A Comparison of Some Granular Carriers for Chlordan and Heptachlor against the Imported Fire Ant

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A study was made of carriers used in formulation of granular heptachlor and chlordan for their effect on residues in soil and on grass, and for their efficacy in total formulation against the imported fire ant. Montmorillonite formulations, as used in these studies, gave as good control and soil residues and less contamination of grass than the specification attapulgite formulations. The low bulk density of vermiculite and poor absorbancy of bentonite granules are disadvantages in formulation of insecticide granules for imported fire ant eradication.

When the Imported Fire Ant Eradication Program was begun in 1957, the immediate recommendations for insecticidal application were 2 pounds per acre of granular dieldrin or heptachlor, or 4 pounds of granular chlordan. From the economics standpoint, except when white fringed beetle also occurred, only heptachlor was used. The purchase of granular heptachlor was governed by federal specifications (*2*).

Methods improvement studies soon showed that single applications of lower dosages of heptachlor were highly effective, while only fractional poundages per acre would result in complete kill of the ants if two applications were made (*5*).

At the same time the above studies were made, other work was initiated to compare various granular carriers for heptachlor and later for chlordan with the object of determining the most effective formulation for the purpose of eradicating the imported fire ant. The present paper is a report on the progress of these carrier studies.

Procedure

Test 1. The first test series was conducted on a Bahia grass pasture heavily infested with imported fire ants near Portage Bayou, Miss. One-acre plots were set up for treatment with six different granular formulations at nominal rates of 1 pound per acre of heptachlor. All treatments were made in triplicate. Pretreatment counts were made of active ant colonies as well as posttreatment counts at 1, 2, 4, 8, and 12 months. Three randomly selected, untreated check plots were included. This is the general procedure reported in earlier work (*3, 5*).

The clay carriers used were vermiculite, bentonite, montmorillonite (calcined and noncalcined), and attapulgite (extruded, calcined, and noncalcined).

Except for the bentonite, which because of its low absorbency could only retain 2.5% heptachlor, all of the clays were formulated with 10% heptachlor as active ingredient in an equal weight of heavy aromatic naphtha solvent. The montmorillonite and attapulgite clays were deactivated with 7% deactivator "H", a product of Velsicol Chemical Corp. consisting of a mixture of polyethylene glycols, predominantly di- and triethylene glycols. All formulations were freshly prepared and assayed immediately before use.

In addition to the observation on control of the imported fire ants, the soil on the plots was sampled by chemical analysis after 1 year for insecticide residues. In this test and the subsequent ones, both heptachlor and heptachlor epoxide were determined by the method of Murphy and Barthel (*4*).

Test 2. Subsequent to the previous experiment, other investigations (*3, 5*) demonstrated that two $^1/_4$-pound per acre applications of heptachlor, spaced 3 to 6 months apart, gave complete control of imported fire ant colonies. This treatment procedure was adopted as a standard eradication method. As a result, in the following studies with granular carriers, the heptachlor application rate was reduced to $^1/_4$ or $^1/_2$ pound per acre.

The chemical residue studies indicated that the vermiculite formulations left the greatest soil residue. However, these granules have a very low bulk density. As a result, at similar insecticide concentrations, the volume of vermiculite granules per unit weight is approximately three to four times that of attapulgite granules. The increased volume is a great disadvantage when the load-carrying capacities of various types of application equipment are considered. Because of this, a test was conducted to compare the control of imported fire ant with granular vermiculite formulations containing three different percentages of heptachlor but applied at rates which gave a constant $^1/_4$ pound per acre insecticide deposit. A standard application of $^1/_4$ pound per acre of heptachlor on attapulgite granules was included for comparison. Ten pounds of the attapulgite formulation is approximately equal in volume to $2^1/_2$ pounds of the vermiculite formulation. Two and one-half, 5, and 10% heptachlor on vermiculite were compared at the rate of 10, 5, and 2.5 pounds per acre, respectively.

Test 3. A chemical study was set up in triplicate using small plots near Gulfport, Miss., to compare the persistence of heptachlor formulated with the various types of granules and applied at a nominal dosage of 0.5 pound per acre. The applications were made with a 25-foot Gandy fertilizer distributor. At the same time, the Bahia grass from these plots was sampled and analyzed for insecticide to determine if there was any considerable difference in the contamination of grass.

