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INVESTIGATION OF ORGANOCHLORINES RESIDUE IN STORED CEREALS FROM SOME SELECTED MARKETS IN JALINGO, NIGERIA

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Abstract

Cereal grains such rice, sorghum, maize and millet among others are the staple foods in large parts of the world, supplying most of the energy and bulk in diets. Pesticide residue analysis in cereals from the various sampling locations for this dissertation revealed the following organochlorines Aldrin, Dichloran, Dieldrin, Endrin, Endosulfan, Mirex, Lindane, Methoxychlor, Heptachlor Epoxide, 1,1,1-Trichloroethane, Hexachlorocyclohexane, and 2,2-bis(p-chlorophenyl). The presence of pesticide (Organochlorines) residues is detected in the samples of cereals (Rice, Maize, Millet and sorghum) analyzed. This could be as a result of high utilization of various pesticides during plant, cultivation and storage thus leading to the bioaccumulation of this substance in the individual cereals. It indicates high levels of non-carcinogenic risk associated with the life time consumption of cereals produce and sold within this region. Organochlorine pesticides also detected suggest the continuous use of obsolete banned pesticides in the cultivation and storage of cereals. Based on findings from this study's, I hereby make the following recommendations. Regulatory agencies in Nigeria should step up efforts to ensure compliance with the ban on these chemicals. Farmers and other pesticide users also need to be educated on the dangers of using banned products and on the over application of these pesticides. Furthermore, farmers should be educated on the properly usage of these chemical product, during plant, harvest and storage. Also, manufacturers of these chemicals should produce less toxic pesticide using organic materials in other to prevent the health risk associated with chemical pesticide and their residues in food crops and the environment.

Keywords: Cereal; Jalingo; Organochlorines; GC-MS

INTRODUCTION

Maintaining high agricultural output requires the use of pesticides, since, in high-input agricultural production systems, pests, among other crop invaders, including weed and fungi, inevitably need to be managed (Kortenkamp, 2007; Mnif *et al*., 2011). However, reliance on pesticides is unsustainable due to their harmful effects on the environment and human health. The risk to human health comes from direct or indirect exposure to pesticide residues in primary or derived agricultural products (Aravinna *et al*., 2018). Pesticide residue refers to the pesticides that may remain on or in food after they are applied to food crops. It is defined as any substance or mixture of substance in food for man or animals resulting from the use of pesticide and includes any specified derivatives, such as degradation and conversion products, metabolites, reaction products, and impurities that are considered to be of toxicological significance. Exposure to pesticide residues through the diet is assumed to be up to five times the magnitude of exposure through other routes such as air and drinking water (Zhang *et al*., 2017). Infants, children and adults are commonly exposed to pesticides by eating them on and in our food (Zhang *et al*., 2013). The presence of pesticide residues in fruits and vegetables can be a significant route to human exposure and most of organochlorine pesticides have been banned because they are highly persistent insecticides, and their residues still appear as pollutants in food as well as in the environment (Bando *et al*., 2022). Pesticides play a role in many human health problems, and can exert acute effects, such as dizziness, headaches, rashes, and nausea, and chronic effects, such as cancers, neurotoxicity, genotoxicity, birth defects, impaired fertility, and endocrine system disruption (Aravinna *et al*., 2018).

Pesticides applied to food crops in the field can leave potentially harmful residues (Gao *et al*., 2008). According to Mazlan *et al.* (2017), after pesticides are applied to the crops, they may interact with the plant surfaces, be exposed to the environmental factors such as wind and sun and may be washed of during rainfall. The pesticide may be absorbed by the plant surface (waxy cuticle and root surfaces) and enter the plant transport system (systemic) or stay on the surface of the plant (contact). The pesticides that get into the plant tissues may be transformed (metabolised) or sequestered in the tissues to form the pesticide residue. Pesticide residues are the deposits of pesticide active ingredient, its metabolites or breakdown products present in some component of the environment after its application, spillage or dumping (Dasika *et al*., 2012). The presence of pesticide residues is a concern for consumers because pesticides are known to have potential harmful effects to other non-targeted organisms than pests and diseases (Chakraborty *et al*., 2015). Residues of pesticides can be found in a great variety of everyday foods and beverages, including for instance cooked meals, water, wine, fruit juices, refreshments, and animal feeds (Witczak *et al*., 2014; Chourasiya *et al*., 2015). Furthermore, it should be noted that washing and peeling cannot completely remove the residues (Reiler *et al*., 2015). In the majority of cases, the concentrations do not exceed the legislatively determined safe levels (Chourasiya *et al*., 2016). However, these "safe limits" may underestimate the real health risk as in the case of simultaneous exposure to two or more chemical substances, which occurs in real-life conditions and may have synergistic effects (Kurwadkar, 2017). Worldwide pesticide production increased at a rate of about 11% per year, from 0.2 million tons in the 1950s to more than 5 million tons by 2000 (Carvalho, 2017). The large amounts of remaining pesticides penetrate or reach non-target plants and environmental media. As a consequence, pesticide contamination has polluted the environment and caused negative impacts on human health (Hernández *et al*., 2013).

