The Relation of Particle Size to the Toxicity of DDT Dusts[1]

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The purposes of this work were to find the most effective particle-size range of the toxicant DDT against the adult Mexican bean beetle, *Epilachna varivestis* Muls.; and to find which, if any, particle-size range of the diluent, *Pyrax ABB*, is the most effective in combination with DDT.

With the beginning of the investigations of the relation of particle-size to toxicity, it was found that generally the smaller particles of any particular poison were the most efficient for killing insects. However as more evidence was assembled it became apparent that a small-particle size of toxicant could not arbitrarily be rated as the most efficient killing size for all insects; and that any particular insect usually has a specific particle-size range for each particular insecticide or group of insecticides to which it is susceptible. Seigler and Goodhue (7) in tests on codling moth larvae found that there was little difference in the toxicity of coarse, medium and fine particles in calcium arsenate, Paris Green and cryolite samples, although the medium size was slightly superior to the coarse and fine. The respective sizes of these materials in their order listed were: 28, 5, 3 microns; 29, 10, 3 microns; 28, 8, 2 microns. Smith *et al* (9) observed that the coarse samples (samples containing the smallest percentage of particles below 5 microns in size) of Calcium arsenate caused the highest mortality to the boll weevil, and the larvae of the cotton leaf worm. Gaines (2) showed that the coarse sample of calcium arsenate was six times and the medium sample four times as toxic as the finest sample to fifth instar cotton leaf worm. McGovran *et al* (6) showed that the smallest particles of Paris Green, averaging 1.1 microns, caused the highest mortality and the least feeding; and the largest size particles, averaging 22 microns, gave the least kill and permitted more feeding among adult Mexican bean beetles. Smith (8) in tests on mosquito larvae found that the smaller particles were more toxic and induced more rapid paralysis than the larger particles because the fine powder covered more surface. With DDT, Woodruff and Turner (11) found that a reduction of DDT particle size caused an increase in toxicity of residues to the housefly. Also DDT dusts of the smallest particle size (10-20 microns) gave the best control of potato insects, particularly the potato flea beetle, and the heaviest yield of potatoes.

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[1] The data presented herein were obtained during the preparation of his thesis for the Master's degree at Purdue University. Contribution from the Entomology Department.

With regards to particle size of the diluent as effecting toxicity, Shipitzina found in 1935, as quoted from McGovran *et al* (6) in tests concerning the natural abilities of insect larvae to ingest different sizes of particles of insecticidal materials that "first-instar mosquito larvae (*Anopheles maculipennis messeae*) ingested quartz particles with maximum diameters ranging from 22.8 to 34.2 microns, and that fourth instar larvae ingested particles 68 to 165.3 microns in size." Chiu (1) found that in testing inert materials against the adult bean weevil, they are effective as killing agents, presumably because of the desiccating effect. The particles of finer sizes gave higher insecticidal efficiency. Hunt (4) found that abrasion is a primary action of a diluent in effecting toxicity of complete dusts, and that this abrasive action ultimately effects the speed of penetration of the toxicant present, and the desiccating effect of the dust upon the insects. The importance of these modes of action are, in turn, dependent upon climatic conditions, one particular set of climatic conditions favoring at any given time one or the other actions.

Materials and Equipment

The diluent for the dusts in this series of experiments was *Pyrax ABB* produced by the R. T. Vanderbilt Company of New York, and manufactured by the Standard Mineral Company of Robbins, North Carolina. According to Watkins and Norton (10) the material undergoes a milling and air-flotation process; the color is cream; the specific gravity is 2.84; the density is 30#/cu. ft. by the Scott volumeter; the average particles are flat, plate-like flakes. The average chemical analysis is as follows:

Ignition loss	4.00%
H2A12(SiO3)4	93.96%
MgO	0.02%
CaO	0.06%
K2O	1.54%
Fe203	0.07%
Na20	0.20%
TiO3	0.15%

The DDT used for these tests was manufactured by Geigy Company Incorporated of New York. The material possessed a melting point of 90 degrees, and is known as a technical commercial type. According to Ginsburg (3) a sample of this chemical consists largely of two isomers, P,P'-DDT (1-trichloro-2,2-bis(p. chlorophenyl) ethane) and O,P'-DDT (1-trichloro-2,O-chlorophenyl-2-p. chlorophenyl ethane). These isomers are present in quantities that may vary from 70-80 and 15-20 per cent, respectively. In addition there are usually present in small quantities, other isomers, by-products and impurities. The material is white, somewhat damp to the touch and is composed of clotted clumps of irregular crystals.

