

INSECTICIDE RESIDUES IN SOME STORED GRAINS IN MUBI, ADAMAWA STATE, NIGERIA AND THEIR POTENTIAL HEALTH HAZARDS ON THEIR CONSUMER

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ABSTRACT

The study was conducted to assess the level of residue of permethrin (pifpaf), Aluminium phosphide (cyclone) and Dichlovors (dakshi) in protected grains of cowpea, maize and bambaranut groundnut each stored for 1 month, 3 month, and 6 months in Mubi (10° 16 N, 13° 10 E) Nigeria. Based on grain type and time of storage, the pesticide were extracted from 2g of powdered grains using acetonitrile and methanol (4:1) and then analyzed for the composite fractions using the high performance liquid chromatography technique. The result shows that each of the grains has residues of all the insecticide considered in the study. On average, cowpea had higher levels of insecticide residues while maize had lower over the study period. The lowest residue of any of the insecticide (1.0mg/kg of dakshi, 1.04mg/kg of cyclone and 1.11mg/kg of pifpaf) 6 months after storage was higher than the FAO no-observed adverse effect level, even though residue levels decreased with storage time. The implications of the findings on insecticide based stored grains protected by Nigerian merchants/farmers are discussed.

Key words: Insecticide, Residue, Stored grains, Pest management.

INTRODUCTION

Food security in sub-Saharan Africa largely depends upon improved food productivity through the use of sustainable agricultural practices and the reduction of post harvest losses caused by pests and diseases (Adedire, 2001). Storage insect pests cause substantial damage to the stored grain (Mvumi and Stathers, 2003), however, Kennedy (1998), pointed out that to ensure high food quality and standards, which are acceptable to the consumer, quality control, including good storage and handling prac-

tices, must be observed at all times. Managing stored grains requires the use of various techniques to ensure that the quality of the grain entering the storage facility does not deteriorate over time. These measures include: the use of sanitation; storing sound, dry grain; managing temperature and aeration; and using chemical protectants, regular sampling, and fumigation (Okori *et al.*, 2004).

For decades, the pest control policy in developing countries has been dependent upon the use of synthetic pesticides (Ogendo *et al.*,

2004). Pesticides are an important part of food production. Without pesticides food would be more expensive, food production would require more labor and more intensive, knowledgeable management (Okori *et al.*, 2004). In addition, farmers would produce less food and supplies would be more variable, our food supply's quality would be lessened, and storage life of some fresh foods will be reduced (Huis, 1989; Kumar, 1991).

Pesticides are chemical substances defined as poisons and used in certain circumstances to kill specifically targeted pests (Wassemann, 1972). Some of these highly stable compounds can last for years and decades before breaking down. They circulate globally, and persistent pesticides released in one part of the world can be transported through a repeated process of evaporation and deposit through the atmosphere and as residue on treated surfaces of material to regions far away from the original source (Williams, 2000). Pesticides are toxic in nature and do not differentiate between target and non-target species, and hence should essentially be subject to safe and judicious use. Due to injudicious and indiscriminate use of pesticides, many accidents have occurred in different parts of the World, and presence of pesticides in foods, fruits, vegetables, and environment and even in mother's milk is a matter of grave concern (FAO/WHO, 2005). Of all the pesticides released into the environment every year by human activity, persistent pesticides are among the most dangerous (FAO/WHO, 2005). They are highly toxic, causing an array of adverse effects, notably death, diseases and birth defects among human and animals. Specific effect can include cancer, allergies and hypersensitivity, damage to the central and peripheral nervous

systems, reproductive disorders, and disruption of the immune system (Strict, 1981; Maroni, 1990).

Insecticides are the quickest and most pragmatic means of combating an infestation, but their uses have been restricted in many parts of Africa because of cost and their ability to contaminate the environment, leaving harmful residue in produce and induce resistance in pest species (Lale, 2002). According to Mvumi and Stathers (2003), there is a global drive towards reduction in pesticide use and eventual phasing out of synthetic based chemical grain protectants, articulated mainly by consumers and environmentalists who are concerned with health risks and environmental damage.

Huge quantities of grains are stored in Mubi each year and these grains are usually protected with synthetic pesticide as the farmers and traders alike awaits better price. It is equally a common practice that calendar application of such pesticides are widely practices by both farmers and traders, this particular practice is capable of increasing pesticide residue accumulation in such stored grains. Similarly, both groups have total disregard and in most cases ignorant on residues in grains even though cases of pesticide poisoning and even death are commonly associated with such usage. Therefore, there is need to study the presence of such residues to unearth their residue levels in some stored grains in Mubi.

The objectives of the study are: To assess damage by insect pest on grains protected with synthetic pesticides and to determine the presence of residues of some commonly used insecticides by farmers and traders in Mubi on stored grains.

