THE EFFECTS OF PROCESSING AND STORAGE OF DAIRY PRODUCTS ON CHLORINATED INSECTICIDE RESIDUE[1,2]

I. DDT AND LINDANE

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Summary

The effects of processing and storage of butter, ice cream, Swiss-type cheese, condensed milk, and dry whole milk powder from milk containing DDT, lindane, and DDT and lindane in combination were studied. The only change in structure occurred to DDT and lindane during drying of the milk into powder. Lindane suppressed the amount of DDT residue in milk when both insecticides were fed together. In general, the finished products other than dry whole milk contained the same amount of insecticide as the raw milk when expressed on a fat basis.

The literature contains many reports on how chlorinated insecticides enter milk *(3, 4, 5)* but there is a general lack of information concerning the effects of processing and storage of dairy products on chlorinated insecticide residues *(5)*. Mann et al. *(2)* reported that pasteurization had very little effect on the amount of DDT in the milk. They also found that DDT followed the fat phase resulting in higher concentration of DDT in the high fat products.

This study was conducted to determine what effects, if any, processing and storage had on the insecticide residues present in milk or milk products and how the residues were partitioned during the manufacture of butter, ice cream, Swiss-type cheese, condensed milk and dry wholemilk powder.

This paper reports results on the effects of processing and storage of dairy products manufactured from milk containing DDT, lindane, and DDT and lindane in combination.

Methods

Milk with the desired concentration of insecticide residues for the manufacture of the dairy products was obtained as follows:

1. Insecticides were incorporated into milk by feeding. Capsules containing the desired insecticide were fed to Holstein cows. Each animal was fed 250 mg technical grade DDT daily (approximately 72% pp' DDT). Technical grade lindane was fed at the rate of 760 mg per animal. The same amount of each insecticide was fed when used in combination. The milk was processed when the concentration of the insecticide residue in the milk was approximately 0.8 ppm. It usually required a week to reach and maintain this concentration.

2. Insecticides were added directly to milk. The desired insecticide was dissolved in 75 ml ethanol and then added to milk in a vat. The milk was heated to 80 F and agitated for 15 min. Both 0.1 and 1.0 ppm concentration of the insecticides were added to milk in this way.

Manufacture of the Products

In the following the term "milk" refers to whole milk containing insecticides.

Butter. Butter was manufactured from cream separated from milk pasteurized at 145 F for 30 min. The cream was churned in 1-gal glass Dazey butter churns equipped with mechanical paddles. The buttermilk was removed through an opening at the top of the churn. The butter was stored at −15 F for 4 months.

Ice Cream. The mix used for the manufacture of ice cream was pasteurized at 165 F for 30 min, homogenized at 2000 and 500 psi, 1st and 2nd stage, respectively, cooled and stored overnight at 37 F. A Cherry-Burrell, Model FR40B batch-type freezer was used to freeze the mix into ice cream. The ice cream was stored at −15 F for 4 months.

Swiss-Type Cheese. The milk used for the manufacture of the cheese was pasteurized at 145 F for 30 min. Starter bacteria and *Propionibacterium shermanii* were added and the milk was ripened for 30 min at 86 F. Thirty min after the addition of rennet, the curd was cut to the size of wheat grains. Next, whey equal to 1/3 of the volume of the original milk was removed and was replaced with hot water to give a final cooking temperature of 102 F. Cooking required 30 min. The curd was placed in hoops, and pressed for 3 hr at 4-5 psi pressure. After pressing, the cheese was placed in a brine solution for 24 hr. The cheese was dried, sealed in Cryovac bags under vacuum, stored at 60 F for 8 weeks and at 45 F for another 8 weeks.

Condensed and Dried Milk. The milk was forewarmed at 190 F for 10 min, and then condensed in a laboratory model Rogers Vacuum pan at 115 F under 25 in vacuum. The milk was concentrated

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[2]Presented at the 145th National Meeting, American Chemical Society, New York City, September 8-13, 1963.

TABLE 1. DISTRIBUTION OF DDT AND LINDANE DURING THE MANUFACTURE AND STORAGE OF BUTTER

Product	Per cent butterfat		ppm (wt basis)		ppm (fat basis)	
	DDT[a]	Lindane[b]	DDT[a]	Lindane[b]	DDT[a]	Lindane[b]
Raw Milk	3.40	3.90	0.90	0.98	26.47	25.13
Pasteurized Milk	3.40	3.90	0.87	0.80	25.59	20.51
Skim Milk	0.02			0.19		
Cream	37.00	33.00	10.70	9.60	28.89	29.09
Butter	86.20	84.80	19.20	20.00	22.27	23.60
Buttermilk	2.30	1.40	0.73	0.71	31.74	50.71
Butter after storage	86.20		21.80		25.29	

[a]DDT added to milk.
[b]Milk from cows fed lindane.

