

STUDIES ON MOTHPROOFING OF WOOL

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Loose wool samples were treated with a range of concentrations of Dieldrin, Aldrin, Toxaphene and a newly developed insecticide of the chlorinated hydrocarbon type, Petkolin A. The resistance of these samples against the insect *Anthrenus vorax* was estimated in terms of feeding damage and mortality. The fastness of the treatments to the various conditions of use, washing, dry-cleaning, sunlight and daylight was also evaluated.

Wool is prone to be damaged by a number of insects, the prominent among which are the webbing clothes moth (*Tineola bisselliella*), the case-bearing clothes moth (*Tinea pellionella*) the black carpet beetle (*Attagenus piceus*) and some other beetles of genus *Anthrenus*, such as *Anthrenus vorax* and *Anthrenus schrophulariae*. Distribution of these and other insects in the various geographical regions is not uniform¹ and it is desirable to adopt measures of mothproofing with reference to special situations.

With the emergence of the woollen industry in developing countries, the quality of products is also being constantly improved. At present very little mothproofing is being done and the need for introducing effective and economical methods is evident.

For the control of damage caused by these insects, a few insecticides, particularly Dieldrin have found use.² Investigations of other potential compounds with special emphasis on economy would be of much assistance to the textile industry. Incidentally, the P.C.S.I.R. has evolved some new insecticides of the chlorinated hydrocarbon type,³ obtained from petroleum hydrocarbons boiling in the range of 35 to 135°C. These fractions are chlorinated at a temperature not exceeding 30°C till a product of specific gravity 1.40-1.45 results. The pilot plant production of the product is presently under investigation at the P.C.S.I.R. Laboratories, Karachi. Of the products obtained Petkolin A is the more stable one.⁴ It is desirable to evaluate this insecticide, in comparison to others, for its textile application.

A preliminary survey of a number of localities in West Pakistan indicated that the carpet beetle *Anthrenus vorax* was the most common wool pest in the region. A number of recent studies have particularly concentrated on *Tineola bisselliella*^{5,8} *Tinea pellionella*⁶ and *Attagenus piceus*⁹ but detailed studies on the control of damage by *Anthrenus vorax* seem to be scarce and hence the need for such an investigation.

The objectives of the present paper were thus to evaluate the comparative efficiency, from the textile standpoint, of some of the commonly available insecticides and also of the newly developed Petkolin A, employing *Anthrenus vorax* as the test insect. More particularly, the fastness of the insecticides to the various conditions of use viz. washing, dry-cleaning, daylight and sunlight was evaluated for the comparison.

Materials and Methods

Insects.—The larvae of *Anthrenus vorax* from our own stock culture maintained under natural conditions at Peshawar were used. All the experiments were conducted in March and April (in 1967 and again in 1968), when the activity of the larvae is at its maximum.

Insecticides.—Dieldrin was employed mainly as a standard for comparison. Two more commonly available chlorinated insecticides of the same class viz. Aldrin and Toxaphene were selected for investigation, the latter largely for being comparatively inexpensive. The newly developed Petkolin A was also included for its evaluation.

Wool Samples.—There seems to be a wide variation in the form of the samples prescribed or selected for investigations on mothproofing. Thus carpet pieces,¹⁰ various types of fabric^{7,8} and also of yarn⁹ have been used in testing for mothproofing. Even if fabrics only are employed in a number of studies, comparison of results can be very difficult, as the particular geometry of a fabric can play a significant role in comparative resistance to insect damage. Thus IWTO workers have been concentrating on detailed investigations to evolve procedures for comparing results obtained with a variety of fabrics with those obtained with certain fabrics as reference standards.⁷ Such methods are, however, cumbersome, limiting their application to the cases of dispute only.¹¹

For the purposes of the present investigation, raw wool samples, instead of any processed article, were employed, thus precluding any bias introduced by the geometry of the manufactured material. Further, to eliminate any bias due to the differences in the various loose wool samples, a single full fleece (of Hashtnagri type) was used in these experiments. The fleece was scoured with soap and soda, and after drying, was handcarded and conditioned to the room environment.