MATERIALS AND METHODS

Experimental site

Grain samples for this research were collected at two (2) grain markets: new and old markets, Mubi North Local Government Area which lies on 10°16 N and 13°10 E. The markets are popular for grain storage and marketing.

Sample collection for residue analysis

Samples for the study were randomly selected. Three samples of 250g each from maize, cowpea, and Bambaranut were collected from the grains treated and stored for 1, 3, and 6 months for residue analysis in laboratory. Similarly, 250 g of non-treated grains of maize, cowpea, and bambaranut were collected and used as control for each category of 1, 3 and 6 months. The presence and levels of pifpaf (permethrin), cyclone (Aluminum phosphide) and Daskshi (Dichlovers) were determined using High Performance Chromatography technique of chemical analysis.

Extraction of insecticide

The insecticide fractions from the grains were extracted with acetonitrile and methanol (4:1). Powdered seed samples (2 g) were mixed with 25 ml of acetonitrile and metha-

anol. The mixtures were shaken vigorously and then allowed to stand for one hour. The supernatants were filtered using Whatmann number 4 filter paper. The extracts were transferred into a sterile bottle for chromatography analysis (Gesald, 2009).

High performance liquid chromatography (Hplc)

The extracted insecticides were analyzed by injecting 1 ml of each of the extract into bulk scientific Column BLC 10/11 high performance liquid chromatography with flame curization detector. The compound separated were detected and quantified from eluting chromatographic columns. The fact that all the stored grains have a trace of all the pesticide listed; the maximum residue level should not be exceeded. 1 ml of extract each of the grain was pipette into a small test tube. 3 ml of methanol-acetonitrile was added and swirl to mix. There after 1 ml of 0.05 M sodium phosphate buffer was added and swirled to mix. The sample loop of the HPLC was loaded with a 100 ul syringe and the sample was injected into a column and the peaks that come off the system were recorded and compared to a standard to identify and measure the compounds present (Gesald, 2009).

Damage assessment

One kilogram Samples each of maize, cowpea and Bambaranut grains were collected for insect pest damage assessment. The grains in the samples were separated into damaged and undamaged grains; percentage damaged was assessed as;

$$\frac{\text{Number of grains with hole in sample}}{\text{Total number of grains in sample}} \times 100$$

Assessment of grain weight loss:

This was computed using the FAO (1985) method of grain weight loss assessment;

$$\text{Percentage weight loss} = \frac{U_a N - [U + D]}{U_a N} \times 100$$

Where; U= Weight of undamaged grain.

D= Weight of insect damaged grain.

N= Total number of grains in sample.
 Ua= Average weight of one undamaged grain.

Data analysis and presentation

Data on damaged and weight loss in grains were analyzed using descriptive statistics (percentage).The data on residue analysis were presented by comparison to FAO/WHO units for residue levels measurement.

presented on Table 1.The result revealed that the highest average seed damage was recorded on Bambaranut with (16.83%), and lowest in maize (4.75%). The percentage weight loss is highest in cowpea (47.71%) and the least in maize (18.52%).

RESULTS

Damage and Weight loss Assessment

The result for the damage assessment is

Table 1: Damage Assessment on treated grains

Grain type seed	Percentage (%)Damage	Percentage (%) weight loss
Maize	04.75	18.52
Cowpea	13.71	47.71
Bambaranut	16.83	33.70

Measurement of pesticides residues in grains by isocratic HPLC procedure

The result for pesticide residue analysis in grains stored by traders for 1 month, 3 months and 6 months are presented on Table 2.The result indicates that all the grains have some levels of residues of all the commonly used pesticides in Mubi markets by the traders.

After storage for 1 month Dichlovers was highest in cowpea (30.10 mg/kg) and lowest in maize (21.00 mg/kg). The highest level of Aluminium phosphide (Cyclone) was found on cowpea (33.00 mg/kg) and the least on maize (20.10 mg/kg). Highest level of Permethrin (Pifpaf) was found in maize (38.40 mg/kg) and lowest in Bambaranut (26.50 mg/kg).

After 3 months of storage Dichlovers (D.D.force) was highest in cowpea (13.00 mg/kg), and lowest in Maize (10.20 mg/kg). Aluminium phosphide was found to have 14.10mg/kg in cowpea and reduces drastically to (9.20 mg/kg) in Maize. In case of Pifpaf, cowpea was found to have 12.00 mg/kg and the least was found in maize (8.72 mg/kg). Lastly, after 6 months of storage by the traders waiting for good price, Dichlovers (D.D.force) was highest (4.80 mg/kg) in cowpea and lowest (2.31 mg/kg) in Maize. Similarly, Aluminium phosphide (Cyclone) in Bambaranut was the highest and the least level were found in cowpea (3.00 mg/kg); Permethrin (Pifpaf) level of 3.00 mg/kg in cowpea was higher than those of Bambaranut (2.60 mg/kg) and maize (2.11 mg/kg).

