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Organochlorine Insecticide Residues in Cattle Feed

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The role of organochlorine insecticides (OCIs) has been very vital in public health and agricultural production in developing countries including India. Despite the restricted use and/or banning of these compounds, several reports indicate that pollution with OCIs still exists, and may be of public and environmental health significance even in developed countries (Kim 1984; Sawhney and Hankin 1985; Brunn et al. 1985; Rogan et al. 1986). Milk and milk products play a central role in human nutrition. The OCIs are highly lipophilic and easily get accumulated in fat- rich milk and milk products, animal meats, etc (Kaphalia et al. 1981; Richard and Dulley 1983; Takroo et al. 1985; Pines et al. 1988). Animal feed, feed mixtures and fodder grasses have been the major source of contamination (Duggan 1968; Kaphalia and Seth, 1982; Pierson et al. 1982; Waliszewski et al. 1985). This paper describes the preliminary observations about the presence of OCIs in commercial cattle feed in India.

MATERIALS AND METHODS

Analytical standards for OCIs were obtained from U.S.F.P.A., Pesticides and Industrial Chemical Repository (MD-8), Research Triangle Park, NC, USA. All solvents used in extraction and cleanup process were of A.R. Grade and distilled in all glass still prior to use. Florisil (Fluka, A.G.), activated charcoal (E. Merck India Ltd.), Sodium sulphate anhydrous (Sarabhai Chemical Bombay) used in this experiment, were of high purity.

A total of 32 samples collected randomly from the local market were used for this analysis. Each sample of

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feed in triplicate was weighed (25 gm), blended for 3 min with 150 ml double distilled acetonitrile in high speed homogenizer. The mixture was filtered with suction through a Whatman No. 1 filter paper. The generation head and jar was washed with 25 ml acetonitrile. The filterate was transfered to a 1L separatory funnel. 450 ml water containing sodium sulphate was added to the separatory funnel and shaken vigorously for 3 min. Extraction was carried out consecutively with 100, 50 and 50 ml portions of n-hexane, shaking each time for 3 min. The extracts were filtered through 30 g anhydrous sodium sulphate under high pressure. The sodium sulphate layer was rinsed with 20 ml n-hexane. The combined hexane extract were concentrated to 1 ml volume with vacuum rotary evaporator at 35°C, dissolved in 25 ml distilled acetonitrile and washed with 4% sodium sulphate. It was extracted again with 25 ml n-hexane and filtered through 30 g anhydrous sodium sulfate layer. The sodium sulfate layer was washed again with 10 ml portion of n-hexane. The combined hexane extracts was concentrated to 1 ml volume. The mixed phase column was dry packed with 1 x 5 cm bed of anhydrous sodium sulfate at the bottom followed by 1 x 10 cm bed of 2% deactivated Florisil (Fluka A.G.), 1 x 10 cm layer of activated charcoal (E. Merck, India, Ltd.), 1 x 10 cm layer of silica gel (Glindia Ltd.) and approximately 1 x 5 cm bed of anhydrous sodium sulfate layer at the top. The concentrated extract was then applied to the pre-washed chromography column with 40 ml of n-hexane to remove other contaminants (Picer et al. 1978). The fractions containing organochlorine insecticides were collected, concentrated and transferred to a 10 ml volumetric flask and made to volume of 10 ml in n-hexane. Analysis was carried out by using a Varian Aerograph series 2400 equipped with a 3 H electron capture detector. A glass column (1 x 5m x 2 mm i.d.), packed with 1.5% OV-17 + 1.95% OF-1 on 100-120 mesh Chromosorb WHP was used. Operation temperatures were 195, 200 and 220°C for column, injecter, and detector respectively. Purified nitrogen gas passing through silica gel (8 x 10^{-9} °A) and molecular seives (2 x 10^{-9} °A) was used as a carrier gas at a flow rate of 60 ml/min. Data for GLC analyses were further confirmed by TLC.

Procedural blanks, consisting of all reagents and glass ware used during the analysis, were periodically determined to check the cross contamination. Since no compound that interferred with OCIs was detected, the sample values were not corrected for procedural blanks. Recovery studies with fortified sample have indicated that overall recovery values exceeded 95% except for beta-HCH 81%. Results were not adjusted for percent

recovery. Identification and quantification were accomplished using a known amount of external standards (AOAC, 1984).

