Insecticidal Residues in Milling Fractions from Wheat Treated with Methoxychlor, Malathion, and Lindane[1]

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ABSTRACT

Milling tests were conducted to determine the insecticide residue in flour derived from wheat to which three candidate protective treatments had been applied as dusts or sprays at several rates. Some lots were milled 9 to 10 days after treatment and others after aging 18 to 24 months. Samples analyzed were the wheat just before and after cleaning, and the flour, bran, shorts, and germ. In most cases, cleaning removed only a small amount of insecticide. The highest residues were in the bran and shorts. Very little insecticide carried over into the flour, 0 to less than 0.3 p.p.m. from malathion applied at 2.5 to 7.5 p.p.m.; 0.3 to 1.8 p.p.m. from methoxychlor applied at 5 to 50 p.p.m.; and 1.3 to 2.6 p.p.m. from lindane applied at 2.5 to 7.5 p.p.m.

With the accumulation of large reserve stocks of grain during the past few years, handling and storage practices have changed. Grain is stored for much longer periods and as a result improved methods of insect control are required. The emphasis is now on preventive rather than curative methods in protecting stored grain from insect attack.

In line with this trend, protective treatments of stored wheat have been investigated since 1952. As a result of these investigations, formulations of synergized pyrethrins are recommended for application as dusts and sprays to stored wheat to protect it from insect attack. There are a number of other insecticides that are more stable and longer lasting than pyrethrum. Some of these other insecticides have a relatively low level of toxicity to warm-blooded animals, and it is possible that they can be used safely on wheat and that residue tolerances can be established to permit their use. Preliminary tests with some of these materials have shown promise of effectiveness from the standpoint of killing established infestations as well as preventing new ones.

A necessary part of the development of protective treatment for stored wheat is the determination of the fate of the insecticidal residue on the wheat when it is milled. This paper reports the results of milling tests of wheat treated with methoxychlor, malathion, and lindane. The treatment studies were conducted by the Stored-Grain Insects Laboratory, Manhattan, Kansas. The analysis of residue was done by the Chemical Unit of the Stored-Product Insects Section at Savannah, Georgia. The wheat was milled in the pilot mill of Kansas State College at Manhattan.

MATERIALS AND METHODS.—The wheat used in the test was furnished by Commodity Credit Corporation from reserve stocks stored in circular metal bins, each having a capacity of 3,250 bushels. Each lot of wheat consisted of 30 bushels and was bagged as it ran from the storage bin in clean, 10½-ounce, lintless jute bags for transporting to the mill. Precautions were taken to protect each lot of wheat from contamination by other insecticides.

Samples for residue analyses were drawn from the last two-thirds of each 30-bushel lot as it was being milled. Samples of the whole grain before and after cleaning and tempering were taken directly from an auger inside the mill. Samples of the milling fractions were taken at points in the mill where the desired sample could be easily obtained.

Each sample was composed of a blended composite of 25 sub-samples taken at 5-minute intervals during the last two-thirds of each 30-bushel run. Each composite sample amounted to about 1 gallon, with the exception of the germ sample. Since the total amount of germ for each 30-bushel lot amounted to less than 1 gallon, the entire amount from the last two-thirds of the run was collected and used in the same manner as the composite samples of the other milling fractions.

In addition to the germ sample, samples were obtained from the bran, shorts, and flour fractions. The flour was a composite of all flour streams rather than separate samples of each flour stream.

Each day's grinding was started by milling a 30-bushel untreated control lot of wheat. This amount was necessary to "warm-up" the mill. "Warm-up" refers to making all the necessary settings and adjustments in the mill. Samples were taken from the untreated control lots and submitted in the same manner as those from the treated lots of wheat.

Each composite sample was divided with a Boerner Sample Divider. Two quarts were sealed in a plastic bag and shipped to Savannah for residue analyses. The remainder of the sample was sealed in a clean glass jar and held at the Manhattan laboratory as a reserve until the results of analyses were received.