Cereal grains are the seeds that come from grasses such as wheat, millet, rice, barley, oats, rye, triticale, sorghum, and maize (corn). About 80 percent of the protein and over 50 percent of the calories consumed by humans and livestock come from cereal grains (Sarwar, 2008). The United States is a major supplier of cereal grains to the rest of the world and some impoverished countries depend on gifts of both unmilled and processed grains from America to keep their people from starving. The global importance of cereal crops to the human diet and on the written history of man and agriculture cannot be over emphasised. Cereal grains are also referred to as the fruit of plants belonging to the grass family (Gramineae). Pesticides are chemical substances widely used against plant pests and diseases. The presence of pesticide residues in fruits and vegetables can be a significant route to human exposure and most of organochlorine pesticides have been banned because they are highly persistent insecticides, and their residues still appear as pollutants in food as well as in the environment (Zhang *et al*., 2013; Arowora *et al*., 2020). The food crops treated with pesticides invariably contain unpredictable amount of these chemicals, therefore, it becomes imperative to find out some alternatives for decontamination of foods. The washing with water or soaking in solutions of salt and some chemicals e.g. chlorine, chlorine dioxide, hydrogen peroxide, ozone, acetic acid, hydroxy peracetic acid, iprodione and detergents are reported to be highly effective in reducing the level of pesticides (Arowora *et al*., 2020). This research aimed to estimate the level of Organochlorines residue in some selected cereals produced consumed within Jalingo town, Taraba state, Nigeria.

METHODS

Study Area

Jalingo LGA is roughly located between latitudes 8º 47' to 9º 01'N and longitudes 11º 09' and 11º 30'E. It is bounded to the north by Lau Local Government Area, to the east by Yorro Local Government Area, to the south and west by Ardo Kola Local Government Area. It has a total land area of about 195km^2 with an estimated human population of 139,845 people according to the 2006 national population census.

Sample preparation

Cereal (Maize, millet, sorghum and rice samples) were cleaned by picking out stones, weevils and other non-essential materials. The different samples were then milled separately, first, using a mortar and pestle and then finally milled to powder using a hand-grinding machine. The bean samples were purchased in dry form and no further drying was required. Precautionary measures were taken to avoid cross-contamination of the different samples during and after milling.

Extraction

Extraction was done using the method of Zhang *et al.* (2011), with slight modifications. An aliquot of 10g of each powdered sample was weighed, using an electronic weighing balance, into a 250 ml beaker. Initial weight of the beaker was recorded as Wb and the weight of beaker with sample was recorded as Wb+s. 30 ml of the extraction solvent was introduced into 10g of the powdered sample contained in the beaker. The mixture was properly sealed airtight using foil and tape. This was allowed for 48 h for extraction. The samples after extraction were filtered using filter paper and funnel and then concentrated by evaporation at room temperature for 24 to 72 h. The organic residue was then diluted with 1ml n-hexane and stored in reagent bottles ready for Gas chromatography–mass spectrometry (GC-MS) analysis.

Identification and quantification

Pesticide residues were identified by comparison of the retention times, peak area and peak heights of the sample with those of the standards. Pesticide concentrations were within 5% of the standards.

Data analysis

Statistical analysis was done using SPSS version 20. Results were presented as mean ± standard deviation.

