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Full Length Research Paper

Dichlorvos concentrations in locally formulated pesticide (*Ota-piapia*) utilized in northeastern Nigeria

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Samples of locally formulated *Ota-piapia* utilized in northeastern Nigeria were investigated for concentration levels of dichlorvos aimed at specifying the preponderant active pesticide ingredient in the local formulation from this region. Gas chromatography-Flame Ionization detection (GC-FID) technique was used for the determination. Results indicated that all *Ota-piapia* samples studied showed presences of dichlorvos. The frequency distribution curve of concentration revealed 64% of total sample concentration cluster around the modal region of 7.7% w/v of dichlorvos. Though content uniformity was very poor with highest variability at mid concentration (5 - 10% w/v), but dichlorvos can safely be confirmed as the preponderant active pesticide ingredient in the local formulation of *Ota-piapia* from this region. The linearity of the method was good with determination coefficients of > 0.996 for target analyte. Therefore it is a matter of public health significance to regularly monitor pesticide residues in foods and humans in order to assess the food safety risk and population exposure to pesticides. The illegal formulation of *Ota-piapia* in this region by traders with little or no knowledge of public health policy must be checked through adequate control of the trade, use of pesticides and the enforcement of appropriate sanctions.

Key words: Organophosphate, pesticide, local formulation, concentration levels, GC-FID.

INTRODUCTION

Reputable reports assert that *Ota-piapia* is an unspecified insecticide/pesticide and its application regarded as a dangerous practice since its chemical constituent is unknown (FAO, 2001; PAN, 2007; Akunyili, 2007). Thus, this work is aimed at determining the concentration levels of dichlorvos in the locally formulated *Ota-piapia* utilized in northeastern Nigeria and thereby specify the preponderant active pesticide ingredient in the local formulation from this region.

Dichlorvos (Figure 1), also known as DDVP (O.-O-dimethyl-O-2, 2-dichloro-vinyl phosphate) (USEPA, 2007) is an organophosphate insecticide and have been applied in northern Nigeria as mosquitoes insecticides over the decades (Foll et al., 1965; Foll and Pant, 1966) since its commercial manufacture started in 1961 (BCERF, 1999). A number of evidences indicate that dichlorvos is most likely the major active pesticide ingredient of *Ota-piapia* formulation in northeastern Nigeria. These consist of the fact that generally, dichlorvos is used as an agricultural insecticide on crops, stored products, and animals. It is used as an insecticide for slow release on pest-strips for pest control in homes. It is also used as an anthelmintic (worming agent) for dogs, swine, and horses, as a botacide; agent that kills fly larvae (USEPA, 1994), the later, being a major menace in northeastern Nigeria and hence the observed large tonnage of various brands of dichlorvos in the open market.

Ota-piapia is a vernacular (Igbo) of eastern Nigeria origin and a household name for insecticide/ rodenticide/pesticide, which literally translates into "that which completely consumes/devours". Local pesticide makers emphasized the potency of their pesticides by the word "*Ota-piapia"* indicating that such products will completely take care of our little pest problem (Mortui, 2006). Its acceptance and wide spread proliferation in Nigeria have been due solely to its cheap production, efficacy, accessibility and affordability (Essiet, 2009). The product is still not registered with NAFDAC (Akunyili, 2007), but

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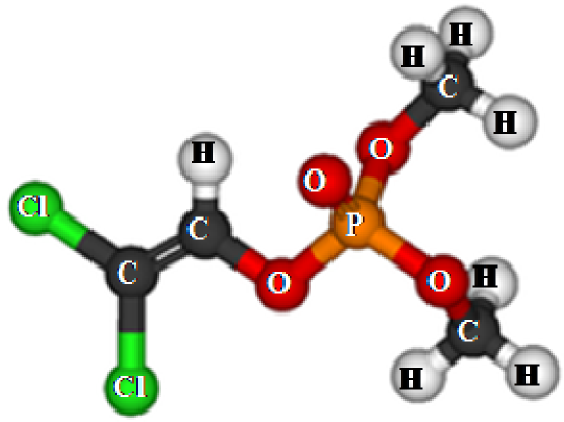

Figure 1. Chemical Formula of Dichlorvos (CAS # 62-73-7).

have been commonly used as insecticide, especially mosquitoes (Foll et al., 1965), food storage, such as grains and preventing insect infestation on fish (Vardell et al, 1973; Desmarchelier, 1977; FAO, 2001). This is the trend of application in northeastern Nigeria.

