

Evaluation of permethrin in industrial application on wool against *Tinea translucens* and *Anthrenus flavipes*

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The effectiveness of permethrin in industrial applications on wool was studied against the larvae of *Tinea translucens* Meyrick (the tropical case-making clothes moth) and *Anthrenus flavipes* Le Conte (the furniture carpet beetle). Permethrin at 0.1% (on wool weight) was applied on fabrics at the dyeing and last scouring stages, and on wool tops at the dyeing stage in two woollen mills. Permethrin-treated fabrics were found insectproof after 10 hand /machine washings or 10 drycleanings or 28 days of sunlight exposure. But fabrics treated at 50-55°C and 70-75°C in the last scouring stage were not found insectproof after 10 drycleanings and 20 hand washings respectively. The treatment at higher temperatures (above 60°C) showed better exhaustion and fastness of permethrin on the fabrics. About 1.5-10.5% of applied permethrin was lost in three and a half years storage in various climatic conditions of the country. The maximum loss of permethrin occurred in hot and high humid places. The treated fabrics and garments were found safe to the users.

Keywords: *Anthrenus flavipes*, Permethrin, *Tinea translucens*, Wool fabric

1 Introduction

Certain insect species of beetle family, Dermestidae (Coleoptera) and moth family, Tineidae (Lepidoptera) are able to feed and derive nourishment from animal polymer like keratin. Their larval feeding on woollen textiles and fur- and feather-lined clothing have resulted in heavy losses in army depots as well as in industrial, commercial and domestic stores in India. For instance, insects have damaged barrack blankets worth Rs 1.4 million in an army depot in Kanpur¹. Among these insect species, *Tinea translucens* Meyrick (the tropical case-making clothes moth) and *Anthrenus flavipes* Le Conte (the furniture carpet beetle) are serious pests of wool².

There was no suitable chemical for durable protection of wool fabrics and garments from insects' attacks after the ban on dieldrin (an organochlorine insecticide) in the seventies in India and elsewhere³. Subsequently, a number of chemicals were evaluated in various laboratories for their insectproofing activity of wool fabrics. Of these chemicals, permethrin (a synthetic pyrethroid) has shown, in laboratory studies⁴⁻⁸, a great promise of meeting the criteria which a candidate insectproofing agent must satisfy⁹. In a mill trial application of permethrin on carpet, Byrne *et al.*¹⁰

found it suitable for the insectproofing of carpets which require low wet fastness. However, no such study has been carried out for fabrics which require high wet fastness, especially in Indian conditions. It has been observed that the effectiveness of an insectproofing agent depends upon various factors such as the application methods and manufacturing processes, care claims of the fabrics and garments, insect species and its population level, and climatic conditions¹¹.

In this paper, we report the results of mill trials with permethrin for insectproofing of woollen fabrics which are frequently cleaned and thus require high wet fastness. Permethrin was applied on wool tops at the dyeing stage and on fabrics at the dyeing and last scouring stages and its fastness on fabrics was studied after several washings/drycleanings, sunlight exposure, storage and users' wearing.

2 Materials and Methods

2.1 Materials

Wool fabric containing 100% Indian virgin wool of 40-45s and weighing 700 g/m² was used. In tops dyeing treatment, scoured, carded Merino wool of 64s was used.

Permethrin [3-phenoxybenzyl (1R1S)-*cis-trans*-2, 2-dimethyl-3-(2, 2-dichlorovinyl)cyclopropane-

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carboxylate] with *cis/trans* ratio of 40/60 was used in the form of a 25% (w/v) self-emulsifiable concentrate containing 7% (w/v) ionic and non-ionic emulsifier (ethylene oxide condensate) and 1% stabilizer in a solvent supplied by M/s Bharat Pulverising Mills Ltd, Bombay.

2.2 Methods

2.2.1 Application of Permethrin to Wool Fabric and Wool Tops

Permethrin treatment trials were carried out at two woollen mills located in Kanpur and Ludhiana. Permethrin at 0.1% on wool weight was applied during dyeing of wool fabric and tops and at the last scouring stage of fabric. The details of permethrin treatments are given in Table 1. Before adding to the treatment baths, permethrin was diluted with 3 times water. This solution was added to the cold bath (40-50°C) having previously wetted fabrics and tops and then circulated for 6-7 min. Lastly, dye and auxiliaries were added to the dyebath and the temperature was raised to boiling (90-95°C) in 30 min. In scouring application, a permethrin premix solution was added to bath having previously scoured, wetted fabric and no detergent. The temperature of the treatment bath was then raised to 70-75°C where it was maintained for 45 min. In one set of scouring treatment, temperature was maintained at 50-55°C. The dyebath-treated material was rinsed twice with water and then hydroextracted, while the fabric treated at scouring stage was only hydroextracted. Dyed tops after hydroextraction were sun-dried for 3-4 days before spinning.

