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Pesticide Residues of Organochlorine in Some Common Foods Cultivated in South Gezira Valley (Sudan)

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Abstract
This study was conducted to assess the presence of organochlorines insecticide residues in some common foods from El-Hosh Town community, South Gezira. 48 samples (16 tomatoes, 16 meats and 16 eggs) were randomly collected during the period December 2007 to August 2008. Pesticide residues were determined by gas chromatograph with electron-capture detector (GC-ECD) after dichloromethane extraction and cleanup on Silica gel phase cartridges. The results revealed that, only DDE was detected. All food samples were free of organochlorine pesticides in the four seasons of the years except for two meat samples in spring season. Concentrations of these samples were 1.102 to 0.811 ppm. These findings suggest that, a restricted and controlled use of such persistent pesticides may be useful for decreasing their contamination levels in different food items, therefore, the monitored varieties of food are safe to people's health. This study recommended continuous monitoring of persistent organochlorine pesticides in food in order to safeguard human health and mark their decline with time after the restriction of their usage.

Keywords: Residues of pesticide, Monitoring, Some Common Foods, Sudan.

1. Introduction
Food has been recognized as a major source of human exposure to organochlorine pesticide residues (OCP). Because of their lipophilic properties and their high persistence they can be accumulated in human body by regular consumption of plant products [1]. Live- stock meat and dairy products are a primary source of human dietary exposure to organochlorine (OC), since between 60 – 85% of the mean daily intake arose from these particular food classes. OCP predominately accumulate in the lipid fractions of human food chain and hence animal fatty foods have become a major route of exposure for humans [2].

Animals, such as cows, living in areas where the OCP are present in the environment accumulate their residues when they eat contaminated feed [3-6] and when they inhale contaminated air [7-10]. OCP volatilization is considered as a major process in moving these chemicals from treated areas through air currents, resulting in potential exposure to pasturing animals [11-13]. Agricultural soils of different countries are suspected to be an important source of old OCP due to the large quantities used [14]. Therefore, consumption of meat and fat may expose people to unexpected high residue levels [15].

The OC are a problem because they persist in the environment, posing a potential risk of residue in grazing cattle, in both meat and milk product. OC residues are stored in the fat deposits of cattle. The concentration of the chemical in the fat of cattle is higher than in the feed usually by a factor 10 to 15 [16]. Generally, there is a movement of OC residue from contaminated soil to pasture plant, although some root crops (potato, turnip, beet root, etc) can accumulate residues is the tubers or root growth. FAO/WHO have reported that, the incidence of HCH and DDT residues are higher in fatty food items such as milk, milk product, fat, edible oils and meat than in non fatty foods [17]. In most studies, 80 – 90% of the total intake of OCP in non occupationally exposed human was accumulated via food [18], so in many countries OCP in food and animal products are monitored to ensure that public health is not endangered by residues taken daily in excess of the recommended tolerance levels [2].

In spite of the restriction of their use three decades ago, residues of halogenated insecticides continue to be detected in environmental samples from the Gezira area. In view of this, and considering the long history of usage, OC are expected to be present as contaminants in the Gezira for decades to come.

2. Experimental

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Sampling
The common diet components, tomato, meat and eggs were selected for the study. Samples were collected during the period december 2007 to august 2008. 48 tomato fruit, meat and eggs samples were randomly collected from El-Hosh market at different regions. The samples were collected and were stored at 5 °C until analysis.

Chemical and analytical standard
Acetone, dichloromethane, n-hexane celite, alumina and potassium hydroxide anhydrous sodium sulphate (pesticide residue grade) were obtained from Panreac (Barcelona, Spain). Silica gel adsorbent was obtained from Sigma–Aldrich (St. Louis, MO, USA). Certified standards of Gamma-HCH (99.6% purity), Aldrin (99.0% purity) and Heptachlor epoxide (99.0% purity) DDE (99.2 purity%) were supplied by Dr. Ehrenstorfer (Augsburg, Germany). Individual stock standard solutions were prepared in acetone. Standard solutions for gas chromatographic (GC) analysis were prepared by suitable dilution of the stock standard solutions with n-hexane.