Both of the dust constituents were separated into the various particle-size ranges separately on a Ro-Tap Test-in Sieve Shaker. The

Pyrax ABB was shaken for a period of one half hour, at which time the various particles had settled into their respective screen sizes (Figs. 1-5).

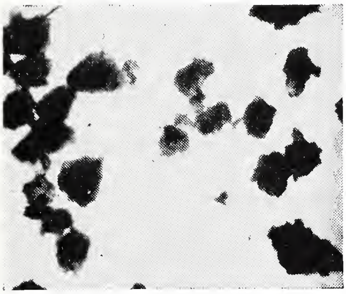

Figure 1. Microphotograph of DDT particles of size-range 104-149 microns (X110).

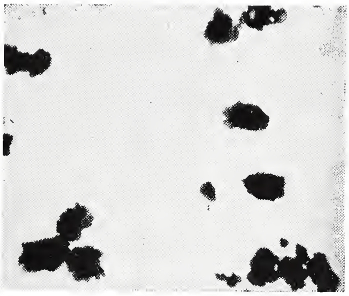

Figure 2. Microphotograph of DDT particles of size-range 74-104 microns (X110).

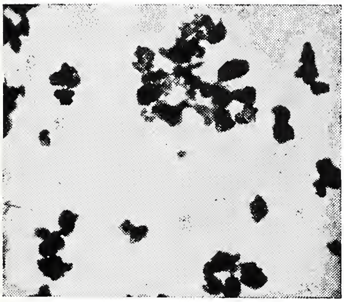

Figure 3. Microphotograph of DDT particles of size-range 53-74 microns (X110).

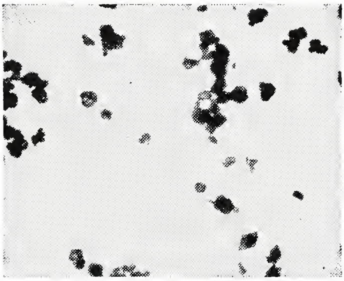

Figure 4. Microphotograph of DDT particles of size-range 44-53 microns (X110).

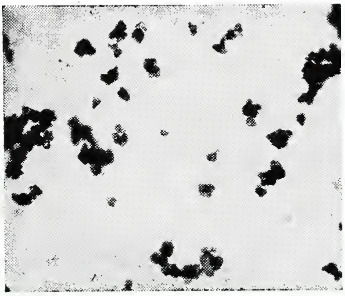

Figure 5. Microphotograph of DDT particles of size-range 0-44 microns (X110).

The sieve nest was then removed from the machine and each sieve was tapped separately to free the meshes of clogging particles. The nest then was replaced in the machine and shaken for an additional fifteen minutes. The DDT was treated in a like manner, except it was first ground in a mortar. Since the DDT caused extreme clogging of the sieve meshes, the sieved-out portions of DDT were removed at the end of the first half-hour, and the sieves were washed with carbon disulphide to dissolve the remaining DDT and dried. Then the DDT particles were placed in their respective sieves and shaken for the remaining fifteen minutes. The resulting particle-size ranges, the sieve sizes and their micron equivalents, plus the resulting particle-size analysis by this method of fractionation for the *Pyrax ABB*, are given in Table I. It was not possible to secure a reliable particle-size analysis for DDT, because of the excessive amount of clogged material that had to be dissolved out of the sieve screens. The various particle sizes of DDT obtained are shown in the microphotographs (Figs. 1-5).

TABLE I. The physical data of the sieving operation.

Sieve sizes in meshes per inch	Micron-size Equivalents	Micron-size ranges of sieved particles		*Pyrax* particle -size analysis
		DDT	*Pyrax ABB*	
100	149		Above 149[2]	0.16%
150	104	104-149	104-149	2.22%
200	74	74-104	74-104	0.83%
275	53	53-74	53-74	3.19%
325	44	44-53	44-53	6.11%
....	....	0-44	0-44	87.84%

[2] This particle-range size material was not used in the experiments.