The local formulation of *Ota-piapia* is thought to entails repackaging into a small (about 10 - 15 mL) retail bottle of an active ingredient which is unspecified pesticides from those imported, which include cypermethrin, *dichlorvos*, gammalin 20, gammalin super, lindane, capsitox 20 (PAN, 2007). Some may contain homemade cocktail of kerosene, oil, alcohol and any suitable solvent with the pesticide.

Ota-piapia had caused the death of so many Nigerian families in recent times (Olebunne, 2009) and worldwide (USEPA, 2007), specifically through food contamination (Akunyili, 2007). Children are especially prone to accidental poisoning of this product (Okeniyi and Lawal, 2007). Therefore they have been call for a complete ban and eradication of all streets hawking of locally adulterated, unregistered, unlabelled, repackaged, uncertified and expired chemical pesticides in the form such as "Otapiapia" among others (Maduako, 2009).

Detailed risk characterization of dichlorvos has been well documented in CEPA (1996), its toxicological profile in ATSDR (1997) and environmental assessment in APVMA (2008).

Dichlorvos specifically inhibits cholinesterase enzymes. It is poisonous if swallowed, inhaled, or absorbed through the skin. Most standard industrial product indicates safety information which is not adhered to: "Wear protective gloves, clean protective clothing, goggles, and a respirator of the organic-vapour type when handling this material. Avoid prolonged exposure to fumes. Wash hands and exposed skin after handling and before eating, and bathe immediately after work. Keep the material out of the reach of children and well away from foodstuffs, animal feed and their containers. Ensure that containers are tightly sealed, and stored and disposed of in such a way as to prevent accidental contact. In case of contact, immediately remove contaminated clothing and wash the skin thoroughly with soap and water; for eyes, flush with water for 15 min. If poisoning occurs, call a physician. Atropine and pralidoxime are specific antidotes and artificial respiration may be needed" (WHO, 1999, Gupta, 2006).

Table 1. GC Temperature programme.

Rate	Temperature (°C)	Hold time (min)
-	1.00	80.0
10.0	1.00	120.0
35.0	12.00	280.0
45.0	2.00	290.0

MATERIALS AND METHODS

Samples and sampling

The *Ota-piapia* samples were purchased at random from the Baga and Maiduguri fish markets from August to December, 2008. A total of 100 samples were collected during this period.

Sample preparations and analysis

The preparation and analysis of *ota-piapia* samples were carried out in accordance with methods described in the AOAC (1996) and WHO (1999). For each sample of *Ota-piapia,* 0.01 g was weighed into a 100 mL volumetric flask and made up to volume with acetone. This was serially diluted with acetone to obtain solutions of about 1.0 mg/mL concentration. The exact concentrations of the final solutions were read directly from the calibration curve and automatically related to the content of the original preparation using appropriate dilution factors.

Standard solutions of dichlorvos at 3 levels (0.5, 1.0 and 2.0 mg/mL) were analyzed to obtain a calibration curve using Rtx-14% cyanopropylphenyl-86% dimethyl polysiloxane, 30 M x 053 mm id x 1 µm df column. The linearity of the flame ionization detector (FID) and the relative standard deviation (RSD) for 6 replicate injections were checked prior to each analysis. Determination was carried out using an automated *Shimadzu* Gas chromatography (GC-17A) equipped with fused-silica capillary column. The analyte identification was confirmed on a second column of differing polarity from the analytical column, and using a different set of chromatographic conditions. The chromatographic conditions used for analysis consists: The inlet head pressure = 16 psi, injector temperature = 270°C and the detector temperature = 290°C. The carrier gas was nitrogen (N_2) at a flow rate 165 ml/min. The initial oven temperature was 80°C and ramped as indicated on Table 1.

Data analysis

Results of the analysis were statistically analysed using coupled Microsoft Excel + Analyse-it v. 2.10 (Analyse-it®, 2007) software. Variations were considered significant at $p < 0.05$.

