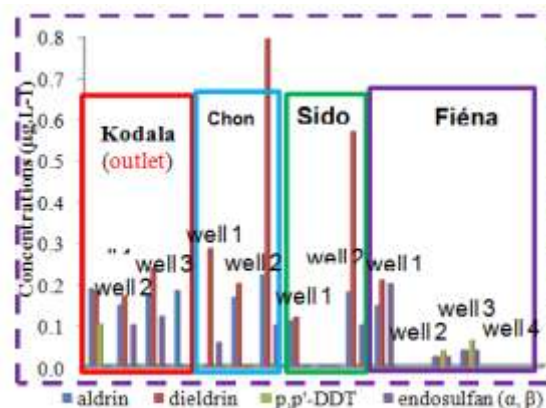


Fig. 4: Occurrence of organochlorine pesticides in 11 investigative domestic wells of Korokoro watershed (September 20, 2011).

In sum, experiments released in 2010 and 2011 in lysimeters showed that endosulfan residues were more transported in runoff process ($6.5 \pm 2.9\%$ of exported matter) than infiltration one ($0.1 \pm 0.09\%$ of exported matter). These results can more explain the contamination observed in all domestic wells water. At lysimeters scale, the importance of the transport by runoff process is due to significant rainfall events (52.5 mm, 101.4 mm, 37.8 mm and 28.2 mm) occurred the fourth, sixth, seventh and ninth day after the first treatment on cotton plants (August 28th, 2010) and those occurred on the eighth day (62 mm) after the second treatment (September 11th, 2010). At Korokoro watershed scale, such rainfall can be mainly factors of domestic wells water contamination. In fact, field plowing can promote pesticide residues which were accumulated in the surface horizon (0-20cm) to rise to soil surface and during significant rainfall events these pesticide residues are transported by runoff process to domestic wells. In addition, other factor as aeolian transport can contribute also in these domestic wells water contamination. Indeed, Organochlorine pesticides, like endosulfan and lindane are known to be transported to long distance compared to others like triazines [41;42]. As well as, in Korokoro watershed, certain factors such as the downstream position of domestic wells compared to the upstream one of cotton fields, the physical state of all domestic wells (lack of curbs, lids etc.) and the fact that pesticide solutions still prepared in the same fields, can contribute easily to domestic wells water contamination by runoff process [12;13].

IV. CONCLUSION

In Korokoro watershed, cotton farmers commonly use several chemical insecticides. The results of all the analyzes carried out in 2010 and 2011 in respectively 9 and 11 domestic wells water showed a contamination of all water samples by organochlorine pesticides except for profenofos and cypermethrin. This contamination of these domestic wells water is generally due to past and recent agricultural use of organochlorine pesticides in cotton production program. Higher concentrations have been measured than the guideline values recommended by European Union Directive Council. The occurrence of these pesticides residues in groundwater (domestic wells) of Korokoro watershed is a consequence of their potential persistence in environmental compartments. However, the experiments performed in lysimeters showed that endosulfan residues are more present in runoff processes than infiltration ones. According to these

results the occurrence of organochlorine pesticides in domestic wells water of Korokoro watershed can be considered as a runoff process contribution and an accidental one. However, this contamination of drinking water is a public health risk for all inhabitants of Korokoro watershed. There is therefore a need for further assessment in order to plus understand Korokoro watershed groundwater contamination by organochlorine pesticides.

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