TABLE 2. DISTRIBUTION OF DDT AND LINDANE DURING THE MANUFACTURE AND STORAGE OF ICE CREAM

Product	Per cent butterfat		ppm (wt basis)		ppm (fat basis)	
	DDT[a]	Lindane[b]	DDT[a]	Lindane[b]	DDT[a]	Lindane[b]
Ice Cream Mix	10.85	11.35	0.318	0.190	2.93	1.67
Pasteurized Mix	10.85	11.35	0.372	0.170	3.43	1.50
Mix, 1 day old	10.85	11.35	0.372	0.188	3.43	1.66
Ice cream	10.85	11.35	0.298	0.170	2.65	1.50
Ice Cream, 4 mos. old	10.85	11.35	0.260	0.177	2.39	1.60

[a]DDT added to ice cream mix.
[b]Ice Cream mix which has Lindane added.

TABLE 3. DISTRIBUTION OF DDT AND LINDANE DURING THE MANUFACTURE OF SWISS-TYPE CHEESE

Product	Per cent butterfat		ppm (wt basis)		ppm (fat basis)	
	DDT[a]	Lindane[b]	DDT[a]	Lindane[b]	DDT[a]	Lindane[b]
Milk	4.10	3.50	0.83	0.84	20.24	24.00
Milk plus cultures	4.10	3.50	0.77	0.84	18.78	23.43
Whey		.04		0.34		85.00
Curd	28.50	27.00	6.10	4.30	21.41	15.91
Cheese	28.50	27.00	6.40	4.10	22.46	15.17

[a]Milk from cows fed DDT.
[b]Milk which had Lindane added.

TABLE 4. DISTRIBUTION OF DDT AND LINDANE DURING THE MANUFACTURE OF CONDENSED MILK AND DRY WHOLE MILK POWDER

Product	Per cent butterfat		ppm (wt basis)		ppm (fat basis)	
	DDT[a]	Lindane[b]	DDT[a]	Lindane[b]	DDT[a]	Lindane[b]
Milk	3.50	4.00	0.91	1.00	26.00	25.00
Forewarmed Milk	3.50	4.00	0.91	1.00	26.00	25.00
Condensed Milk	7.71	7.57	1.78	2.00	23.05	26.42
Sterilized Milk	7.71	7.57	1.94	1.94	25.12	25.63
Spray dried	27.97	27.84	2.75	1.22	9.85	4.38
Drum dried	28.11	29.19	12.20	2.71	43.43	9.27

[a]Milk which had DDT added.
[b]Milk from cows fed Lindane.

approximately 2 to 1. A portion of the condensed milk was standardized and sealed in 8-oz cans. The canned condensed milk was sterilized by bringing the temperature to 240 F in 20 min, holding at 240 F for 15 min and cooling under tap water. The remainder of the condensed milk was used for the manufacture of dry whole milk powder. Two methods were used for the manufacture of the dry whole milk powder:

1. Spray drying. The condensed milk was heated to 120 F before being dried in a Swenson Research Spray Dryer with an air inlet temperature of 275 F and an air outlet temperature of 185 F.

2. Roller drying. The condensed milk was dried on a Buflovak double roll dryer under atmospheric pressure with a temperature of 270-280 F in the

rollers.

Samples were removed and analyzed for chlorinated insecticide residue at different intervals during the manufacturing process for all products and also after a normal period of storage. All samples were analyzed by the method of Langlois, et al. *(1)*.

The effect of feeding the cows DDT and lindane in combination on the amount and structure of the insecticides in the milk was also studied. Animals were fed either DDT or lindane for a month and then both insecticides were fed together. Samples of milk were analyzed at weekly intervals and the amount and structure of the residues was determined.

RESULTS AND DISCUSSION

Typical results for the various dairy products manufactured and analyzed are presented in Tables 1, 2, 3, and 4. All results were expressed on a fat basis for easier comparison.

The buttermilk contained more fat than under normal commercial conditions. This was due mainly to the method used to remove the buttermilk from the butter churn and the inefficient churning action of this equipment. This would partially account for the higher concentration of the insecticides in the buttermilk.

More lindane than DDT was found in the whey during the manufacture of the Swiss-type cheese. This helps to explain why the curd and cheese manufactured from milk containing lindane contained less insecticide than that from milk containing DDT. This would suggest that lindane is more soluble in whey than DDT.

The only significant loss of DDT and lindane occurred during the manufacture of dry whole milk powder. There was loss of both DDT and lindane during spray drying and loss of only lindane during roller drying. Similar results were obtained in duplicate experiments.