Extraction and clean up procedure
Samples of tomatoes and meat were extracted using the method described by Ambrus [19] and Miyahara [20]. Samples were cut and shaken well by hand and 25 g were weighted out. 100 ml acetone were added to jar containing 25 g tomatoes sample and blended for 2 min at high speed. The extract was filtered and transferred to a separatory funnel. 225 ml distilled water were added and the mixture was extracted in methylene chloride. The extracts were filtered through 15 g of anhydrous Na_2SO_4 and rinsed with 20 ml methylene chloride. The combined methylene chloride extract was reduced to 2 ml in a vacuum rotary evaporator at 30°C. 10 ml acetone was added and evaporated to 2 ml. 15 g of silica gel deactivated with 10% water was packed in a column (22 mm id. x 300 mm), with slight tapping. The extract was deposited on the top of the column and was eluted with 150 ml of a mixture of hexane and dichloromethane (80/20). The eluate was evaporated and the residue dissolved in 5 ml of hexane. All samples were mixed with two drop of 10% potassium hydroxide in order to convert DDT into DDE prior to analysis.

Samples of eggs were extracted by using the method described by Wardall [21]. 5 g of eggs were ground with 20 g of anhydrous sodium sulphate. 5 g of celite was added and mixed. Chromatography column was filled to half with hexane. 5 g of alumina were packed into the bottom of the column and egg powder was added by tapping care for removal of any air bubbles. The tap of the column was opened and 100 ml of the eluate was collected in a calibrated flask. All extracts were mixed with two drops of 10% potassium hydroxide.

Condition chromatographic
TLC plates were divided into longitudinal columns made by scraping the silica gel. About 10 µl of pesticide standard and samples extract were spotted, the plates were placed in a developing tank containing 100 ml of n–heptane which was the best system for separation of the pesticides in the current investigation. When the solvent front approached 10 cm above the base line, the plates were removed from the tanks, allowed to dry at room temperature and after dryness the plates were visualized under short wave ultraviolet lamp at 254 nm.
The analysis was carried out by gas chromatography. Using a carlo Erba Fracto Vap 2101 equiped with electron capture detector (ECD). Glass column used was 3m x 0.25 mm id packed with 5% OV-210 on chromosorb WHP 80-100 mesh. Temperature of the injection block, oven and detector were 250, 190, 300°C respectively. Nitrogen carrier gas flow rate 60 ml/minute. Injection volume for standards and samples was 1µl.

Statistical analysis
The theoretical limit of detection, defined as the concentration of analyte that gives a signal equivalent to the blank signal plus three times its standard deviation, was calculated for each individual pesticide. In this work, the limit of detection (LOD) was taken to be the amount of analyte that gave a signal that was clearly distinguishable from the background noise of the instrument [22]. The theoretical limit of quantification (LOQ) was defined as the concentration of analyte that gave a signal equivalent to the blank signal plus ten times its standard deviation [22]. The analytical determinations were made in triplicate for each sampling. Mean values and standard deviations were calculated and analysed by JMP package program (SAS Ins., CARY, NC, USA) for analysis of variance. Statistical discrimination of the mean values was performed using the method of contrasts [22].

3. Results and Discussion

Performance of the analytical method
The linear dynamic range, precision (as relative standard deviation) and sensitivity (as limit of detection) values for determination of Aldrin, DDE, gamma HCH and Heptachlor epoxide are reported in Table 1.

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Table 1. Figures of merit obtained for the used method.

Analyte	Linear range µg mL^{-1}	Y = (a ± S_a) X + (b ± S_b)	R^2	LOD µg mL^{-1}	LOQ µg mL^{-1}	RSD (%) (n=5)
Gamma HCH	0.015-0.100	(8363.5±156.4801) X - (0.651±0.6520)	0.9995	0.003	0.009	3.6
Heptachlor epoxide	0.020-0.200	(20041±123.3099)X - (55.442±7.5006)	0.9997	0.001	0.003	4.3
DDE	0.025-0.200	(9373.5±166.4601) X - (0.875±0.8750)	0.9998	0.002	0.007	4.7
Aldrin	0.003-0.050	(19041±1136) X - 7.5006)	0.9999	0.001	0.004	4.3

a; slope a; b, Intercept; *R*, regression coefficient; LOD, limit of detection; LOQ, limit of quantification, RSD, relative standard deviation.

Linear range: Individual calibration graphs were run with mixtures of all pesticide studied at concentrations in the range 0.003–0.200 µg mL^{-1}. Each solution was injected five times. The linear range, intercept and slope of the curve are given in Table 1 along with the regression coefficient for each pesticide.

Sensitivity: The LODs calculated for all pesticide in this way were 0.001 µg mL^{-1} to 0.003 µg mL^{-1}. The limits of quantification (LOQ) were 0.003 µg mL^{-1} to 0.009 µg mL^{-1}.