RESULTS AND DISCUSSION

The concentrations of OCIs in cattle feed samples are summerized in Table 1. Presence of alpha, beta and

Table 1. Concentration of organochlorine residues (ppm) in cattle feed

OCIs	This report	Kaphalia & Seth 1982
	@	
alpha-HCH	0.181 (0.140-0.285)	-
beta -HCH	0.085 (0.075-0.099)	-
gamma-HCH	0.127 (0.110-0.148)	0.107 (0.083-0.139)
HCH (Total)	0.393 (0.332-0.497)	0.373 (0.313-0.413)
pp'-DDE	0.068 (0.056-0.081)	0.049 (0.033-0.070)
op'-DDT	0.027 (0.025-0.029)	0.051 (0.044-0.074)
pp'-DDD	0.077 (0.062-0.098)	0.038 (0.032-0.046)
pp'-DDT	0.064 (0.055-0.076)	0.151 (0.121-0.214)
DDT (Total)	0.236 (0.197-0.283)	0.301 (0.248-0.414)
Aldrin	0.156 (0.140-0.202)	-
Endrin	0.020 (0.013-0.027)	-
Endosulfan I	0.029 (0.028-0.031)	-

@Indicate the respective mean values
Values under parenthesis indicate range

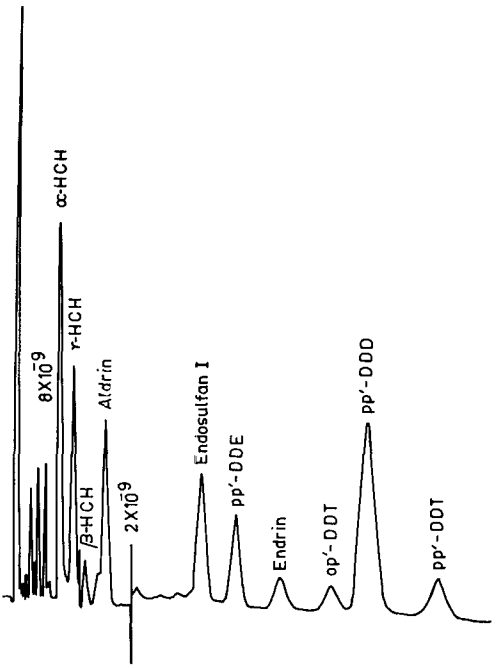

Figure 1. Characteristic chromatogram of OCIs present in cattle feed.

gamma-isomers of HCH were found in all the samples subjected to GLC analysis. The mean concentration of HCH (Total) was 0.393 ppm which was more than 3 fold higher than the gamma -HCH. Residues of pp'-DDE, op'-DDT, pp'-DDD and pp'-DDT were also present in all the samples. The total DDT content was 0.236 ppm which suggested its persistence even in animal feed samples. Since the transported DDT is known to undergo metabolic conversion and dehydrochlorination (Matsumura, 1973), presence of pp'-DDE and pp'-DDD encountered in this study might be due to such metabolic processes. The mean concentration of pp'-DDE and pp'-DDD were higher than those of DDT isomers (pp'-DDT and op'-DDT). The concentration of pp'-DDE and pp'-DDD in the feed were found to be comparable. The level of aldrin was found to be 0.156 ppm. Endrin, one of the most toxic OCIs was also present (0.02 ppm) in all the feed samples alongwith Endosulfan I (0.029 ppm) (Figure 1).

Entry of OCIs to human body is through contamination of food and feed from accidential and/or incidental exposures. Dietary intake of various food items such as meat, fish,oils and fats, milk and milk products have substantially contributed to the higher accumulation of

OCIs in the body (Dale et al. 1965). The uses of HCH in India is increasing while there has been a tremendous decline in the consumption of DDT and other chlorinated insecticides over the last few years (Pesticide Information, 1986). This may be one of the reasons for decrease in the concentration of DDT (Total) and an increase in the concentration of HCH (Total) recorded in the present study.

The levels of total HCH (0.393 ppm) and DDT (0.230 ppm) in cattle feed are in agreeement with those of an earlier report (Kaphalia and Seth, 1982). The residue of HCH in paddy straw was found to be higher (110 ppm). In contrast the residues of HCH in paddy straw at harvest and postharvest were found to be 33.14 (120 days of crop), 16.95 (165 days of crop) 11.0 ppm (25 days after harvest) respectively. It has been advocated that cattle feed contaminated with high residues of OCI should be consumed after a several month waiting period (Murthy et al. 1980).

This study calls for a continuous monitoring program for animal feed, mixtures of feed and fodder grasses at different agrarian locations in the country to understand the national scenario with particular reference to OCI contamination of animal feed.

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