The Gandy fertilizer distributor applies a very evenly distributed swath but is difficult to calibrate to the exact dosage per acre desired in this work. To obtain uniformity, the same calibration was used on each replicate. Observation of weights showed less than 5% variation between dosages applied to replicates of the same formulation. Calculation to determine actual dosage per acre was made from the weights actually applied per plot.

Test 4. Data from these previous studies indicated that there was quite a considerable difference in contamination of the grass by the different formulations. To obtain more data, additional plots were set up on Bahia grass pasture to study both heptachlor and chlordan formulated with three different carriers using butyl Cellosolve as an auxiliary solvent instead of the usual heavy

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aromatic naphtha. Butyl Cellosolve was of interest in that it had lowered the tendency of granular formulation to contaminate grass in earlier studies (7). Loss of insecticide from the soil was more rapid than in previous tests apparently because the waterlogged condition of soil caused rapid breakdown of granules.

Test 5. Since the chemical residue data indicated that the montmorillonite granular insecticide formulations gave as good soil residues as the attapulgite formulations, and a small plot of imported fire ant control tests showed that the montmorillonite formulation gave as good control of ants, a larger scale test was conducted with these two carriers. This test was conducted in conjunction with another experiment on the effectiveness of chlordan for controlling imported fire ants, and, therefore, this insecticide was used instead of heptachlor. Standard formulations consisting of 2.5% chlordan, 7% deactivator, 10% heavy aromatic solvent, and 80.5% carrier were used. The plots were located north of Gulfport and included varying types of land usage and ground cover. Plot 1 was treated with the attapulgite formulation and contained about 450 acres. Plot 2, treated with the montmorillonite formulation, was 120 acres in size. Sixteen subplots for making pre- and posttreatment counts of active mounds were set up in plot 1, and six in plot 2. Each subplot was about 1 acre in area. The formulations were applied with a 450-h.p. Stearman aircraft equipped with a Swathmaster spreader. Two applications of $^1/_4$ pound per acre of chlordan were made, one during the first part of February and one the first week of June.

Results

The control of the imported fire ants with the various formulations in the first

Table I. Control of Imported Fire Ants with Heptachlor Formulated on Various Types of Granules

Application rate 1 pound per acre; applied with a Gandy fertilizer distributor[a]

Type of Granule	Pretreatment Count of Active Ant Colonies	Per Cent Reduction in Active Colonies after Following Months: 1	2	4	8	12
Vermiculite	22	59	70	100	100	98
Bentonite	18	73	91	100	100	100
Montmorillonite (noncalcined)	25	32	46	95	100	100
Montmorillonite (calcined)	28	13	32	91	100	99
Attapulgite (noncalcined)	20	64	79	97	100	98
Attapulgite (calcined)	22	39	39	97	100	100
Check	33	0	0	0	51	41

[a] Bentonite granules contained 2.5% heptachlor, and all others, 10%.

Table II. Analysis of Soil Following Application of 1 Pound of Heptachlor per Acre Using Various Carriers

Treated November 12, 1959; sampled November 23, 1960

Carrier[a]	Insecticide Actually Applied, P.P.M.	Insecticide Remaining, P.P.M.: Heptachlor	Heptachlor epoxide	Total	Insecticide Remaining, %
Attapulgite (calcined)	2.6	0.00	0.15	0.15	6
Attapulgite (noncalcined)	3.6	0.00	0.08	0.08	2
Montmorillonite (calcined)	2.7	0.00	0.11	0.11	4
Montmorillonite (noncalcined)	3.3	0.09	0.23	0.32	10
Vermiculite	3.5	0.43	0.15	0.58	17
Bentonite	3.8	0.00	0.23	0.23	6

[a] Nominal particle size of all carriers was such that 97% passed through a size 18 sieve, and 95% was retained on size 40 sieve.