Sterilization caused some shift in the structure of DDT. There was some shift from the pp' DDT peak to the DDD (TDE) peak. Except for this one shift there were no other detectable changes in the structure of either DDT or lindane during the manufacture or storage of the dairy products.

In general, the changes in the DDT and lindane residues during processing, and storage of various dairy products suggest that they were essentially stable under the conditions used during this study. Therefore, the amount of residue in the finished product will be essentially the same as that in the raw milk when expressed on a fat basis.

During the feeding of DDT and lindane in combination to the animals, it was observed that the amount of DDT residue in the milk was less than

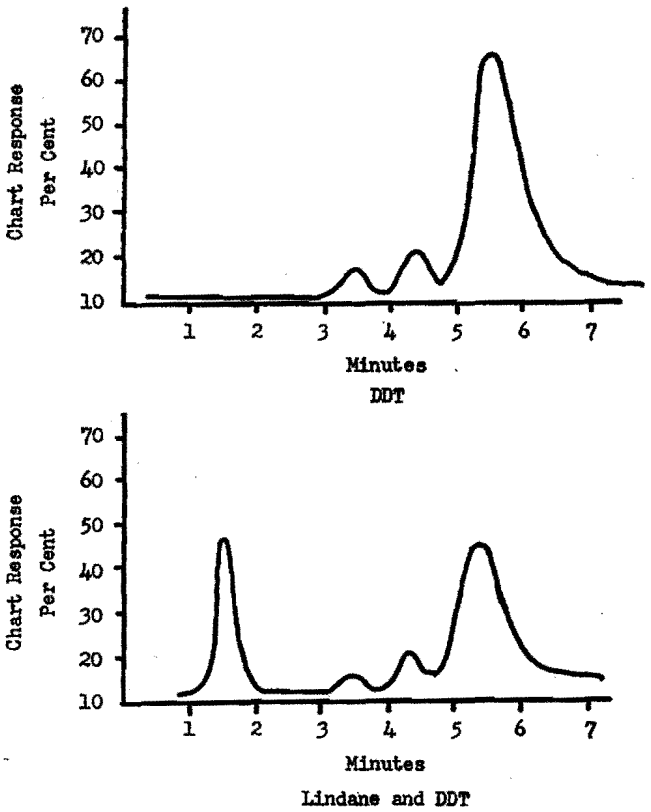

Figure 1. Comparison of chromatograms showing the relative amount of DDT Residue in milk from a cow fed DDT alone and lindane and DDT.

when DDT was fed alone. Four cows, very similar in body weight and milk production, were fed 250 mg DDT per day for one month, then 250 mg DDT and 760 mg lindane daily for the next month, to determine what effect lindane had on the amount of DDT residue in the milk. The amount of DDT residue in the milk was approximately 0.8 ppm when just DDT was fed; however, the DDT residue decreased to 0.3 ppm when lindane was fed in combination with DDT. Figure 1 shows chromatograms of DDT residue in milk from a cow fed DDT alone and also of DDT residue from the same cow after feeding DDT and lindane in combination. The chromatograms differ only in the amount of DDT residue.

The effect feeding DDT would have on the amount of lindane residue in the milk was also studied. Two cows, similar in body weight and milk production, were fed 760 mg lindane daily for one month, followed by feeding 760 mg lindane and 250 mg DDT daily for the next month, to determine what effect feeding DDT would have on the amount of lindane residue in the milk. The amount of lindane residue in the milk was the same throughout the two months. The feeding of DDT with lindane did not affect the amount of lindane residue in the milk.

From these results it would appear that lindane has an effect on the amount of DDT residue in the milk from cows fed both insecticides.

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REFERENCES

1. Langlois, B. E., Stemp, A. R., and Liska, B. J. Rapid Cleanup of Dairy Products for Chlorinated Insecticide Residue Analysis. J. Agr. & Food Chem. (In Press).

2. Mann, H. D., Carter, R. H., and Ely, R. E. The DDT Content of Milk Products. J. Milk and Food Technol. 13: 340. 1950.

3. Marth, E. H. and Ellickson, B. E. Insecticide Residues in Milk and Milk Products I. Insecticide Residues in Milk from Treatment of Dairy Cows and Barns. J. Milk and Food Technol. 22:112. 1959.

4. Marth, E. H. and Ellickson, B. E. Insecticide Residues in Milk and Milk Products II. Insecticide Residues in Milk from Dairy Cattle Fed Treated Crops. J. Milk and Food Technol. 22:145. 1959.

5. Marth, E. H. and Ellickson, B. E. Insecticide Residues in Milk and Milk Products III. Insecticide Residues in Dairy Products and Associated Problems. J. Milk and Food Technol. 22:179. 1959.