2.2.2 Larval Feeding Test (Bioassay)

Larval feeding tests were carried out with 10 weeks old larvae of *Anthrenus flavipes* and 25-27 days old larvae of *Tinea translucens* at $27 \pm 1^\circ\text{C}$ and $65 \pm 5\%$ RH following the standard proce-

dures¹² where the assessment of fabric damage was made on the basis of fabric weight loss and visible damage to the naked eyes. A fabric is considered proofed to the larvae of textile pests if the mean weight loss of four test samples is less than 15 mg or 20 mg in any one of the samples and/or no surface cropping and holes are visible to the naked eyes. In control samples, the mean weight loss of four samples should not be less than 35 mg or 25 mg in a single sample. Also, not more than 25% of larvae should die or pupate during the experiment of 14 days.

2.2.3 Estimation of Permethrin Residue on Wool Fabric and in Spentbath Liquor

After conditioning at $27 \pm 1^\circ\text{C}$ and $65 \pm 5\%$ RH for 12 h, about 10 g of permethrin-treated wool material was Soxhlet extracted. Extraction was done with 2-methoxyethanol for about 6 h at a rate of 6 solvent exchanges/h. Permethrin was estimated using a gas chromatograph equipped with a flame ionisation detector. The other conditions were as follows: column—a glass column with 2% OV-17 on Chromosorb W AW, DMCS, 1 metre, 3 mm i.d.; carrier gases flow: nitrogen—76 ml/min, hydrogen—40 ml/min, air—300 ml/min; oven temp., 263°C; detector and injector temp., 290°C. At these conditions, the retention time for permethrin was 250 sec and the sample analysis time, 18 min.

Permethrin from spentbath liquor was extracted by saturating 100 ml of liquor with sodium sulphate and shaking it with 10 ml xylene for 2 h. The organic phase was recovered and analyzed by the gas chromatograph with conditions as given above.

2.2.4 Fastness Tests

The hand washing and drycleaning were done as described earlier¹³ and exposure to sun light

Table 1—Details of permethrin treatment

Treatment stage	Permethrin applied % oww	Wool-to-liquor ratio	pH of bath	Duration of treatment min	Temp. of treatment °C	Permethrin recovered on		
						Unfinished fabric % oww	Spent liquor % w/v	Finished fabric % oww
Fabric dyeing ^a	0.1	1 : 20	6	45	Boil	0.094	0.0005	0.085
Tops dyeing ^b	0.1	1 : 30	4	45	Boil	0.071	0.0008	0.051
Last scouring	0.1	1 : 20	6	45	50-55	0.05	0.0015	0.047
	0.1	1 : 20	6	45	70-75	0.068	0.0008	0.064

^aDye—2% MTR Zadaloon (Navy blue colour); Auxiliaries—0.5% Amolan FP, 4% Ammonium sulphate, and 1% Acetic acid.

^bDye—Ranolan (O.G. colour); Auxiliaries—4% Glauber salt; 2% Ammonium sulphate and 1% Acetic acid.

was done as per the Indian standard specifications¹⁴. Machine washing was done with an automatic Saviour AX 303 using 0.2% neutral soap.

2.2.5 Toxicity of Treated Wool Material to Adults of Pest Species

The toxicity of treated wool material to adults of *Anthrenus flavipes* and *Tinea translucens* was determined according to the method given by Bry *et al.*⁶ where 2-3 days old 10 adults of test species were released in a 0.25 l glass jar on a sample of 5 cm diam which substantially covered the bottom of the jar. The mouth of the jar was covered with a piece of muslin cloth held by a rubber band. This was replicated four times. Controls consisted of untreated fabrics only. The jars were kept in a dark cabinet at $27 \pm 1^\circ\text{C}$ and $65 \pm 5\%$ RH and the adult mortality was determined after 24, 72 and 96 h of exposure. The jars were then returned to the holding cabinet for an additional 6 weeks to determine whether any adult was able to oviposit.

2.2.6 Storage Stability of Permethrin on Treated Fabrics

The stability of permethrin on wool fabrics was determined by storing them for more than 3 years in seven stores located in different parts of the country having climatic conditions varying from extreme cold to extreme hot and moist to dry. Aged wool fabrics were tested both biologically and chemically to determine their insectproofness and for the degradation of the compound.