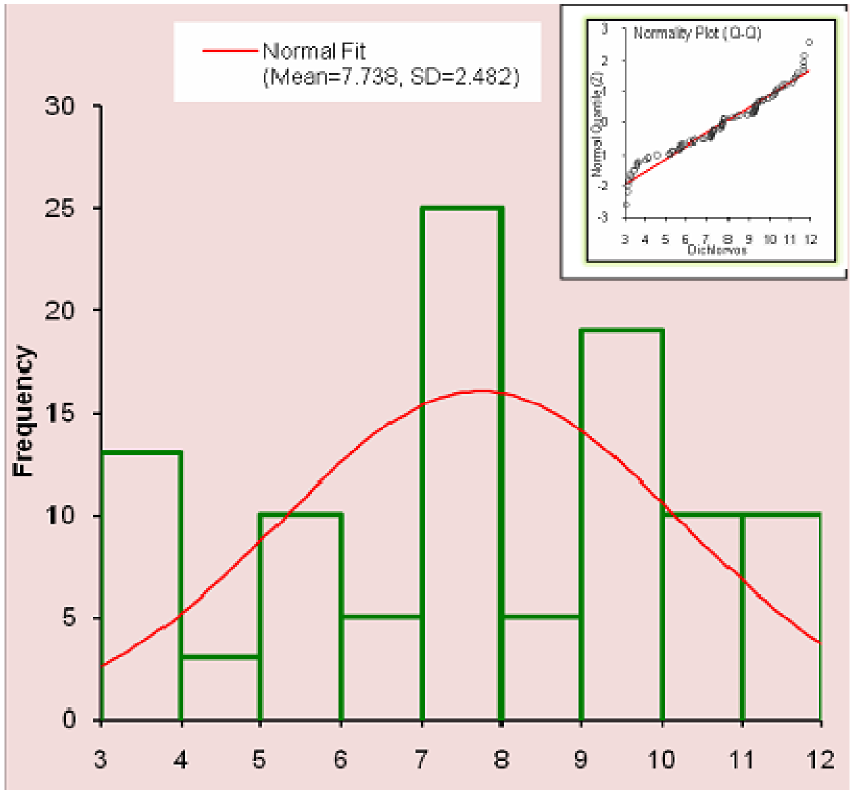

Figure 2. Frequency distribution of dichlorvos concentrations %w/v in *Ota-piapia* samples.

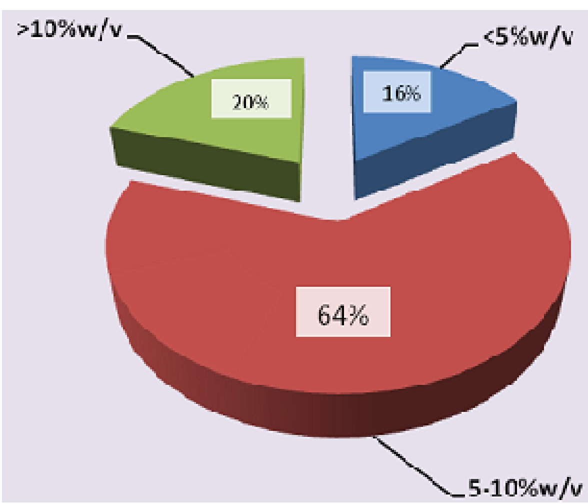

Figure 3. Percentage of distribution range of dichlorvos in *Ota-piapia* samples.

RESULTS AND DISCUSSION

The summary of descriptive statistics for dichlorvos concentrations in *Ota-piapia* samples is shown on Figure 2. All samples indicated the presence of dichlorvos. The linearity of the method was good with determination coefficients of > 0.996 for the target analyte. The variation in concentration between the minimum and maximum is very wide, but most concentrations were, from the frequency distribution curve found to be highly clustered around the modal region of 7.7% w/v. This concentration was statistically significant ($p < 0.05$) following the Shapiro-Wilk's test for normality and shows a normal distribution from a single population. From the frequency distribution curve (Figure 2) the maximum content of dichlorvos found in the samples contained 41.1% more dichlorvos than the mean. The minimum content was 61.1% of the mean sample concentration. This variation between the concentrations was significant ($p < 0.05$), and shows the lack of uniformity of product content. The normal dichlorvos concentration for use in household pest control products is 1.0% (WHO, 1997).