RESULTS

Pesticide residue analyzed in cereals from Mile six market is presented in table 1. The following organochlorine pesticide residues were present in the cereals samples; Aldrin, Dichloran, Dieldrin, Endrin, Endosulfan, Mirex, Lindane, Methoxychlor, Heptachlor Epoxide, 1,1,1-Trichloroethane, Hexachlorocyclohexane, and 2,2-bis(p-chlorophenyl). From the result presented, among all the pesticide present in rice as shown in table 1, Endosulfan has the highest concentration 0.064±0.001 followed by Aldrin 0.053±0.001, on the other hand, 2,2-bis(p-chlorophenyl) had the lowest concentration in rice followed by Dieldrin 0.021±0.000. For Sorghum, the highest pesticide residue was Endosulfan with a concentration 0.018±0.002 followed by Aldrin with a concentration of 0.015±0.002 while the lowest concentration observed in millet was in 2,2-bis(p-chlorophenyl) with a concentration of 0.005±0.000 followed by Dieldrin and Hexachlorocyclohexane with a concentration of 0.006±0.001. The evaluation of organochlorines in Maize at Rafinkada indicate Endosulfan had the highest concentration 0.028±0.000 followed by Aldrin with the concentration of 0.024±0.001 while the pesticide residue with the lowest concentration was 2,2-bis (p-chlorophenyl with a concentration of 0.008±0.002 followed Dieldrin with a concentration of 0.009±0.002. For millet, the pesticide residue with the highest concentration was Endosulfan with a residual concentration of 0.141±0.002 followed by Aldrin with a concentration of 0.118±0.002. The lowest concentration was however observed in 2,2-bis(p-chlorophenyl) with a residual concentration of 0.042±0.003 followed by Dieldrin with a concentration of 0.047±0.003.

Table 1: Organochlorines present in cereals samples obtained from Mile six market.

Mile six market		Cereals			
Organochlorines (ppb)	Rice	Sorghum	Maize	Millet	MRL(EU/WHO)
Aldrin	0.053 ± 0.001^{c}	0.015 ± 0.002^{b}	0.024 ± 0.001^{c}	0.118 ± 0.002^{b}	0.010
Dichloran	0.046 ± 0.002^{c}	0.013 ± 0.001^{b}	0.020 ± 0.001^{c}	0.102 ± 0.001^{b}	0.050
Dieldrin	0.021 ± 0.000^{a}	0.006 ± 0.001^{a}	0.009 ± 0.002^{b}	0.047 ± 0.003^{a}	0.100
Endrin	0.038 ± 0.002^{b}	0.011 ± 0.000^{b}	0.017 ± 0.001^{c}	0.083 ± 0.025^{b}	0.100
Endosulfan	0.064 ± 0.001^{d}	0.018 ± 0.002^{b}	0.028 ± 0.000^{c}	0.141 ± 0.001^{b}	0.100
Mirex	0.045 ± 0.001^{c}	0.013±0.001b	0.020 ± 0.000^{c}	0.098 ± 0.023^{b}	0.050
Lindane	0.048 ± 0.000^{c}	0.014 ± 0.001^{b}	0.021 ± 0.001^{c}	0.106 ± 0.005^{b}	0.050
Methoxychlor	0.045 ± 0.002^{c}	0.013 ± 0.000^{b}	0.020 ± 0.000^{c}	0.100 ± 0.000^{b}	0.014
Heptachlor Epoxide	0.049 ± 0.005^{c}	0.014 ± 0.001^{b}	0.022 ± 0.000^{c}	0.109 ± 0.004^{b}	0.050
1,1,1-Trichloroethane	0.040 ± 0.000^{b}	0.011 ± 0.000^{b}	0.018 ± 0.001^{c}	0.089±0.005b	0.010
Hexachlorocyclohexane	0.022 ± 0.001^{a}	0.006 ± 0.001^{a}	0.010 ± 0.000^{b}	0.048 ± 0.003^{a}	0.020
2,2-bis(p-chlorophenyl)	0.019 ± 0.003^{a}	0.005 ± 0.000^{a}	0.008 ± 0.002^{a}	0.042 ± 0.001^{a}	0.010

Result presented in mean ± standard deviation. Result with the same superscript down the column indicates no significant difference, while result within the same column with different superscripts indicates significant difference ($p\leq0.005$).