The chemical analyses of the insecticidal residues were made by the methods that appear to give the greatest accuracy in the presence of grain products. The malathion was determined by the method of Norris *et al.* (1954) as modified in a private communication (1955) for use with milled wheat products: wheat, flour, bran, shorts, etc. The methoxychlor was determined by the colorimetric method of Claborn & Beckman (1952), and the lindane by that of Schechter & Hornstein (1952).

The wheat used in this test was from two dates of treatments: (1) wheat treated 18 to 24 months previously and designated as "aged" treatments, and (2) wheat treated a few days before milling and designated as "fresh" treatments.

Several levels of application rates were used in the tests in order to observe whether the residues followed in the same comparative order as the application rates.

The malathion spray formulations were made from a

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57% emulsifiable concentrate and the lindane sprays from a 20% emulsifiable concentrate. These were applied at approximately 5 gallons of dilute spray to 1,000 bushels of wheat. A refined grade of malathion was used.

The dusts were all made with wheat flour as the carrier at concentrations of 0.5 and 1% of malathion, 1, 2, 3, and 5% of methoxychlor, and 0.5% of lindane. These dusts were applied at rates of 30, 60, and 90 pounds per 1,000 bushels of wheat as necessary to obtain the desired dosage of toxicant in parts per million.

Malathion

1. Malathion (57% emulsifiable concentrate)..... 6 grams
   Water..... 744 ml.
   Application rate: above amount applied to 30 bushels of wheat
2. Malathion..... 0.5%
   Wheat flour..... 99.5%
   Application rate: 30, 60, and 90 lb./1,000 bu. for dosages of 2.5, 5, and 7.5 p.p.m. of malathion

Lindane

1. Lindane (20% emulsifiable concentrate)..... 8.5 grams
   Water..... 741.5 ml.
   Application rate: above amount and 3 times above amount applied to 30 bushels for dosages of 2.5 and 7.5 p.p.m. of lindane

AGED TREATMENTS

Malathion

1. Malathion (57% emulsifiable concentrate) 9 liquid ounces
   Water..... 5 gallons
   Application rate: 5 gal./1,000 bu.
2. Malathion..... 0.5%
   Wheat flour..... 99.5%
   Application rate: 30 lb./1,000 bu. for dosage of 2.5 p.p.m. of malathion
3. Malathion..... 1.0%
   Wheat flour..... 99.0%
   Application rate: 30 lb./1,000 bu. for dosage of 5 p.p.m. of malathion

Methoxychlor

1. Methoxychlor..... 1.0%
   Wheat flour..... 99.0%
   Application rate: 30 and 60 lb./1,000 bu. for dosages of 5 and 10 p.p.m. of methoxychlor
2. Methoxychlor..... 2.0%
   Wheat flour..... 98.0%
   Application rate: 30 lb./1,000 bu. for dosage of 10 p.p.m. of methoxychlor
3. Methoxychlor..... 3.0%
   Wheat flour..... 97.0%
   Application rate: 30 lb./1,000 bu. for dosage of 15 p.p.m. of methoxychlor
4. Methoxychlor..... 5.0%
   Wheat flour..... 95.0%
   Application rate: 30 and 60 lb./1,000 bu. for dosages of 25 and 50 p.p.m. of methoxychlor

Lindane

1. Lindane..... 0.5%
   Wheat flour..... 99.5%
   Application rate: 60 lb./1,000 bu. for dosage of 5 p.p.m. of lindane

RESULTS.—The amounts recovered are given in table 1. In two-thirds of the cases, the amount of insecticide recovered from the treated whole wheat was one-half or less of the amount of insecticide applied.

The results from analyzing the milling fractions for insecticidal residues are also given in table 1. From these figures it is apparent that most of the retained insecticide

Table 1.—Insecticidal residue found during milling and in the milling fractions of wheat treated with malathion, methoxychlor, and lindane.