The DDT and *Pyrax ABB* were mixed at a 2 per cent concentration by weight, as determined by preliminary tests for the L.D. 50. These materials were mixed in relatively small quantities, approximately 100 grams, because of excessive amount of time needed to obtain the various particle-size fractions by the sieving process. The mixing was done on a Model 10 Oster Mixer modified to the mixing of dusts. A 150 Watt bulb was put into the circuit as resistance to reduce the speed of the mixing blades to the point where the size of the particles was not affected. The upright mixing blades of the machine were inverted and another blade was added to create stirring in all parts of the mixing container. The original mixing container was discarded and a pint Ball jar was substituted. Each sample was mixed for fifteen minutes.

The testing cages used were 10-inch lantern globes covered at one end with cheese cloth. Six-inch, two-leaved *Stringless Greenpod* beans, grown in three-inch pots, were used as the testing plants. A turntable set to revolve once in three seconds was the dusting platform. The dusting equipment had as its air source the motor from a Hudson Electric-Spray, Paint sprayer 305. The dusting container consisted of an ordinary, insect suck-bottle, with a piece of glass tubing slightly drawn to serve as a nozzle. The speed of the duster motor was cut by inserting a 150 Watt bulb in the circuit to prevent excessive blowing and wasting of the dust material. Early second-brood and early third-brood adult Mexican bean beetles, *Epilachna varivestis* Muls., approximately one week old and collected in the field as needed, were used as test insects.

Experimental Procedures

The general plan of procedure for this series of experiments was to secure various particle-size ranges of DDT and similar particle-size ranges of *Pyrax ABB*, and test, as separate samples, all combinations of these two materials possible, within the limits of time and the material on hand. This plan resulted in the running of four series of experiments. In the first series, DDT and *Pyrax ABB* of the same particle size were mixed and tested; in the second series the largest DDT particles were mixed with Various *Pyrax ABB* particles; in the third series, the smallest-sized particles of DDT were mixed with the various *Pyrax ABB* particles; in the last series, the next to the smallest DDT particles were mixed with the various *Pyrax ABB* particles and tested. Table II lists the combinations of the dust mixtures used in these four series of tests.

Each sample of dust in a series was run on five plants at a time, each plant being infested with five beetles, making a total of 25 beetles per test for each sample. Each test was replicated five times, each on different days. Untreated checks were used with all tests. Checks treated with *Pyrax ABB* particles of the same size-range as those in the dust being tested were run with the first series of tests, but these were discontinued when no mortalities over the normal amount in untreated checks were produced. This non-toxic characteristic of *Pyrax* materials is indicated by Hunt (4) in his paper on the toxic action of diluents.

The five plants used for each sample were dusted at the same time by placing them on the dusting turntable together. The dust gun was held two feet from the plants during the dusting operation which lasted for six revolutions of the turntable. For three turns the dust was directed at the topside of the leaves, and for three turns at the lower side of the leaves. The 25 beetles for each sample test were placed together in a pint ice cream container covered with a layer of cheese cloth, and dusted therein for a count of three seconds. This method for dusting the insects was used as it was impossible to retain the beetles on the plants during the dusting operation. Lantern globes then were put over the plants and the beetles were added, five per plant. All of the tests of one replication of a series were run at the

TABLE II. The various dust mixtures for the four series of tests, showing the sample designations for each mixture, and the formulation of the mixture according to micron size of the DDT and *Pyrax ABB* particles.