However, when the entire concentration range was sub-divided into low (<5% w/v), medium (5 - 10% w/v) and high (>10% w/v) as shown on Figure 3, it was seen that the medium concentration range presented the highest of about 64%; of this, 70% was contributed by the modal concentration.

All the *Ota-piapia* samples analyzed in this work were inadequately labelled. The samples had 'trade' names but without any information on identity and content of active ingredient as required by standard labelling regulations. Thus, there was neither an assumed active ingredient to

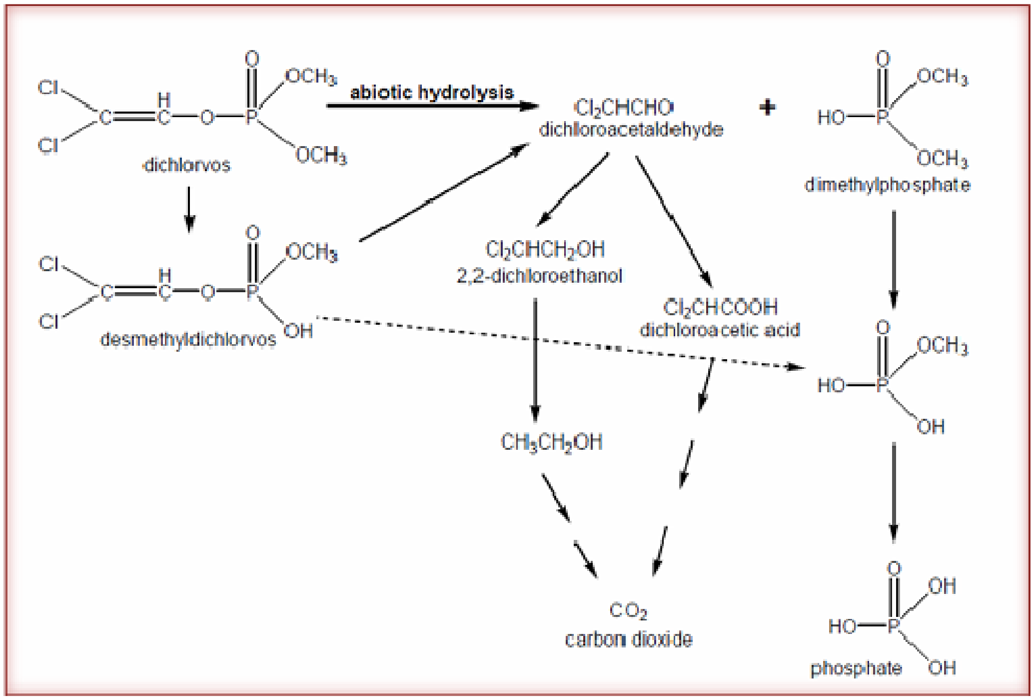

Figure 4. Proposed pathway for the breakdown of dichlorvos in soil and in water/sediment systems, including abiotic hydrolysis and microbial degradation steps (APVMA, 2008).

identify in the formulations nor a labelled claim to verify for quality compliance. However, all the sample runs revealed the presence of single peaks in the chromatograms which had the same retention time (8.79 min) as the peak of dichlorvos standard. In this work we did not establish any fake *Ota-piapia* formulations in the market; all the samples contained the same active ingredient, dichlorvos. However, it is likely that the remaining percentage may contain other pesticide.

This finding was however not unexpected since petty traders involved in the sale of public health pesticide products only involuntarily react to the dynamics of the market by introducing all types of locally produced formulations completely unaware of quality control requirements. Consequently, such inconsistently formulated insecticides could leave excessive residue levels in raw agricultural products if used for food preservation or on foods sold in the local market too soon after treatment with pesticide. These have been the common problems with pesticide use in developing countries (Akerblom and Cox, 1996). The illegal use of chemical insecticides to preserve dry fish in remote coastal isolated islands and in inland depressions which lack chilling and freezing facilities have been reported (Eyo and Mdaihli, 1997; Bhuiyan et al., 2008), but the use of insecticides directly on fish for is not recommended by Codex (2009). The application of *Ota-piapia*, an unspecified insecticide to prevent insect infestation of fish is still remains common practices (Eyo and Mdaihli, 1997; FAO, 2001).