Table III. Effect of Different Bulk Application Rates of Heptachlor-Vermiculite Granules on Control of the Imported Fire Ant When Insecticide Dosage Remained Constant at 1/4 Pound per Acre

Average of 3 replications; applied with Gandy fertilizer distributor in October 1960

Type of Granule and Heptachlor Concentration	Application Rate, Pounds/Acre	Av. Pretreatment Count of Active Ant Mounds	Per Cent Reduction in Active Ant Colonies after Following Months: 1	2	4	8	12	18	24
Vermiculite (2.5%)	10	19	45	50	64	93	88	95	78
Vermiculite (5.0%)	5	26	51	78	77	100	95	94	73
Vermiculite (10.0%)	2.5	20	44	58	69	100	93	86	42
Attapulgite (2.5%) (standard)	10	19	53	66	74	98	95	98	67
Check	...	22	0	0	0	10	11	14	27

Table IV. Insecticide Residues on Grass and in Soil Following Granular Application of Heptachlor Formulated with Different Clay Carriers[a]

Clay Carrier[b]	Amount Applied to Soil: Lb./A.	P.P.M.[c]	Residue in Soil, P.P.M., Sampled April 14, 1961: Heptachlor	Heptachlor epoxide	Total	Insecticide Residues on Grass, P.P.M., Sampled March 10, 1961: Heptachlor	Heptachlor epoxide	Total	Sampled April 25, 1961: Heptachlor	Heptachlor epoxide	Total
Attapulgite AARVM	0.56	1.68	0.21	0.13	0.34	0.90	0.22	1.12	0.40	0.52	0.92
Montmorillonite AARVM	0.64	1.92	0.52	0.10	0.62	0.56	0.16	0.72	0.18	0.30	0.48
Montmorillonite ARVM	0.77	2.31	0.48	0.09	0.57	0.20	0.28	0.48	0.10	0.24	0.34
Montmorillonite ALVM	0.48	1.44	0.33	0.07	0.40	0.10	0.14	0.24	0.10	0.08	0.18
Bentonite	0.4	1.30	0.06	0.07	0.13	0.26	0.36	0.62	0.24	0.32	0.56
Vermiculite	0.8	2.40	0.83	0.05	0.88	0.40	0.36	0.76	0.28	0.30	0.58

[a] All formulations made to nominal percentage of 5% except bentonite, which was formulated at 1.19%. Application made February 15–16, 1961.
[b] AARVM = extruded, dried, but not calcined; ARVM = dried, but not extruded or calcined; ALVM = calcined, but not extruded.
[c] Calculated in top inch of soil.

Table V. Summary of Residue Data from Table IV after Correction for Nonuniform Application[a]

Clay Carrier	Insecticide Residue, P.P.M. after Days Indicated: In soil, 57–58 days	On Grass: 22–23 Days	On Grass: 68–69 Days
Attapulgite AARVM	0.30	1.00	0.82
Montmorillonite AARVM	0.48	0.56	0.38
Montmorillonite ARVM	0.37	0.31	0.22
Montmorillonite ALVM	0.42	0.25	0.19
Bentonite	0.16	0.78	0.70
Vermiculite	0.55	0.47	0.36

[a] Adjusted for application of 0.5 pound per acre. Residue includes heptachlor plus heptachlor epoxide.

test is reported in Table I. The data obtained show that, under these conditions, 100% control of imported fire ants was obtained with all the granular formulations after 8 months. Some reinfestation had occurred on a few of the plots after 12 months. The speed of control varied considerably with the different treatments. The montmorillonite clays gave the slowest control, and the bentonite the fastest. The calcined granules gave slower kill than the noncalcined. The rate of control appeared to be correlated with the water-breakdown properties of the granules—i.e., those which disintegrated most rapidly gave the fastest kill. The one exception to this was vermiculite. The faster kill with this granular formulation could be due to better distribution of the heptachlor because of the much larger number of granules per unit weight.

Results of chemical analysis of the soil from plots in test 1 are given in Table II.

The results of the second test of the various vermiculite formulations are given in Table III, and indicate that the more concentrated heptachlor formulations (5 and 10%) gave slightly better control after 8 to 12 months than the lesser concentration (2.5%). A faster loss of insecticide from highly concentrated granular formulations is suggested by the data from 18 and 24 months. Other unpublished work by the laboratory has indicated this is also true of attapulgite granular formulations.