Test Series	Sample	Formulation by Micron Size	
		DDT	*Pyrax ABB*
One	A1	104-149	104-149
	B1	53-74	53-74
	D1	44-53	44-53
	E1	0-44	0-44
	F1	Untreated	Untreated
Two	B2	104-149	74-104
	C2	104-149	53-74
	D2	104-149	44-53
	E2	104-149	0-44
	F2	Untreated	Untreated
Three	A3	0-44	104-149
	B3	0-44	74-104
	C3	0-44	53-74
	D3	0-44	44-53
	F3	Untreated	Untreated
Four	A4	44-53	104-149
	C4	44-53	53-74
	D4	44-53	44-53
	E4	44-53	0-44
	F4	Untreated	Untreated

same time. The tests of Series One were run in the month of August, and were kept together in a room of fairly constant temperature. The tests of the other three series were run in October and kept in a greenhouse. The different climatic factors resulting from the differences in season and site of operation may have affected the final results of the experiments somewhat. Mortality counts were taken every twenty-four hours for a ninety-six hour period. Gross feeding estimates also were made at the twenty-four period to determine if the dusts had any effect on the rate of feeding. These were rated as none, slight, moderate and excessive damage (Fig. 6). The numerals 0, 1, 2 and 3 were used as working designations for these criteria, respectively, and the total average feeding per sample was expressed as a mean of the arithmetic sum of all the tests of a sample. The larger the numerical values the heavier was the feeding.

Observations were made on the coverage and adherence of each particle-size range, and some variation of these properties was found. The *Pyrax ABB* particles controlled the coverage and adherence, for they

Fig. 6. Contact photographs showing slight, moderate and extensive feeding on bean leaves, referred to as (1), (2), and (3) degree damage.

comprised the bulk of any sample. A dust sample of the E1 size (0-44 microns), the smallest, gave the smoothest and the most consistent and best sticking coat on the bean leaves, as well as on the beetles. Dusts samples of the D1 size (44-53 microns), the next to the smallest, gave a coating that covered the foliage and beetles very well, though it did not adhere quite as well as did the smallest particle-size sample. Samples of the C1 size (53-74 microns) gave good coverage to foliage but only a fair coverage to the beetles, and did not stick very well to either. Samples of the B1 size (74-104 microns) gave a fairly consistent coverage to the foliage, but none to the beetles; and a slight jar would cause the material to fall from the foliage. The individual particles of the coating were discernible to the unaided eye at this size range. Samples of the A1 size (104-149 microns) gave a fairly consistent gross coverage, though very few of the particles would stick to the beetles. If a leaf dusted with this size particle was disturbed in any manner, the particles would fall. The individual particles of this size range were plainly discernible.

The data were reviewed statistically according to Love (5) and subjected to an analysis of variance to determine the reliability of the data. The least significant difference for mortalities between samples of a series were ascertained, also.

Results

Table III presents the average net mortality of bean beetles for the four dust samples in the first series of experiments. These samples were composed of combinations of diluent and toxicant of the same particle-size range and the untreated check.

These tests show that sample D1, representing a combination of the next to the smallest particle-size range, 44-53 microns, of toxicant and diluent was the best. Sample C1, representing a particle-size range of 53-74 microns, gave the next best mortality count, but the differences between C1 and D1 were not significant and cannot be accurately differentiated. The largest and smallest particle-size ranges, 104-149 and

TABLE III. The effect of particle size of a two per cent DDT dust in *Pyrax ABB*, of Series One, on the feeding and mortality of Mexican bean beetle adults.

Sample	Feeding Rate 24 Hours	Micron-size Range DDT	*Pyrax ABB*	Per Cent Mortality 72 Hours
A1	2.0	104-149	104-149	41.6
C1	1.6	53-74	53-74	63.2
D1	1.5	44-53	44-53	76.8
E1	1.8	0-44	0-44	35.2
F1(Check)	2.8	...-...	..-.	1.6

Difference in mortality required for significance at odds of 99:1, 20.4

0-44 microns respectively, gave the least kills; the former gave a higher, but not a significantly higher, mortality count than the latter. Statistically the overall data were significant at odds of 99:1, and the difference made sample D1 significantly better than samples A1, E1 and the check, but not C1. Sample C1 also was significantly better than samples A1, E1 and the check. Samples A1 and E1 were only better than the check.

The sample in this test, D1, which caused the greatest mortality, also permitted the least feeding. Also the dusts of all the samples apparently had some repellency effect on the beetles, for more feeding was consistently done on the untreated checks than on any one of the four treated samples. The D1 size dusts gave good coverage and adherence, but not nearly as good as the E1 sample. This would indicate that particle-size has more effect on mortality than does coverage and adherence.