2.2.7 Users' Acceptance Trial of the Treated Fabrics

A users' acceptance trial was carried out by wearing treated fabrics by the volunteers for one year. The users' acceptability was recorded in prescribed questionnaires. Used fabrics were bioassayed and chemoassayed.

3 Results and Discussion

A total of 6 trials were carried out in two mills. Checks for the levelness of permethrin application were made by chemical analysis on unfinished and finished wool fabrics. Permethrin residue recovered on treated wool fabrics and in spentbath liquor are shown in Table 1. It is observed that in dyebath treatment, 71 and 94% of applied amount of permethrin was exhausted onto the tops and fabrics respectively, but only 51 and 85% of permethrin was recovered on finished fabrics in tops and fabric treatment respectively. Thus, the maximum loss of permethrin (about 20%) was observed in tops treatment; this loss occurs mainly during sun drying, spinning and

weaving processes. In fabric treatment, 9% of permethrin was lost. In scouring treatment, 50-68% of the applied amount of permethrin was recovered on unfinished fabrics and slightly less (about 2-3%) permethrin was estimated on finished fabrics. At lower temperature (50-55°C), permethrin exhaustion on fabric was 50% which is 18% less than that at slightly higher temperature (70-75°C).

Thus, about 26% more permethrin is exhausted on fabric at fabric dyeing stage than at scouring stage. This is mainly due to the high temperature in dyebath which gives better penetration of permethrin into the wool fibres. In top dyeing treatment, 20% permethrin was lost between the unfinished and finished fabric stages. This loss of permethrin may be mainly due to sun drying of tops. Duffield⁵ has also reported that about 23% of applied permethrin was decomposed in dyebath treatment.

Permethrin residues in spentbath liquors were 0.0005 and 0.0008% (w/v) for fabric and tops dyebath treatments respectively. In scouring treatments at 50-55°C and 70-75°C, permethrin residues were estimated to be 0.00015 and 0.008% (by wt) respectively.

3.1 Fastness of Permethrin on Treated Fabrics

Data on permethrin residues recovered on treated fabrics after various fastness tests are given in Tables 2 and 3. It is observed that all the treated fabrics were found insectproof against the larvae of both the pest species after various fastness tests, except the 20 times washed fabric treated at scouring stage and 10 times drycleaned fabric treated at 50-55°C in scouring stage. The chemical analysis showed that 0.008% (owf) permethrin was present on 10 times drycleaned and 20 times washed fabrics (Tables 2 and 4). It is interesting to note that a small amount of permethrin (5-8%) was lost in dyebath-treated fabrics during drycleaning while the loss was 61-62% in tops treated fabrics. This difference may be due to the presence of some grease in wool which absorbed permethrin and was removed in drycleanings. The same effect was also reported by Byrne *et al.*¹⁰.

3.2 Storage Stability of Permethrin

Fabrics treated at dyebath and scouring stages were stored in 7 different stores for three and a half years. The chemical analyses of the stored fabrics showed that 1.5-10.5% of permethrin was lost during exposure in these stores (Table 5). The maximum amount of permethrin (8-10.5%) was lost on stored fabrics in hot and high humid places like Jodhpur and Silcher.

3.3 Toxicity of Treated Fabrics to Adults of Pest Species

Samples of fabrics treated at dyebath and scouring stages were exposed to the adults of pests and the results are shown in Table 6. All the adults of *Tinea translucens* died within 24 h of exposure. In case of *Anthrenus flavipes*, 85-91% adults were knockdown but no mortality was observed after 24 h exposure, 6-10% adults died after 48 h exposure and all the adults died after 96 h exposure.

On exposure to 10 times drycleaned treated fabrics, 72-92% adults of *Tinea translucens* died

Table 2—Permethrin levels in fastness tests on fabric treated with permethrin (0.1% on wool weight)

Fastness test	Permethrin (% oww) recovered on treated fabric		
	Fabric dyeing	Tops dyeing	Last scouring
Hand wash			
5	0.072 (15.2)	0.038 (25.4)	0.039 (39)
10	0.061 (28.2)	0.028 (45)	0.036 (43.7)
20	0.028 (67)	0.012 (76.4)	0.008 ^a (87.5)
Machine wash			
5	0.066 (22)	0.030 (41.1)	0.034 (46.8)
10	0.05 (41.1)	0.021 (48.8)	0.020 (68.7)
Drycleaning			
5	0.08 (5.8)	0.0196 (61.5)	0.035 (45.3)
10	0.078 (8.2)	0.019 (62.7)	0.018 (71.8)
Sunlight exposure,			
16 weeks	0.045 (47)	0.030 (41.1)	0.03 (53.1)
Wearing for			
one year	0.065 (23.5)	—	0.042 (34.3)

Figures in parentheses show the percentage loss of initial residue of permethrin on finished fabric.