Table VI. Analysis of Soil and Bahia Pasture Grass Following Granular Application of Heptachlor and Chlordan in Various Formulations[a]

Formulation[b]	Amount Applied Lb./A.	Amount Applied P.P.M.[c]	Insecticide Found on Grass, P.P.M., Sampled May 3, 1961: Heptachlor	Heptachlor epoxide	Chlordan	Total	Insecticide Found on Grass, P.P.M., Sampled May 11, 1961: Heptachlor	Heptachlor epoxide	Chlordan	Total
5% Heptachlor										
Attapulgite AARVM with Naphtha Solvent	0.59	1.77	0.24	0.30		0.54	<0.10	<0.10		<0.10
Attapulgite AARVM with Butyl Cellosolve	0.51	1.53	0.16	0.22		0.38	<0.10	<0.10		<0.10
Vermiculite with Butyl Cellosolve	0.61	1.83	0.16	0.32		0.48	<0.10	<0.10		<0.10
Montmorillonite ARVM with Butyl Cellosolve	0.49	1.47	0.24	0.26		0.50	0.21	0.24		0.45
5% Chlordan										
Attapulgite AARVM with Naphtha Solvent	0.50	1.50		0.00	0.24	0.24		0.00	0.28	0.28
Attapulgite AARVM with Butyl Cellosolve	0.86	2.58		0.00	<0.10	<0.10		0.00	<0.10	<0.10
Vermiculite with Butyl Cellulose	0.79	2.37		0.00	0.30	0.30		0.00	0.38	0.38
Montmorillonite ARVM with Butyl Cellosolve	0.60	1.80		0.00	0.18	0.18		0.00	0.18	0.18

Formulation[b]	Amount Applied Lb./A.	Amount Applied P.P.M.[c]	Insecticide Found on Grass, P.P.M., Sampled May 3, 1961: Heptachlor	Heptachlor epoxide	Chlordan	Total	Insecticide Found in Soil, P.P.M., Sampled May 11, 1963: Heptachlor	Heptachlor epoxide	Chlordan	Total
5% Heptachlor										
Attapulgite AARVM with Naphtha Solvent	0.59	1.77	0.00	0.10		0.10	0.23	0.09		0.32
Attapulgite AARVM with Butyl Cellosolve	0.51	1.53	0.00	0.12		0.12	0.06	0.00		0.06
Vermiculite with Butyl Cellosolve	0.61	1.83	0.00	0.16		0.16	0.39	0.02		0.41
Montmorillonite ARVM with Butyl Cellosolve	0.49	1.47	0.00	0.14		0.14	0.19	0.03		0.22
5% Chlordan										
Attapulgite AARVM with Naphtha Solvent	0.50	1.50		0.00	0.00	0.00		0.00	0.12	0.12
Attapulgite AARVM with Butyl Cellosolve	0.86	2.58		<0.10	0.00	<0.10		0.00	0.16	0.16
Vermiculite with Butyl Cellosolve	0.79	2.37		0.00	<0.10	<0.10		0.01	0.18	0.19
Montmorillonite ARVM with Butyl Cellosolve	0.60	1.80		0.00	0.14	0.14		0.01	0.21	0.22

[a] Applied April 18–19, 1961, with Gandy fertilizer distributor to dry grass.
[b] AARVM = extruded, dried, but not calcined. ARVM = dried, but not extruded or calcined.
[c] Calculated for top inch of soil.

Table VII. Summary of Residue Data from Table VI, after Correction for Nonuniform Application[a]

Formulation	Insecticide Residue in Soil in P.P.M. after 1 Week	Insecticide Residue on Grass, P.P.M., after Indicated Days: 15	22	45
5% Heptachlor				
Attapulgite AARVM with Naphtha Solvent	0.38	0.35	<0.10	0.12
Attapulgite AARVM with Butyl Cellosolve	0.06	0.22	<0.10	0.12
Vermiculite with Butyl Cellosolve	0.50	0.39	<0.10	0.20
Montmorillonite ARVM with Butyl Cellosolve	0.22	0.26	0.45	0.14
5% Chlordan				
Attapulgite AARVM with Naphtha Solvent	0.12	0.24	0.28	0.00
Attapulgite AARVM with Butyl Cellosolve	0.28	<0.10	<0.10	<0.10
Vermiculite with Butyl Cellosolve	0.30	0.47	0.60	<0.10
Montmorillonite ARVM with Butyl Cellosolve	0.48	0.22	0.22	0.17

[a] Adjusted for application at 0.5 lb./acre.