Table 2: Organochlorines present in cereal samples obtained from Jalingo main market

Jalingo main market	Cereals				
Organochlorines (ppb)	Rice	Sorghum	Maize	Millet	MRL(WHO)
Aldrin	0.196±0.032[c]	0.009±0.004[b]	0.008±0.002[b]	0.327±0.005[c]	0.010
Dichloran	0.170±0.020[c]	0.008±0.003[b]	0.007±0.000[b]	0.284±0.005[c]	0.050
Dieldrin	0.079±0.001[a]	0.004±0.000[a]	0.003±0.000[a]	0.131±0.001[a]	0.100
Endrin	0.138±0.005[b]	0.007±0.002[b]	0.005±0.001[b]	0.230±0.006[b]	0.100
Endosulfan	0.235±0.003[c]	0.011±0.001[b]	0.009±0.004[b]	0.391±0.005[d]	0.500
Mirex	0.164±0.005[c]	0.008±0.003[b]	0.006±0.002[b]	0.273±0.002[c]	0.050
Lindane	0.176±0.004[c]	0.008±0.001[b]	0.007±0.001[b]	0.293±0.001[c]	0.050
Methoxychlor	0.166±0.005[c]	0.008±0.002[b]	0.006±0.001[b]	0.277±0.003[c]	0.014
Heptachlor Epoxide	0.181±0.001[c]	0.009±0.004[b]	0.007±0.001[b]	0.302±0.001[c]	0.050
1,1,1-Trichloroethane	0.148±0.005[b]	0.007±0.002[b]	0.006±0.002[b]	0.246±0.005[b]	0.010
Hexachlorocyclohexane	0.079±0.004[a]	0.004±0.001[a]	0.003±0.000[a]	0.132±0.001[a]	0.020
2,2-bis(p-chlorophenyl)	0.070±0.006[a]	0.003±0.000[a]	0.003±0.000[a]	0.116±0.002[a]	0.010

Result presented in mean ± standard deviation. Result with the same superscript down the column indicates no significant difference, while result within the same column with different superscripts indicates significant difference ($p \leq 0.005$).

Table 3: organochlorines present in cereal samples obtained from Mayo dassa market

Mayo dassa market			Cereals		
Organochlorines (ppb)	Rice	Sorghum	Maize	Millet	MRL (EU/WHO)
Aldrin	0.013±0.001[b]	0.147±0.002[b]	0.019±0.004[a]	0.928±0.005[b]	0.010
Dichloran	0.011±0.000[b]	0.128±0.003[b]	0.039±0.005[d]	1.367±0.002[c]	0.050
Dieldrin	0.005±0.001[a]	0.059±0.004[a]	0.013±0.001[a]	0.154±0.001[a]	0.100
Endrin	0.009±0.002[a]	0.104±0.001[a]	0.026±0.003[bc]	0.206±0.000[a]	0.100
Endosulfan	0.015±0.001[b]	0.176±0.005[c]	0.016±0.002[a]	1.021±0.000[c]	0.500
Mirex	0.011±0.000[b]	0.123±0.002[b]	0.038±0.003[d]	0.477±0.002[b]	0.050
Lindane	0.012±0.001[b]	0.132±0.000[b]	0.030±0.005[c]	1.044±0.001[c]	0.050
Methoxychlor	0.011±0.000[b]	0.124±0.002[b]	0.036±0.003[d]	1.016±0.001[c]	0.014
Heptachlor Epoxide	0.012±0.001[b]	0.136±0.005[b]	0.027±0.004[bc]	1.212±0.001[c]	0.050
1,1,1-Trichloroethane	0.010±0.000[a]	0.111±0.001[b]	0.027±0.001[bc]	0.966±0.001[c]	0.010
Hexachlorocyclohexane	0.005±0.001[a]	0.060±0.005[a]	0.020±0.005[b]	1.124±0.051[c]	0.020
2,2-bis(p-chlorophenyl)	0.005±0.001[a]	0.052±0.003[a]	0.025±0.000[b]	1.038±0.001[c]	0.010

Result presented in mean ± standard deviation. Result with the same superscript down the column indicates no significant difference, while result within the same column with different superscripts indicates significant difference ($p \leq 0.005$).