^aNot proofed against the larvae of *Tinea translucens* and *Anthrenus flavipes*.

Table 3—Permethrin residue on treated fabric at last scouring stage at 50-55°C after hand wash and drycleaning

Fastness test	Permethrin residue % oww
Nil	0.047
Hand wash	
5	0.021
10	0.018
Drycleaning	
5	0.015
10	0.008 ^a

^aNot insectproofed

within 24 h and all died within 48 h. No adult mortality of *Anthrenus flavipes* was observed after 48 h exposure, but 72-100% adults were knockdown. All adult beetle died within 96 h of exposure. After 6 weeks exposure, no live or dead larva of pest species was observed. How-

Table 4—Bioassay of permethrin-treated fabric at last scouring stage at 50-55°C after hand washings and drycleanings

Fastness test	Visible damage		Wt. loss mg	Assessment
	Cropping ^a	Hole ^b		
<i>Tinea translucens</i>				
Nil	1	A	0.48	Proof
Hand wash				
5	1	A	0.375	Proof
10	1-2	A	5.09	Proof [*]
Drycleaning				
5	1-2	A	5.57	Proof
10	2-3	B	15.87	Not proof
Control	3-4	B-C	41.34	
<i>Anthrenus flavipes</i>				
Nil	1	A	1.17	Proof
Hand wash				
5	1-2	A	4.51	Proof
10	1-2	A	4.96	Proof
Drycleaning				
5	1-2	A	9.91	Proof
10	3	B	48.20	Not proof
Control	4	D	104.3	

^aCropping (Surface damage): 1—Not detectable; 2—Very slight; 3—Moderate; 4—Very heavy.

^bHole: A—No detectable damage; B—Yarns partially severed; C—Few small holes; D—Several large holes.

Table 5—Permethrin residue on wool fabrics stored in different climates of the country for three and a half years

Locality	Climate	Permethrin recovered (% oww) on	
		Dyed fabric	Scoured fabric
Kanpur	Tropical	0.083	0.062
Jodhpur	Hot dry		
	tropical	0.078	0.061
Silchar	Humid tropical	0.076	0.061
Bombay	Tropical	0.082	0.062
Pathankot	Tropical	0.079	0.063
Srinagar	Temperate	0.080	0.061
Gwalior	Tropical	0.078	0.061

Table 6—Effect of exposing adults of *Tinea translucens* and *Anthrenus flavipes* to wool fabrics treated at dyeing and last scouring stages initially and after 10 drycleanings

Fabric	After 24 h exposure		After 48 h exposure		After 96 h exposure	
	Knockdown ^a	Dead + Moribund ^b	Knockdown ^a	Dead + Moribund ^b	Knockdown ^a	Dead + Moribund ^b
<i>Tinea translucens</i>						
<i>Treated at</i>						
Dyeing stage	0	100	0	100	0	100
Last scouring stage	0	100	0	100	0	100
Dyeing stage and drycleaned 10 times	8	92	0	100	0	100
Last scouring stage and drycleaned 10 times	18	72	0	100	0	100
<i>Anthrenus flavipes</i>						
Dyeing stage	91	0	90	10	0	100
Last scouring stage	85	0	78	6	0	100
Dyeing stage and drycleaned 10 times	60	0	100	0	0	100
Last scouring stage and drycleaned 10 times	35	0	72	0	0	100

^aKnockdown—When adults become immobilized after exposure.

^bMoribund—When adults after knockdown become stationary and respond to probing by movements of legs, mouth parts or antennae.

ever, a few dead eggs of moth species were found on samples. In controls, several live larvae of both the pest species were present.

3.4 Users' Acceptance Trial of Treated Fabrics

The treated fabrics were used by the volunteers for one year and were found safe to users. About 24-34% of permethrin was lost during wearing.

4 Conclusion

Permethrin at 0.1% (on wool weight) can effectively protect the wool fabrics and garments throughout their useful life from the larval feeding of *Anthrenus flavipes* and *Tinea translucens* when it is applied on tops at the dyeing stage and on fabrics at the last scouring stage of fabric manufacturing. However, for the treatment of fabric at the dyeing stage, less than 0.1% oww permethrin may be applied for insectproofness when there is no wet process after the treatment.

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