

Assessment of the Losses of DDT and HCH Residues in Wheat Flour During *Chapati* Making

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The widespread contamination of wheat grains and flour with DDT and HCH has been reported from India (BINDRA and KALRA 1973, JOIA *et al.* 1977). In the Indian sub-continent, however, wheat flour is generally processed into chapatis before consumption. In view of the fact that preparation or processing techniques may have considerable effect on residues in raw food material, it is important to know the effect of chapati making on DDT and HCH residues in wheat flour. Earlier, MUKHERJEE *et al.* (1973) reported 90 and 95 per cent loss of DDT and lindane respectively during the processing of wheat flour into chapatis. However, the wheat flour used by them was fortified with these insecticides at 100 ppm, only 4 h before the processing. These conditions were not representative of the situation in the actual samples. Further, there is no information available on the fate of technical HCH residues either during chapati or bread making. The present study, therefore, reports the results of the investigations on the effect of chapati making on DDT and technical HCH residues in the actual market samples of contaminated wheat flour.

MATERIAL AND METHODS

Preparation of chapatis and extraction of insecticides residues: Two market samples of contaminated wheat flour weighing one kg each were selected during a survey of this commodity in the Punjab. Half of the samples was employed for making chapatis by the indigenous method. For this purpose, the flour was kneaded into dough with 70 per cent of its weight of water and then baked into chapatis on a hot iron plate. The baking of one chapati took about 5 min. The chapatis were cooled to room temperature and cut into small pieces. Representative sub-samples weighing 50g each were drawn in triplicate. These were ground with an equal weight of sodium sulphate and extracted with 250 mL of hexane-acetone (3:1, v/v) in a Soxhlet apparatus for 12 h. Wheat flour samples in triplicate were also extracted likewise.

Cleanup: The extract was washed with 100 mL of 2 per cent aqueous NaCl solution. The n-hexane phase, thus obtained, was partitioned thrice into acetonitrile. The acetonitrile fraction after dilution with 5 volumes of 2 per cent aqueous NaCl solution was extracted thrice with petroleum ether. The

petroleum ether extract was concentrated to about 5 mL and then cleaned up by using silica gel column chromatography. Silica gel thoroughly washed with acetone and methanol was activated at 130°C for one h, and then packed in a glass column (50 cm x 2 cm diam.) to a height of 10 cm in between two layers of sodium sulphate. The column was pre-washed with 100 mL hexane and the concentrated sample extract was then added to the column. The elution was first done with 100 mL of 2 per cent benzene in n-hexane, this fraction contained primarily p,p'-DDT and o,p'-DDT. The column was then eluted with 100 mL of 40 per cent benzene in n-hexane, this fraction contained isomers of HCH. The eluates were concentrated to small volumes and analysed by gas-liquid chromatography (glc) and thin-layer chromatography (tlc).

Analysis: Appropriate aliquots were injected into a Packard Gas-Chromatograph (Model 7624) equipped with tritium foil electron-capture detector. A pyrex glass column (1.84 metres long and 3.2 mm id) packed with 5 per cent DC-200 on Gas Chrom Q was used. Detector, injector and column temperatures were 200, 210, and 190°C respectively. Nitrogen carrier Gas flow was 80 mL/min.

Under these operating condition, the retention time of p,p'-DDT, o,p'-DDT and p,p'-DDE were found to be 24, 22 and 20 min, respectively. Technical HCH on this column resolved into two peaks corresponding to alpha and gamma isomers with retention time of 2.5 and 3.0 min, respectively. The quantitative estimation was done by comparing with the peak height of the standards run under parallel conditions. Average recoveries from the samples fortified at the levels of 0.1 and 0.5 ppm were 85 and 82 per cent in the case of DDT and HCH respectively. The results obtained were expressed as such and not corrected for recovery. The extraction efficiency of the method was also checked by refluxing the residual chapati/flour material after Soxhlet extraction in acetonitrile and acidic methanol. The extracts, after necessary cleanup when analysed, showed only traces of DDT and HCH.