Table 4: organochlorines present in cereal samples obtained from Mayo gwoi market

Mayo gwoi market		Cereals			
Organochlorines (ppb)	Rice	Sorghum	Maize	Millet	MRL (EU/WHO)
Aldrin	0.575±0.002[c]	0.084±0.001[d]	0.745±0.002[d]	0.273±0.003[c]	0.010
Dichloran	0.847±0.002[c]	0.000±0.000[a]	1.098±0.003[d]	0.402±0.003[d]	0.050
Dieldrin	0.095±0.001[a]	0.024±0.001[b]	0.124±0.001[a]	0.045±0.002[a]	0.100
Endrin	0.127±0.002[a]	0.036±0.003[c]	0.165±0.001[b]	0.060±0.002[a]	0.100
Endosulfan	0.633±0.001[c]	0.024±0.002[b]	0.820±0.005[d]	0.300±0.005[c]	0.500
Mirex	0.295±0.005[b]	0.024±0.001[b]	0.383±0.001[c]	0.140±0.002[b]	0.050
Lindane	0.647±0.001[c]	0.012±0.001[a]	0.839±0.004[d]	0.307±0.002[c]	0.050
Methoxychlor	0.629±0.004[c]	0.012±0.001[a]	0.816±0.001[d]	0.299±0.004[c]	0.014
Heptachlor Epoxide	0.751±0.001[c]	0.036±0.001[c]	0.973±0.003[d]	0.357±0.002[d]	0.050
1,1,1-Trichloroethane	0.599±0.003[c]	0.012±0.000[a]	0.775±0.001[d]	0.284±0.005[c]	0.010
Hexachlorocyclohexane	0.697±0.001[c]	0.012±0.001[a]	0.902±0.100[d]	0.331±0.001[c]	0.020
2,2-bis(p-chlorophenyl)	0.643±0.003[c]	0.036±0.002[c]	0.833±0.005[d]	0.305±0.000[c]	0.010

Result presented in mean ± standard deviation. Result with the same superscript down the column indicates no significant difference, while result within the same column with different superscripts indicates significant difference ($p \leq 0.005$).

DISCUSSION

Analysis of residual pesticide in cereals at the various sampling locations within Jalingo metropolis of Taraba state revealed the presence of organochlorines and organophosphate in the various cereals samples at different concentration. Residues of organochlorine pesticides have the peculiar characteristics of relatively high chemical stability and persist in the environment for long periods (Singh *et al*., 2012; Chakraborty *et al*., 2015). The following organochlorines were confirmed in the cereals samples; Aldrin, Dichloran, Dieldrin, Endrin, Endosulfan, Mirex, Lindane, Methoxychlor, Heptachlor Epoxide, 1,1,1-Trichloroethane, Hexachlorocyclohexane, and 2,2-bis(p-chlorophenyl).

The residual concentration of organochlorines in rice, Sorghum, Maize, Millet ranges obtained from Mile six market ranges between 0.019ppm to 0.064ppm, 0.005±0.000ppm to 0.018±0.002ppm, 0.008±0.002 to 0.028±0.000ppm and 0.042±0.001 to 0.141±0.001ppm respectively. According to literature, the Maximum Residue Limit (MRLs) range of organochlorine is from 0.01 – 1.00 mg/kg; and of which the cereal grains used in this analysis are found to be in this range with exception of Aldrin and Endosulfan whose values are slightly above the EC standard. The levels of organochlorines from samples analyzed from Jalingo main market in rice, sorghum, maize and millet ranges between 0.070±0.006 to 0.235±0.003, 0.003±0.000 to 0.011±0.001, 0.003±0.000 to 0.009±0.004 and 0.116±0.002 to 0.391±0.005 respectively. However, the residual concentration of all the pesticide evaluated in rice and millet in Jalingo main market were above WHO accepted range of pesticide residue in cereals with an exception of Endosulfan whose values is below the maximum acceptable range. On the other hand, pesticide residual concentration evaluated in sorghum and maize from Jalingo main market were below WHO standard of pesticide residual concentration in cereals. Also, the organochlorine residual concentration in cereals samples obtained from Mayo dassa market as presented in table 3 revealed the residual concentration of the evaluated cereals rice, sorghum, maize and millet ranges between 0.005±0.001 to 0.015±0.001ppm, 0.052±0.003 to 0.176±0.005ppm, 0.013±0.001 to 0.038±0.003ppm, and 0.154±0.001 to 1.367±0.002ppm respectively. Pesticide residual concentration in rice and maize were with EC residual limit, on the other hand, pesticide residual concentration in sorghum were slightly EC maximum residual concentration with the exception of Hexachlorocyclohexane and 2,2-bis(p-chlorophenyl) whose residual concentration are within EC maximum residual limits. The residual concentration of pesticide in millet revealed the residual concentration were above WHO, 2012 maximum residual limits in cereals. The residual concentration of organochlorines pesticide in cereals obtained from Mayo gwoi market is presented in table 4. The residual concentration of rice, sorghum, maize and millet ranges between 0.095±0.001 to 0.847±0.002, however this residual range is between EC maximum residual range in cereals, 0.000±0.000 to