Table IV presents the average net mortality of bean beetles for the four dust samples in the second series of experiments. These samples were composed of the largest-sized DDT particles, 104-149 microns, in combination with all other *Pyrax ABB* particle-sizes except the 104-149 micron size, and the untreated check.

These showed that sample C2 representing a combination of the largest-sized DDT particles, 104-149 microns in combination with the middle-sized particle range of diluent, 53-74 microns, was the best. Sample D2, representing a combination of DDT and *Pyrax ABB* of micron-size ranges 104-149 and 44-53, respectively, was the next best. Sample B2, representing a combination of DDT and *Pyrax ABB* of micron size-ranges 104-149 and 74-104 respectively, was the next best. Sample E2, representing a combination of DDT and *Pyrax ABB* of micron size-ranges 104-149 and 0-44 respectively, gave the least kill. However, although statistically the overall data were significant at odds of better than 20:1, the difference in mortality required for significance at these odds was 18.5. At this value none of the mortalities for

TABLE IV. The effect of particle size of a two per cent DDT dust in *Pyrax ABB*, of Series Two, on the feeding and mortality of Mexican bean beetle adults.

Sample	Feeding Rate 24 Hours	Micron-size Range		Per Cent Mortality 72 Hours
		DDT	*Pyrax ABB*	
B2	2.0	104-149	74-104	27.2
C2	2.1	104-149	53-74	33.6
D2	2.2	104-149	44-53	28.8
E2	2.0	104-149	0-44	16.0
F2(Check)	2.6	...-...	...-...	4.0

Difference in mortality required for significance at odds of better than 20:1, 18.5

any of the samples were significantly better than any of the others and could not be positively differentiated. Sample E2 did not even show a significant mortality rate over that of the check, though all other samples of the series did.

The feeding rate among this series of treated tests was very consistent, all of the tests permitting about the same amount of feeding. The untreated check had a much higher feeding rate than any of the treated tests, and this indicates again that the dusts cause some repellency to the beetles. Since the least significant difference between mortalities, which was statistically computed, was greater than any of the differences between the actual mortality figures of any of the tests, it was not possible to draw significant conclusions concerning the effect of coverage and adherence to mortality as opposed to particle-size effect on mortality. But the indications are that particle size has more effect on mortality than does coverage and adherence, as was the case in Series One. Since the tests of Series One showed significantly that the particle size of DDT used as the toxicant of the dust sample in Series Two was one of the least desirable sizes, it might be anticipated that this DDT size would show up better tested with other particle-size ranges of the diluent. In other words, that the particle size of the diluent would have some effect in giving significant differentiations to mortalities. However, this was not the case, which would indicate that the particle size of the diluent does not have the pronounced effect on mortality that the particle size of the toxicant does, although there is some effect indicated.

Table V presents the average net mortality of bean beetles for the four dust samples in the third series of experiments. These samples were composed of the smallest particle-size range of DDT, 0-44 microns, in combination with all other *Pyrax ABB* particle sizes except the 0.44 micron size range, and the untreated check. The mortality data show that sample A3, representing a combination of the smallest-sized DDT particles, 0-44 microns, with the largest-sized particles of *Pyrax ABB*,

TABLE V. The effect of particle size of a two per cent DDT dust in *Pyrax ABB* of Series Three on the feeding and mortality of Mexican bean beetle adults.

Sample	Feeding Rate 24 Hours	Micron-size Range		Per Cent Mortality 72 Hours
		DDT	*Pyrax ABB*	
A3	2.1	0-44	104-149	12.8
B3	2.1	0-44	74-104	11.2
C3	2.1	0-44	53-74	8.8
D3	2.0	0-44	44-53	11.2
F3(Check)	2.3	...-...	...-...	0.0

Difference in mortality required for significance at odds of better than 20:1, 8.2.

104-149 microns, was the best. Sample B3, representing a combination of DDT and *Pyrax ABB* of particle size ranges 0-44 and 74-104 microns respectively, was the next best sample. Sample D3, representing a combination of DDT and *Pyrax ABB* of particle-size ranges 0-44 and 44-53 microns respectively, produced the same kill as sample B3. Sample C3 representing a combination of DDT and *Pyrax ABB* of particle-size ranges 0-44 and 53-74 respectively, was the worst sample. However, the difference between the mortalities of any one of the samples was very small; and although the overall data were significant at odds of better than 20:1, the least significant difference of 8.2, which was computed statistically, between mortalities was greater than any of the differences between the actual mortality figures of any of the tests except the check. Therefore, it was not possible to draw significant conclusions concerning the effects on mortality of the various samples tested.