Treatment with Proofing Agents.—Dieldrin, Toxaphene and Aldrin were available from the manufacturers as emulsifiable concentrates, in concentrations of 20%, 80% and 40% respectively. For Petkolin A, the emulsifier was provided by the PCSIR. Laboratories, Karachi which was used in proportion of 55 a to 45 of the insecticide. The insecticides were applied to the wool samples using a 30:1 liquor to wool ratio at 40°C for 30 min followed by 30 min at the boil. Each insecticide was suitably diluted with water to give 0.05%, 0.5% and 5% compound on the weight of wool. For Dieldrin an additional treatment with 0.005% was also employed. After the treatment, the wool was rinsed in water for $\frac{1}{2}$ min and air-dried.

Evaluation of Insect Resistance.—The A.S.T.M's method D. 1116 was adopted,¹⁰ with appropriate modifications mentioned herein and elsewhere. The samples as treated above were placed in the cages together with 10 larvae of the beetle and both feeding damage and mortality were estimated weekly for a total period of 4 weeks. The damage can be evaluated by either weighing the excrement¹⁰ or the wool samples at the beginning and end of the experiment.¹⁰ In order to avoid any errors associated with the conditioning of the samples, the method of weighing the excrement was preferred. The preliminary trials indicated, however, that for the purposes of experiments on raw wool, damage also included fibrous mass simply cut into pieces by the larvae. Consequently all pieces 1 mm in length or shorter were included in the feeding damage. This resulted in slightly higher estimates of damage at all levels of testing and this has to be kept in view in the following discussions. The ratio of the weight of these fibrous pieces to that of the real excrement was approximately 1:3.

Fastness to Washing.—A method essentially similar to AATCC handwashing method¹³ was employed. 10g wool was washed in a litre of 0.3% solution of Walopal (a non-ionic washing agent from Continental Chemical Industries, Karachi) at 41°C for 15 min, rinsed twice in 41°C water for 1 min and air dried. This was repeated 5 times.

Fastness to Dry-cleaning.—10 g wool was rotated in 0.3 litre benzene at 32°C for 15 min followed by squeezing and air drying. This was also repeated 5 times.

Fastness to Daylight.—The standard procedure for assessment of colour fastness to indirect sunlight¹⁴ was adopted. In order to investigate the effectiveness of the treatments over a long time, the exposure test was carried out rather rigorously as it continued for a full year. In addition to the samples simply treated with the insecticides, samples already tested for fastness to washing and dry-cleaning were also included in these tests, thus providing combinations of the corresponding conditions.

Fastness to Sunlight.—The samples subjected to indirect daylight test were further subjected to test for fastness to sunlight. The standard method for assessment of colour fastness to direct sunlight¹⁵ was adopted. It may be pointed out that this is by far the most severe laboratory test among the various fastness tests. The test was, therefore, continued for a period of 10 days only to make the comparison possible, otherwise any longer exposures could have practically eliminated all or most of the proofing effect, especially in the case of samples treated with low concentrations of the insecticides.

Results and Discussion

The results have been summarised in Tables 1–4 corresponding to the four insecticides. The effect of increasing concentrations of the compounds on the feeding damage has been illustrated for example in Fig. 1 in the case of samples treated with Dieldrin and also of those washed and dry-cleaned subsequently.

Original Treatment.—A comparison of the tables reveals that at almost all the levels of concentration, the order of effectiveness of the insecticides is Dieldrin, Aldrin, Petkolin and Toxaphene. The feeding damage in the case of 0.05% concentration was 4 mg, 12.4 mg, 13.1 mg and 16.2 mg respectively, whilst for untreated samples the damage was 61.4 mg. The difference in effectiveness is more marked at the lower levels of concentration; for higher concentrations viz. 0.5% and above, the distinction between the efficiencies becomes less obvious.

It may be pointed out that the relationship between decrease in the feeding damage and increase in the dosage of insecticide is not direct. Williams⁸ obtained an approximately linear relationship between feeding damage and log con-

TABLE 1.—FEEDING DAMAGE AND MORTALITY IN THE CASE OF TREATMENT WITH DIELDRIN.