Table 2: Level of Residue of Extracted Insecticides

Age of Sample Grains	Dichlovers(Daskshi) mg/kg	Aluminium phosphate (Cyclone) mg/kg	Pifpaf (Permethrin) mg/kg
1Month			
Cowpea	30.10	33.00	28.40
Bambaranut	28.52	31.20	26.50
Maize	21.00	20.10	38.40
Control	ND	ND	ND
3Months			
Cowpea	13.00	14.10	12.00
Bambaranut	11.20	23.10	11.10
Maize	10.20	9.20	8.72
Control	ND	ND	ND
6Months			
Cowpea	2.80	1.10	2.20
Bambaranut	1.00	2.30	1.60
Maize	1.31	1.04	1.11
Control	ND	ND	ND

N.D = Not Detected

DISCUSSION

Damage assessment

Results in Table1 indicate that percentage damage is higher in Bambaranut (16.83%) than Cowpea (13.71%) and Maize (04.75 %). Losses resulting from insect infestations have been reported to be widespread and involve more than loss of quality. Damaged kernels are of lighter weight and result in discounts when marketed. Insect infestation also causes a reduction in nutrients in the grain. (Anon., 2008). This will mean that farmers/traders in Mubi have been experiencing several kinds of damage losses of economic magnitude due to pest infestation. This is so because infestation generally results in dissatisfied customers and related marketing problems that develop from a poor reputation in marketing channels (Owusu, 2007). The most unfortunate con-

sequence of not managing grain properly is the loss of money, time, and effort to produce the grain (i.e. seed, fertilizer, field pest management, harvesting) (Udo, 2000). Similarly, studies elsewhere revealed that the percentage weight loss of the stored grain Bambaranut (33.70%) is higher than that of Cowpea (47.71%) and Maize (18.52%) resulting in a corresponding loss in moisture content and dry matter in both soybeans and in corn over time (Lazzari, 1988). This will mean that greater losses occur in the studied markets; these therefore may pose a serious food security challenges to Mubi and other states or communities that depend on the market for some supply of their needed grains.

Pesticide residue analysis.

Dichlovers (Daskshi)

In 1 month of storage the level of Dichlo-

vors (Daskshi) was highest in cowpea (30.10 mg/kg) followed by Bambaranut (28.52 mg/kg) and lastly Maize (21.00 mg/kg). All the above results are above the FAO (1993) reported level of no-observed adverse effect level or Available Daily Intake (ADI) of 0.04 mg/kg/day. Gillette (1982) reported that children who chewed DDVP-containing resin strip at the above FAO levels, but without clinical symptoms. Hayes (1982), also reported that ingestion of (400 mg /kg) DDVP was sub-lethal to woman who survived narrowly after intensive treatment.

Similarly, Goel and Aggarwal (2007) reported a result of an experiment for the use of Dichlovors as an anti-helminthic using one hundred and eight (108) patients with infection of helminthes. The patients were treated with DDVP in a granular –resin formulation and found DDVP to be a broad – spectrum antihelminthic agent. The patients were divided into two groups of 24 subjects (14 males & 10 females) treated with a dose of 6 mg/kg/day and 84 subjects (37 males & 47 female) at a higher dose of 12 mg/kg/day. The only clinical sign reported was brief mild headaches, diarrhea, and vomiting in few of them. However, this could be due to detoxification of compounds through washing and drying.

The result in the 3rd month of storage indicate cowpea (13.00 mg/kg) having the highest residue followed by Bambaranut (11.20 mg/kg) and lastly maize (10.20 mg/kg) in the case of Dichlovors. It shows the reduction in the level of residue due to volatilization and possible biodegradable processes (Palikhe 2001). After 6 months of storage there was drastic reduction in the level of residue in the stored grains, with cowpea (2.80 mg/kg) followed by Bambaranut (1.00

mg/kg) and lastly Maize (1.31 mg/kg). All results are above the WHO and FAO ADI for Dichlovors. Consumption of Dichlovors of above ADI has been reported to cause organ failure, headache, and diarrhea (FAO/WHO, 1986). Earlier detoxification of these compound are possible of the residues in mg/kg in the stored grains further before consumption may further reduce due to volatilization , processing hydrolysis, photo-degradation and chemical degradation to the ADI levels. As further more processing, for utilization will reduce the level of insecticide in grains (Gasald, 2009).