Organophosphate insecticides are generally chemically unstable with short half lives, and are easily biodegradable into harmless substances which do not pose any serious environmental problems when compared to organochlorines (Manahan, 2005). A proposed pathway for the breakdown of dichlorvos in soil and in water/sediment systems, including abiotic hydrolysis and microbial degradation steps is shown on Figure 4 (APVMA, 2008). Other researchers have indicated the predictive models of decay of dichlorvos residues in stored grain ((Vardell et al, 1973; Desmarchelier, 1977), photocatalytic degradation using two different photocatalysts (TiO_2 and ZnO) (Evgenidou et al., 2005; Rahman and Muneer, 2005), photosensitized degradation under UV irradiation (Oancea, 2008) and ^{31}P-NMR degradation study by Benoit-Marqui et al. (2004).

Generally, exposure to organophosphate pesticides is associated with numerous neurological effects including an acute cholinergic episode and an induced delayed neuropathy (Jamal et al. 2002; Salvi et al. 2003; Costa, 2006). The evidence of chronic neurological effects from long-term low-level exposure in the literature is however contradictory, inconsistent, and inconclusive (Lotti 2002; Kamel and Hoppin 2004). Some studies support evidence of effects (Jamal et al. 2002), while other studies noted only subtle or no effects (Lotti 2002). In a number of studies that investigated possible links between neurological effects and long-term exposures to organophosphates (Pilkington et al., 2001; Alavanja et al., 2004) differences in exposure assessment methodology, effects measurement protocols, and endpoints precluded a conclusive statement regarding the relationship between long-term low exposure to organophosphates and neurological effects (Parsons et al., 2005).

However, there was concern about the vulnerability of the developing human brain to any neurotoxic pesticides (NRC, 1993) because developmental neurotoxicity has implications for many of the cognitive diseases of childhood, such as learning disabilities, dyslexia, mental retardation, attention deficit disorder, and autism (Landrigan et al., 2004), some of which appear to be increasing in prevalence, as well as a role in the neurodegenerative diseases of old age like Parkinson's and Alzheimer's (Allam et al., 2005).

A number of animal studies have reported reproductive effects such as premature ovulation, endocrine disruption, and congenital abnormalities from exposure to organophosphates (Akingbemi and Hardy, 2001; Garcia et al., 2003; Hanke and Jurewicz, 2004), estrogenic and antiandrogenic effects (Tamura et al., 2001) while other studies do not (Chen et al., 2002). Studies on the carcinogenicity of pesticides in long-lived humans have provided evidence that exposure to cholinesterase-inhibiting compounds might be linked to certain lymphatic and blood cancers (Parsons et al., 2005). However, very little has been published regarding the risk of developing leukaemia after exposure to organophosphates. A weak positive association was reported for leukaemia and farming (Keller-Byrne et al., 1995).

In one study elevated risks for leukaemia subtypes with exposure to organophosphates and carbamates over time was reported (Brown et al. 1990). In general, risk for leukaemia was elevated for workers who first used dichlorvos more than 20 years prior to the study (Brown et al. 1990). In another study involving enclosed-space pesticide applicators in Minnesota, two out of three subjects with leukaemia had hairy cell leukaemia, a rare hemeopetic cancer. Thus, the toxicology of organophosphates has become increasingly of concern because exposure to neurotoxic compounds at levels believed to be safe for adults could result in permanent loss of brain function if it occurred during the prenatal and early childhood period of brain development (Rees et al., 1990; NRC, 1993). The genotoxicity and breast cancer of dichlorvos have been reported by Kathryn et al., (2006), Patel et al., (2007), Remington et al., (2008); BCERF (1999).

In conclusion, the finding in this work revealed that products are not well labelled. Though content uniformity was very poor with highest variability at mid concentration (5-10 %w/v), dichlorvos can safely be confirmed as the preponderant active pesticide ingredient in the local formulation of *Ota-piapia* from this region. Therefore it is a matter of public health significance to regularly monitor pesticide residues in foods and humans in order to assess the food safety risk and population exposure to pesticides. The illegal production and use of *Ota-piapia* by traders and consumers with little or no knowledge of public health policy must be checked through adequate control of the trade and the enforcement of appropriate sanctions.

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