Treatment	Concentration of Dieldrin (%)							
	0.005		0.05		0.50		5.00	
	Damage mg	Mor- tality %	Damage mg	Mor- tality %	Damage mg	Mor- tality %	Damage mg	Mor- tality %
Original	14.3	83	6.4	100	1.8	100	0.5	100
Washed	23.6	75	11.2	95	3.3	100	0.6	100
Dry-cleaned	21.5	83	10.8	88	2.9	100	1.5	100
Exposed to daylight	26.4	73	13.7	100	4.1	100	0.8	100
Exposed to sunlight	37.4	85	19.3	95	3.6	100	1.4	100
Washed and exposed to daylight	34.3	73	12.6	98	2.9	100	2.7	100
Dry-cleaned and exposed to daylight	31.7	73	14.7	95	3.1	100	1.2	100

TABLE 2.—FEEDING DAMAGE AND MORTALITY IN THE CASE OF SAMPLES TREATED WITH ALDRIN.

Treatment	Concentration of Aldrin (%)					
	0.05		0.50		5.00	
	Damage mg	Mortality %	Damage mg	Mortality %	Damage mg	Mortality %
Original	12.4	95	3.2	100	0.8	100
Washed	20.6	93	4.0	98	1.1	100
Dry-cleaned	17.5	93	5.6	100	0.8	100
Exposed to daylight	24.2	100	7.5	100	1.3	100
Exposed to sunlight	28.3	95	9.6	100	2.3	100
Washed and exposed to daylight	24.1	95	5.7	98	3.6	100
Dry-cleaned and exposed to daylight	23.7	87	6.6	100	1.4	100

TABLE 3.—FEEDING DAMAGE AND MORTALITY IN THE CASE OF SAMPLES TREATED WITH PETKOLIN A.

Treatment	Concentration of Petkolin %					
	0.05		0.50		5.00	
	Damage mg	Mortality %	Damage mg	Mortality %	Damage mg	Mortality %
Original	13.1	70	51	100	1.0	100
Washed	23.9	78	6.4	95	2.5	100
Dry-cleaned	19.6	68	4.8	88	0.8	100
Exposed to daylight	28.4	70	8.3	100	3.2	100
Exposed to sunlight	50.6	83	16.7	88	6.4	100
Washed and exposed to daylight	28.2	78	7.6	88	1.9	100
Dry-cleaned and exposed to daylight	30.4	65	6.5	98	2.3	100

TABLE 4.—FEEDING DAMAGE AND MORTALITY IN THE CASE OF TREATMENT WITH TOXAPHENE.

Treatment	Concentration of Toxaphene %					
	0.05		0.50		5.00	
	Damage mg	Mortality %	Damage mg	Mortality %	Damage mg	Mortality %
Original	15.2	82	4.6	100	0.5	100
Washed	17.4	75	4.4	100	0.6	100
Dry-cleaned	22.3	78	6.2	100	1.9	100
Exposed to daylight	27.6	68	6.5	93	3.1	100
Exposed to sunlight	58.2	80	20.1	100	5.8	100
Washed and exposed to daylight	29.4	65	6.8	88	3.2	100
Dry-cleaned and exposed to daylight	26.8	68	7.7	88	4.6	100

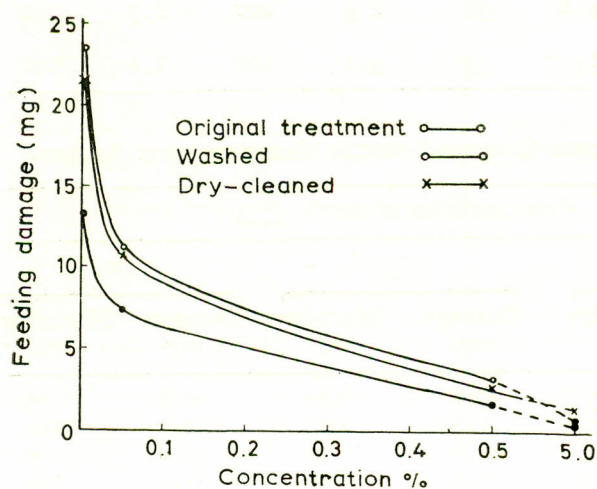


Fig. 1.—Relationship between concentration of Dieldrin and feeding damage.