The feeding rate among this series of treated tests was almost identical in all cases; but the untreated check again permitted more feeding, indicating that the dust samples had some repellency effect on the beetles. As in the cases of Series Two, apparently the particle size of the diluent did not have the effect on mortality that the particle size of the toxicant had, because of the lack of significance between mortalities. It was not possible to draw significant conclusions concerning the individual effects of particle size and coverage in relation to mortality.

Another factor entered into this series of tests which makes all of the data recorded suspect despite the overall statistical significance. The DDT particles of this series were so small they tended to cling together and ball up on mixing. This caused lumps and an uneven distribution of the DDT throughout the diluent. This would cause a change in some of the constant factors of the tests, creating erroneous conclusions.

Table VI presents the average net mortality of bean beetles for the four dust samples in the fourth series of experiments. These samples were composed of a combination of the next to the smallest particle-size range of DDT, 44-53 microns, and the indicated particle-size ranges of *Pyrax ABB*, and the untreated check. The mortality data show that sample D4, representing a particle-size range of the next to the smallest, 44-53 microns, for both the DDT and the *Pyrax ABB*, was the best. Sample A4, representing a combination of DDT and *Pyrax ABB* of particle-size ranges 44-53 and 104-149 microns respectively, was the next best sample. Sample C4, consisting of DDT and *Pyrax ABB* of particle-size ranges 44-53 and 53-74 microns respectively, was the next best sample. Sample E4, consisting of DDT and *Pyrax ABB* of particle-size ranges 44-53 and 0-44 microns respectively, was the worst sample. However, although the overall data were significant at odds better than 20:1, the least significant difference of 15.2 required between mortalities was greater than the differences between the actual mortality figures of any of the samples. Therefore, it was not possible to draw significant conclusions concerning the effects of the various samples on mortalities, except in the case of the check. All of the samples except E4 were significantly better than the check. The feeding rate for all of the samples was very nearly the same; and as was the case in the other series of tests, the untreated checks allowed more feeding than did the treated ones. This indicates that the dusts had some repellency effect on the feeding of the beetles. Also, since there was no difference between the sample mortalities, it can be concluded from this series of tests as was concluded for Series Two and Three, that the particle size of the diluent does not have the effect on mortalities that the particle size of the toxicant does, although some effect is indicated. It was not possible to draw significant conclusions concerning the different effects of particle size and coverage in relation to mortality.

TABLE VI. The effect of particle size of a two per cent DDT dust in *Pyrax ABB* of Series Four on the feeding and mortality of Mexican bean beetle adults.

Sample	FeedingRate 24 Hours	Micron-size Range		Per Cent Mortality 72 Hours
		DDT	*Pyrax ABB*	
A4	1.8	44-53	104-149	20.0
C4	1.8	44-53	53-74	17.6
D4	1.8	44-53	44-53	27.2
E4	1.7	44-53	0-44	16.8
F4(Check)	2.5	......	..	2.0

Difference in mortality required for significance at odds better than 20:1, 15.2.

Conclusions

From the results obtained in these tests, the following conclusions may be drawn:

1. A two per cent dust sample composed of DDT of particle-size range 44-53 microns, in combination with *Pyrax ABB* of the same particle-size range, or a dust sample composed of the same materials each of a 53-74 micron particle-size range, was the most effective against adult Mexican bean beetle. In order of listing dusts composed of these same materials, each with respective particle-size ranges of 104-149 microns and 0-44 microns, were decreasingly effective in relation to the first two dust samples.

2. The coverage and adherence of any dust sample did not have the effect on mortality that the particle-size of the dust toxicant did.

3. The particle size of the DDT in a dust sample had more effect on mortality than did the particle size of the *Pyrax ABB*, although some effect by the latter was indicated.

4. The dusts had some repellency effect on the feeding of the bean beetles.

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