Precision: Untreated samples were fortified by the addition of an intermediate pesticide mixture solution. Samples were allowed to equilibrate for 2 h prior to extraction and were processed according to the procedure described above. The precision values for the method, expressed as relative standard deviation (RSD), were 3.6 to 4.7 % (n = 5).

Evaluation of pesticide residues in some common foods cultivated in South Gezira Valley

The results of analysis of pesticide residues indicated that we have no residues of organochlorine (OC) compounds were detected in all samples such as tomato, meat and egg analyzed in four seasons of the year 2007/2008 except for meat in spring season. DDE was the only organochlorine determined and was found in four samples in spring season. Concentrations of residues in these samples were 1.102 ppm to 0.811 ppm. These results are not surprising despite the fact that, organochlorine persist in the environment, since all these organochlorine compounds were banned or severely restricted in use in most countries in the late 1981. This result is in agreement with Arino [23] and Venant [24] who stated that, in the last 25 years, consumer demand for residue – free food has resulted in the introduction of numerous laws and regulations designed to control environmental distribution of these potential food contaminant, owing to regulation in several countries the levels of most OCP in food showed decreases in the period 1980 – 1990. The fact that, concentrations of DDE were still observed in the meat samples may be due to atmospheric deposition.

The frequent detection of these OCP in cow meat samples from El-Hosh showed their presence in the environment due to their past use in agriculture, contemporary volatilization from contaminated soil and contamination of growing plants.

A significant proportion of residues in animals is acquired by consumption of plants growing in contaminated soil. The amount of soil consumed depends largely on the amount of grass cover, however, cattle can consume between 20 to 1200 grams of soil per day. Contaminated soil can also be transferred to herbage by dust, rain – drop, splash or flooding. Flood rains transport OC that are attacked to soil particles from one property or paddock to another. Run off from contaminated soils to farm water supplies occur, but significant contamination of cattle from this route is unlikely because of the high dilution factor and the fact that, residues are tightly bound to sediment. Mohammed [25] reported that, animal exposure to HCH can occur through the use of lindane as a dipping chemical to control Scab mostly in sheep, which may produce a carcass unsuitable for immediate home and export consumption. This residue can persist in the fat after immersion in a dip of recommended concentration and may take as long as 12 weeks before it drops to a concentration of 1 to 2 ppm, the Codex Alimentarius level of Safety.

The WHO quoted from Mohammed [25] has set a practical residue limit for total DDT in cow's milk of 0.05 ppm. The Food and Drug Administration uses this value as the maximum permissible concentration of total DDT in the regular monitoring of commercial cows milk shipped in interstate commerce. The pesticides tend to become more concentrated as one sample up a food chain [26], that is, meat – eaters (including man) store more DDT in their tissues than do herbivores, such as cattle, hence human milk would be expected to contain more DDT than that from cows. Zweig [27], have reported that, cattle fed a diet containing 0.5 ppm of DDT excreted less than 0.01 ppm of the pesticide in their milk. However, at levels of 1, 2, 3 and 5 ppm of added DDT, proportional to the level of contamination in the feed, were found in the milk of all animals. The cows fed DDE at the lowest level (0.5 ppm) ate

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an average of 20 kg feed/day. Thus their daily dose of DDE was 10 mg/day. Assuming that, the average weight of these animals was 400 kg, it may be calculated that the dosage was 0.025 mg/kg/day. This dosage resulted in a DDT concentration of < 0.01 ppm in the milk of the cows.

As compared with earlier studies, the present levels of the contaminants are substantially low, an indication of the gradual phase out of these compounds as well as a low rate of influx and continued weathering of DDE in the environment.

Conclusions

All food samples such as tomatoes, meat and egg were free of organochlorine pesticide residues in the four seasons of the years except for meat in spring season including low risk of human exposure through food consumption. The advantages of application of pesticides in agriculture to produce better crops, must be weighed against possible health hazard arising from the toxic pesticide residues in food. Pesticides should be applied correctly according to good agricultural practices, using only the required amounts. There is a need for the continuous monitoring of persistent organochlorine pesticides in food in order to safeguard human health and mark their decline with time after the restriction of their usage. The government of Sudan has the resource to use existing public health laboratories to establish a toxicology network, which would support the integrated actions of environmental quality and human health control. However, the first step for pesticide control must be a revision of the state laws on pesticides.

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