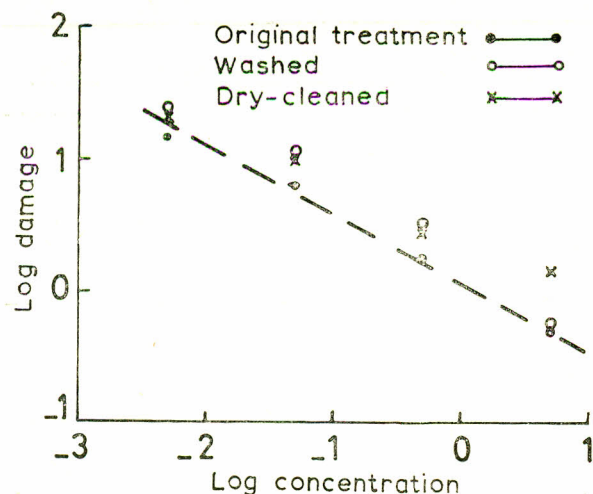
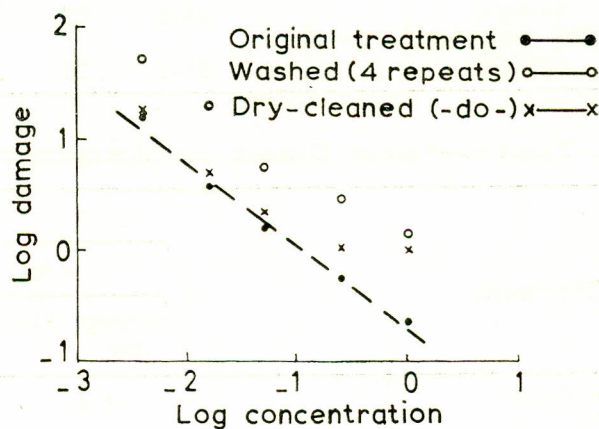


Fig. 2.—Logarithmic relationship between concentration of Dieldrin and feeding damage.

Fig. 3.—Logarithmic relationship between concentration of Dieldrin and feeding damage (Kuwana *et al*'s⁹ results).

centration. An investigation of the form of the relationship for the results of this study revealed that logarithmic curve of the form

$$Y = aX^n$$

seemed to be more suitable in expressing the relationship. Thus it could be expressed by

$$D = aC^n$$

where D = damage, C = concentration and a and n are constants. Employing this, straight lines (Fig. 2) were obtained for the curves of Fig. 1. Kuwana's⁹ data also resulted in straight lines (Fig. 3) when expressed by the above relationship. The relationship seems to be modified by treatments other than the original, such as washing, dry-cleaning etc., in which cases the deviations often become marked and irregular, although the general behaviour still corresponds roughly to the same pattern.

This would all seem to indicate, in agreement with previous studies,^{8,16} that with an initial increase in the dosage of the insecticide, the response would be appreciable but further increase in the dosage, beyond a certain optimum point, would improve the situation rather sluggishly. It would, therefore, seem to be a more reasonable course to apply an optimum quantity of insecticide and uneconomical high dosages may be avoided in general cases.

Washing Fastness.—There is an appreciable damage corresponding to the low dosages (0.05%) of all the insecticides except that of Dieldrin. For the purposes of ASTM's method D1116, against a damage of 61.4 mg on the untreated sample, a maximum damage of 20.4 mg will indicate a "resistant" sample. On this basis 0.05% treatment of Dieldrin and Toxaphene only are satisfactory, but for Petkolin A and Aldrin slightly higher dosages would have to be applied.

Dry-cleaning Fastness.—Dry-cleaning seems to have effects, in general, similar to washing. This is at some variance with the results of Kuwana *et al.*,⁹ who obtained a significantly higher damage for washed samples. It should, however, be pointed out that the washing treatment applied by us was less severe as compared to that of the said authors. A low dosage of Toxaphene (0.05%) seems to be particularly prone to dry-cleaning; similar dosages of the rest of the compounds come up to the "resistant" level.

Daylight Fastness.—It is interesting to note that the damage in the case of samples exposed to daylight for a full year was not conspicuously high, although with the exception of Dieldrin, a minimum satisfactory dosage in the case of the other three compounds seems to be 0.1%. This provides hopes for the general effectiveness of mothproofing treatments for long periods especially in cold environments, where effects due to direct sunlight are not so severe.

Sunlight Fastness.—Sunlight has proved to be the most severe treatment, as expected, and even though the exposure was for 10 days only, the protection level of 0.05% treatments of Toxaphene and Petkolin A was reduced from about 75% for the original treatment to a nominal 5% approximately. In this regard Dieldrin and Aldrin are distinctly superior to the former two. This would seem to limit the use of certain insecticides and, in this instance, especially of Toxaphene where even a 0.5% treatment is hardly satisfactory. However, higher dosages of these compounds such as 1% seem to be quite effective and could be used if comparative prices would permit this.