Lot No.	Formulation	Dosage (p.p.m.)	Date Treated	Date Milled	Recovery of Insecticide (p.p.m.) from: Whole Grain Before Cleaning	Whole Grain After Cleaning	Milling Fractions Shorts	Bran	Germ	Flour
					Aged Malathion					
1	Dust	2.5	9/ 9/55	4/25/57	<0.2	<0.2	2.5	2.0	0.8	<0.2
2	Dust	5.0	9/ 2/55	5/22/57	.4	.4	.8	< .2	< .2	< .2
3	Spray	5.0	10/10/55	5/22/57	.4	< .2	< .2	.4	< .2	.0
					Fresh Malathion					
4	Dust	2.5	6/ 4/57	6/13/57	0	0	1.0	1.2	.5	0
5	Dust	5.0	6/ 4/57	6/13/57	1.2	0	1.3	2.4	1.2	0
6	Spray	5.0	6/ 3/57	6/13/57	1.2	0	3.0	3.4	1.3	0
7	Dust	7.5	6/ 4/57	6/13/57	3.2	.8	2.1	2.1	1.5	< .3
					Aged Methoxychlor					
8	Dust	5.0	9/20/55	1/29/57	2.7	1.8	3.0	6.9	3.9	.6
9	Dust	10.0	10/ 1/55	5/22/57	5.0	5.4	7.8	14.9	7.2	.9
10	Dust	10.0	7/15/54	4/25/57	7.5	5.9	12.3	19.8	14.9	1.5
11	Dust	15.0	10/18/55	5/22/57	7.0	5.9	10.1	17.7	8.7	1.5
12	Dust	25.0	7/ 5/55	1/29/57	14.1	10.8	15.6	27.3	18.8	1.8
13	Dust	25.0	7/16/54	4/25/57	10.8	8.7	20.0	33.0	22.1	.6
14	Dust	50.0	7/17/54	4/25/57	33.6	27.0	43.2	88.7	14.1	.3
					Aged Lindane					
15	Dust	5.0	6/17/53	1/29/57	8.6	7.8	13.0	49.5	11.8	2.3
					Fresh Lindane					
16	Spray	2.5	6/ 3/57	6/13/57	3.5	3.0	7.9	8.0	9.4	2.6
17	Spray	7.5	6/ 4/57	6/13/57		4.0	8.4	16.0	15.0	1.3
					Untreated Controls					
18	—	—	—	4/25/57	0	0	0	0	0	0
19	—	—	—	1/29/57	0	0	0	0	0	0
20	—	—	—	6/13/57	0	0	0	0	0	0

was in the pericarp and was recovered from the bran and shorts.

Malathion was recovered in amounts far below the applied dosage. The residues were found in the shorts and bran, and less than 1 p.p.m. was recovered in the flour fraction.

The results obtained from methoxychlor treatments followed about the same pattern as the malathion tests. The residues in flour were all below 2 p.p.m.

In the tests with fresh lindane, the residue pattern was nearly the same as for the other fresh treatments, except that greater residues were found in the flour. One other exception noted in the lindane tests was that the aged treatment did not follow the same pattern as malathion and methoxychlor in the recovery of insecticidal residue from the whole grain. It had a concentration of 8.6 p.p.m. as compared with 5 p.p.m. applied. Similar results were obtained in a previous milling test. Possible explanations for this are that either the insecticide was not uniformly distributed when applied, or that it was translocated in the grain mass during the storage period by unequal heating, convection currents, or other unknown factors.

ANALYSES OF RESULTS:

1. About one-half or less of the applied malathion or methoxychlor was recovered in the sample taken just prior to the cleaning process. The lindane samples showed residues greater than the application rates.
2. With the exception of fresh malathion treatments, the cleaning process removed only a small amount of insecticides, an indication that the insecticides must have been in the grain.
3. The largest portion of the residual insecticides appeared in the shorts and bran, an indication that the residues were retained in the pericarp.
4. Very small amounts of the insecticides were carried through the milling process into the flour.

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