The nature of the residues was further confirmed by tlc following essentially the method given by THOMPSON et al. (1970) on glass plates coated with alumina-G containing silver nitrate using n-hexane as the developing solvent.

Moisture content of chapatis and flour was determined separately by heating 50 g of the samples at 110°C to constant weight.

RESULTS AND DISCUSSION

The levels of DDT and HCH residues found in the samples of wheat flour and chapatis are given in Table 1. These values have been expressed both on fresh and dry weight basis as the moisture

TABLE 1

Residues of DDT and HCH in wheat flour as such
and after chapati preparation*

Insecticide	Sample No.	Residue (ppm) (Fresh weight basis)		Reduction (per cent)	Residue (ppm) (Dry weight basis)**	
		Flour	Chapati		Flour	Chapati
DDT	I	0.12 ± 0.005	0.06 ± 0.015	50	0.13	0.11
	II	0.57 ± 0.010	0.37 ± 0.010	35	0.63	0.67
HCH	I	2.2 ± 0.15	1.34 ± 0.06	44	2.5	2.3
	II	0.39 ± 0.06	0.26 ± 0.10	33	0.43	0.47

* Mean of 3 replications.

** Per cent reduction was not calculated as there was no significant difference in the residue levels between the flour and chapatis when expressed on dry weight basis.

content in the 2 commodities were quite different. Fresh chapatis contained 45 per cent moisture as compared to 10 per cent in the wheat flour.

The levels of DDT residues in fresh chapatis were 35-50 per cent lower than that in wheat flour. However, the DDT residues in flour and chapatis, when expressed on dry weight basis, were found to be almost the same. Thus, no significant loss in the actual amount of DDT seems to have occurred during chapati making. The data also did not indicate any significant loss of HCH residues during processing. These results are unlike the observations of MUKHERJEE *et al.* (1973) who reported about 90 and 95 per cent reduction in DDT and lindane residues, respectively during chapati making. However, the difference in moisture content in the two commodities does not seem to have been taken into consideration in their results. Further, the high level of fortification and short period of ageing may be among the other factors responsible for high loss of insecticides as observed by them during chapati making. The fate of gamma-HCH (lindane) residues during bread making has been studied by BRIDGES (1958) and SAHA AND SUMNER (1974). According to BRIDGES (1958) only 30 to 40 per cent of the initial lindane residues in wheat flour remained after baking whereas SAHA and SUMNER (1974) reported that about 77 per cent of lindane residues were retained by bread. Major loss of lindane was considered to be due to volatilization which depended upon the time of heating. As the time taken for baking of bread was about 30 min as compared to about 5 min for chapatis, the loss in the latter process is likely to be less. This may also be possible reason for insignificant loss of DDT during chapati making as compared to about 50 per cent loss reported to occur during bread making (LEGGIERI 1950, ZEUMNER and NEUHAUS 1953). The glc and tlc pattern of DDT and HCH residues in the extracts of wheat flour and chapatis did not reveal any degradation products of these insecticides during processing. DDT residues were found mainly as p,p'-DDT and o,p'-DDT in the flour and chapatis. Distinct spots corresponding to different isomers of HCH of almost the similar intensity were found by tlc in the extracts of both flour and chapatis.

As no measurable loss of insecticides residues was observed during the processing, the results were confirmed by repeating the whole experiment thrice starting from the preparation of chapatis to the estimation of insecticides residues. The high value of DDT and HCH residues found in one sample of chapati as compared to the flour may possibly be due to the inherent variation in the technique of residue analysis employed.

JOIA *et al.* (1977) have already reported widespread contamination of wheat flour with these insecticides in the Punjab. The present findings that almost all the DDT and HCH residues present in wheat flour were retained as such by chapatis indicate the possibility of substantial intake of these insecticides by man through the contaminated wheat flour. The results, therefore, emphasise the need for concerted efforts to check the contamination of wheat flour with these insecticides.

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