0.084±0.001, this residual range is however within the EC maximum residual limit in cereals, 0.165±0.001 to 1.098±0.003, similarly, these values are however with the EC maximum residual limits for pesticide residue in cereals except for Dichloran which is slightly above the EC maximum residual limit, similarly the residual concentration of pesticide ranges between 0.045±0.002 to 0.357±0.002, however these residual range are between the EC maximum residual range in cereals respectively. Findings from this study are slightly in consonance with findings of Arowora *et al.*, who reported similar organochlorine pesticide residual concentrations in cereal samples obtained from some selected areas in Wukari (Arowora *et al.,* 2019).

There were 12 different forms of organochlorine pesticide residues observed in the course of this analysis and they ranged from 0.025±0.007 to 1.19±0.084 mg/kg (Table 3). The organochlorine pesticides detected include Aldrin, Dieldrin, Dichloran, Endrin, Endosulphan, Heptachlor Epoxide, Mirex, Lindane, Methoxychlor, 1,1,1-Trichloroethane, Hexachlorocyclohexane, and 2,2-bis(p-chlorophenyl). Aldrin and alpha- BHC were detected in all samples with concentrations ranging from 0.005 ± 0.0017 mg/kg to 0.054 ± 0.0225 mg/kg and 0.015 ± 0.002 mg/kg to 0.075 ± 0.020 mg/kg respectively. These values were below their MRL as indicated in Table 2. A study by Lozowicka *et al.* (2017) also revealed the presence of organochlorine pesticides such as Aldrin, dieldrin, p, p'DDE and p,p'DDD, among others from samples of beans, cucumber, lupine, tomatoes, carrots, celery and parsley collected from Kazakstan and Poland. Previous works by Sultana *et al.* (2014) have detected the presence of organochlorine pesticides such as Aldrin, dieldrin and DDT at concentrations higher than the MRL, set by European Union, in bean samples from Maiduguri and Lagos respectively. Many of the older, cheaper (off-patent) pesticides, such as dichlorodiphenyltrichloroethane (DDT) and lindane, can remain for years in soil, water and food products. These chemicals have been banned by countries who signed the 2001 Stockholm Convention – an international treaty that aims to eliminate or restrict the production and use of persistent organic pollutants (World Health Organisation, WHO, 2012). The MRL is not expected to be exceeded in any foodstuff if the pesticide was applied in accordance with directions for its safe use (Sultana *et al.*, 2014). If, however, a residue in a food sample exceeds the MRL, the food commodity is unsafe for consumption because it contains an unsafe or illegal amount of the residue. This suggests the high use of organochlorine pesticides in the storage and or cultivation of the crops in Jalingo and its environs.

CONCLUSION

Cereal grains such rice, sorghum, maize and millet among others are the staple foods in large parts of the world, supplying most of the energy and bulk in diets. Pesticide residue analysis in cereals from the various sampling locations for this dissertation revealed the following organochlorines Aldrin, Dichloran, Dieldrin, Endrin, Endosulfan, Mirex, Lindane, Methoxychlor, Heptachlor Epoxide, 1,1,1-Trichloroethane, Hexachlorocyclohexane, and 2,2-bis(p-chlorophenyl). The presence of pesticide residues is detected in the samples of cereals (Rice, Maize, Millet and sorghum) analyzed. This could be as a result of high utilization of various pesticides during plant, cultivation and storage thus leading to the bioaccumulation of this substance in the individual cereals. It indicates high levels of non-carcinogenic risk associated with the life time consumption of cereals produce and sold within this region. Organochlorine pesticides also detected suggest the continuous use of obsolete banned pesticides in the cultivation and storage of cereals.

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