The results of the chemical analyses of residues of the plots of test 3 are given in Table IV. Since application to the small plots was not exactly uniform, in Table V are presented data showing what might be expected if all applications were exactly 0.5 pound per acre. These calculations were made by using a suitable factor with the data of Table IV.

Results of both soil and grass analyses from test 4 are given in Table VI. Because of nonuniform application rate on these plots, the data are summarized in Table VII with adjustments made by a factor to show what might be expected at application rates of 0.5 pound per acre.

The control of imported fire ants with attapulgite and montmorillonite formu-

Table VIII. Results of Large Scale Aerial Test to Determine the Effectiveness of Two Applications Each of Two Different Formulations of Chlordan against Imported Fire Ants at Lyman, Miss.[a]

Insecticide Formulation, %	Chlordan Application Rate, Lb./Acre	No. of Subplots	Av. Pretreatment Count of Active Mounds per Subplot	Reduction, %, in Active Mounds after Following Months				
				1	3	5	8	12
Chlordan, 2.5; Montmorillonite ARVM, 80.5; Deactivator "H," 7; Heavy aromatic naphtha, 10	0.25	6	32	29	28	81	86	95
Chlordan, 2.5; Attapulgite AARVM, 83.1; Deactivator "H," 4.4; Heavy aromatic naphtha, 10	0.25	16	31	38	44	85	89	87
Check . . .	. .	7	38	0	14	42	48	

[a] First application made in February 1962, and second in June 1962. Bulk application rate 10 lb./acre.

Table IX. Insecticide Soil Residues Following Granular Application of 1/4 Pound per Acre of Chlordan by Airplane

Type of Granule	Date of First Treatment	Chlordan Found,[a] P.P.M.	Date of Second Treatment	Chlordan Found, P.P.M.
Attapulgite AARVM	2-2-62	0.17 (Sampled 3-19-62)	6-5, 6-62	0.20 (Sampled 10-30-62)
Montmorillonite ARVM	2-12-62	0.08 (Sampled 3-28-62)	6-7, 8-62	0.20[b]

[a] 1/4 pound per acre gives approximately 0.75 p.p.m. in the top inch of soil at time of application.
[b] Also found 0.03 p.p.m. heptachlor epoxide on these plots.

lations of chlordan in test 5 is recorded in Table VIII, and the chemical analyses of insecticide residues from them in Table IX.

The soil from plots treated with chlordan in this and other tests was analyzed, in addition, for heptachlor epoxide resulting from heptachlor impurity in technical chlordan.

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BIRD REPELLENCY

A Laboratory Method for Evaluating Chemicals as Bird Repellents

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THE SEARCH for bird repellents to protect seeds and agricultural crops, as reported by Neff and Meanley (*4*), dates back approximately 150 years; these early studies, however, were usually intermittent and directed at solving specific local problems. A review of previous work in this field (*2–7*) shows that within recent years a more concentrated effort has been made to evaluate the effectiveness of various chemicals as bird repellents; but there is clearly indicated a need for a quantitative method for reliably comparing one chemical against another. Such a method would be of special importance in correlating repellent activity and structure of a large volume of chemicals.

A statistical method for evaluating the effectiveness of candidate bird repellents involving concentration-effect measurements was recently adapted from the method of Litchfield and Wilcoxon (*1*). The adapted method involves the calculation of an R_{50} value, analogous to the LD_{50} used in toxicological studies. A similar procedure was reported by Tigner and Besser (*8*) for appraising chemicals as rodent repellents.

This paper describes the methodology involved in the evaluation of chemicals as bird repellents, including reproducibility data under the chosen laboratory conditions.

Procedure

Choice of Bird Species and Test Seed. The red-winged blackbird (*Agelaius phoeniceus*) was chosen as the test species because it is one of the primary birds

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