Mortality.—Almost all the above treatments indicate high mortality (Tables 1-4 at the end of the 28-day test, even though some of the treatments were not so effective on the basis of damage results. This is not surprising as in the case of the low dosages, the insects caused damage at the initial stages but gradually the treatments would have proved alien to their health, resulting in their ultimate demise. It may be pointed out that the results of such tests cannot be directly compared with practical situations, as in the former case the damage obtained may well be an outcome of the expedient conditions for the insects of having been closed within the test cage together with the treated sample, while they are not forced to live on such treated articles in common use.

As in the case of damage, Dieldrin has proved to be the most effective compound in bringing about the demise of the insects, whilst Aldrin is the next best. Toxaphene proved to be slightly more effective than Petkolin, which is in contradistinction to the damage results. On the basis of these results, approximate equivalents to 0.05% dosage of Dieldrin are 0.05% Aldrin, 0.5% Petkolin and 0.5% Toxaphene.

It may be pointed out that these results for mortality obtained with *Anthrenus vorax* are at some variance with those obtained by Kuwana *et al.*,⁹ who obtained lower mortality with equivalent low dosages of Dieldrin in the case of *Tineola bisselliella*. For instance, they obtained a mortality of 40-69% corresponding to a dosage of 0.062% Dieldrin, and no mortality at all in the case of washed or dry-cleaned samples for the same dosage. The difference may well stem from that in the insects under test which, although give rise to similar damage patterns, behave differently in terms of actual mortality.

Conclusions

The feeding damage decreases as the amount of insecticide increases, the dose-response relationship being logarithmic, approximating to the type $Y=aX^n$. Thus, additions beyond an optimum level of the compound result in smaller responses than expected and therefore excessive application beyond such a level is not economical.

Except for the extraordinary effectiveness of very small dosages of Dieldrin, there appear to be minor differences in the effectiveness of the four insecticides applied as far as their original application, washing, dry-cleaning and daylight fastness are concerned; but the significant difference is demonstrated in the case of sunlight where low degrees of Toxaphene and petkolin prove ineffective.

On an overall basis, approximate equivalents to 0.05% dosage of Dieldrin are 0.05% Aldrin, 0.5% Toxaphene and 0.5% Petkolin.

In cold climate, where effects due to direct sunlight are small, mothproofing could be effected, even with more economical dosages.

Even very small amounts of insecticides bring about a demise of the insects, eventually, and provide hope for some effectiveness in general use.

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References

1. Anonymous, Wool Science Reviews (International wool Secretariat, London, 1965), No. 27, p.1.
2. J.E. Lynn and J. J. Press, *Advances in Textile Processing* (Interscience, New York, 1961), volume 1, p. 213.
3. S. Siddiqui, S.A. Qureshi and S.H. Ashrafi, Pakistan Patent 114,302(1964); British Patent 1015,873(1964); Canadian Patent 717,114(1964).
4. S.A. Qureshi, personal communication.
5. M. Lipson and R. J Hope, *Proceedings of International Wool Textile Conference, Australia*, E523 (1955).
6. Z. Kuwana and S. Nakamura, *Textile Research J.*, **33**, 649 (1963).
7. J.H. Cole and F.G. Sarel Whitfield, *J. Textile Inst.*, **53**, 236(1962).
8. V.A. Williams, *Textile Research J.*, **35**, 1098 (1965).
9. Z. Kuwana, S. Nakamura and H. Sugiyama, *Textile Research J.*, **33**, 489 (1963).
10. *ASTM Designation: D1116-55T, ASTM Standards on Textile Materials* (American Society for Testing Materials, Philadelphia, 1961), second edition.
11. H. J. Hueck, *Ann. Appl. Biol.*, **46**, 511 (1958).
12. International Wool Textile Organization, Tentative Specification, Technical Committee of the I.W.T.O. Sub-committee on Larval Testing (1956).
13. American Association of Textile Chemists and Colourists, Tentative Test Method 88-1961T, *Am. Dyestuff Rep.*, **50**, 674 (1961).
14. British Standard 1006: 1961 (British Standards Institution, London).
15. British Standard 2661-1961:1956, (British Standards Institution, London).
16. Z. Kuwana and S. Nakamura, *Textile Research J.*, **33**, 751 (1963).