Aluminium phosphide (cyclone)

The result from the stored grain analysis (Table 2), with respect to Aluminium Phosphide in the 1 month of direct application indicates that cowpea (33.00 mg/kg) followed by Bambaranut (31.20 mg/kg) and Maize (20.10 mg/kg). Chugh *et al.* (1996) reported that Aluminium phosphide is a toxic material which is attributed to the liberation of phosphate gas, a cytotoxic compound that causes free radial mediate injury. In the 3rd month of storage, there was a slight reduction in the residue level. Bambaranut (23.10 mg/kg) was higher than cowpea (14.10 mg/kg) and Maize (9.20 mg/kg). The major lethal consequences of Aluminium Phosphide ingestion i.e. profound circulation collapse is reported as secondary effect of these toxics generated, which will lead to direct effect on cardial myocytes (Chugh *et al.*, 1996). FAO/WHO (1986) reported Aluminium phosphide poisoning is the most common cause of suicidal death in India. Similarly, after 6 months of storage, Aluminium Phosphide level in Bambaranut (2.30 mg/kg) is higher than cowpea (1.10 mg/kg) and maize (1.04 mg/kg). All the above results are evidently above FAO/ADI of (0.019 mg/kg/day), for Aluminium

phosphide. However, the observed level may yet reduce before consumption; further processing of product under utilization will reduce insecticide in stored grain. Goel and Aggarwal (2007) reported that after ingestion of toxic phosphate gases, symptoms usually develop within few minutes; common clinical symptoms in poisoning cases are nausea, repeated vomiting, headache, abdominal discomfort or pains and tachycardia. Most of these symptoms were reported by the traders in this study.

Permethrin (Pifpaf)

In the 1 month of storage Pifpaf was found to be higher in Maize (38.40 mg/kg) than Cowpea (28.40 mg/kg) and Bambaranut (26.50 mg/kg). Residue level of this product in non-food commodity were reported to be generally higher up to (20 mg/kg). GI-FAP (1987), reported that food of animal origin safe residue level range from (0.01 & 0.2 mg/kg). However, the absorption and elimination of Pifpaf is reported to be rapid in the different mammalian species tested (IRPTC, 1985). After 3 months of storage, the residue in grain was lower than what was found in the grain initially. Cowpea (12.00 mg/kg) is higher than Bambaranut (11.10 mg/kg) and Maize (8.72 mg/kg). IRPTC (1985), reported the major metabolic reaction due to Pifpaf to be cleavage of the esters bond followed hydroxylation and conjugation of the cyclopropane and phenoxybenzyl moieties. This will lessen the adverse effect of poisoning with this product. Similarly, the result of 6 months storage analysis indicates that Pifpaf is higher in Cowpea (2.20 mg/kg) followed by Bambaranut (1.60 mg/kg) and Maize (1.11 mg/kg). IRPTC (1985) reported that the acute toxicity of Pifpaf for mammals is of moderate order, because the highest levels of Pifpaf are found in the body fats, which is

consistently with the lipophilic nature of compound; also the oral LD₅₀ for the rat ranged from 200-400 mg/kg body weight. Short-term and long-term toxicity studies on rat, mice and dog have shown effects on growth, liver, kidney and nervous system. A no-observed adverse effect level of 7.5 mg/kg body weight has been adopted (IRPTC, 1985).

The levels observed in this study are still above the critical levels for safe consumption set by FAO and WHO. This suggests that product treated with Pifpaf will still need storage beyond 6 months before they can be safely consumed or there is need for further treatment to be carried out before safe consumption could be guaranteed.

CONCLUSION AND RECOMMENDATIONS

Pesticides are the potential health hazards, which have drawn attention of food quality control agencies, certification bodies, international community and trading partners. Due to the lack of regular monitoring scheme of Maximum Residue Level (MRL) on pesticides, it has affected the export and other food commodities in the recent years. Similarly, the certification of organic foods has faced problem due to the absence of this monitoring scheme.

For the improvement in present situation regarding the pesticide use, pest resistant to pesticides, environmental contamination, and health hazard to farmers as well as the consumers from the toxic chemicals, the following recommendations are suggested. Laboratory capacities and facilities should be strengthened in order to cope with the newly emerged challenges of contaminants as well as harmonization of analytical methods for reliability of the results. The gap between the

availability and the requirement is very wide, which demands the exchange visits, training and other academic opportunities in the field of food safety and quality as well as laboratory analysis.

In order to protect the consumers against the biological, chemical, and environmental contamination of food, the food safety should encompass the control over all stages of food production, processing and distribution of foods. There is need of development of the ability to follow the movement of food through specified stages of production, processing and distribution. Scientific evaluation of potential adverse effects resulting from human exposure to food borne hazards, Risk analysis is relatively new applied science based on sound scientific footing to protect consumer's health by Appropriate Level of Protection (ALOP). Necessary data and information for the food standardization should be base on risk analysis of particular food commodity.

Export potential products have been reduced due to pesticide residues and other inorganic residues that come from agricultural practice (FAO/WHO, 2005). So horizontal standards of pesticide residues should be enforced in the agro products especially those for exportable products to facilitate trade as well as for the production of safe food.

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