Efficacy of Home Laundering in Removal of DDT, Methyl Parathion and Toxaphene Residues from Contaminated Fabrics

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Cotton fields in Louisiana are heavily sprayed with chemicals to control the pests which attack this crop. The use of these chemicals causes serious problems for man and other non-target organisms. The potential danger of these chemicals in contaminating clothing has received little attention. The work of FINLEY and ROGILLIO (1969) demonstrated that fabric in trousers worn by insect scouts for 8 hours "picked up" DDT and methyl parathion residues from contact with cotton plants. After 1 washing, the average residue retention of DDT and methyl parathion for these fabrics was 30.4 and 2.6%, respectively. The biological significance of residues present in the fabrics was not investigated. QUINBY et al. (1958) reported that cotton scouts working in fields treated with methyl parathion collected on all garments an average of 3 mg/hr. in the first hour after spraying and less than 1 mg/hr. 4 hours after spraying. WARE et al. (1973) published data on the presence of insecticide residue in clothing of cotton scouts that was in general agreement with that of QUINBY et al. (1958).

Because of the urgent need for additional information on the efficacy of home laundering on the removal of insecticide from contaminated fabrics, this research reported herein was undertaken. The objectives of this study were to: 1) test the effectiveness of a series of washings to remove residues from cotton and cotton-polyester fabrics contaminated with methyl parathion and with a mixture of toxaphene-DDT-methyl parathion, 2) ascertain if residues remaining in the 2 fabrics after washing were biologically active, and 3) determine if washing contaminated fabrics with clean fabrics would result in contamination of the clean fabrics.

METHODS AND MATERIALS[1]

Fabrics and Fabric Clean Up. One all-cotton fabric and 1 cotton-polyester fabric in a 50/50 percent fiber blend were used.

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[1] For explicit details of the experimental procedure, see Master's thesis by Gwendolyn I. Metcalfe. 1973. Title, The Absorption and Retention of Selected Chlorinated Hydrocarbons and Organic Phosphate Residues in Cotton and Cotton-Polyester Fabrics. Louisiana State University, Baton Rouge, Louisiana, pp. 108.

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Fabrics made of these fibers are widely used for work-type clothing. White fabrics with no finishes were selected to minimize potential interference in the extraction and identification of insecticide residues. The fabrics were very similar in weave and weight. Sixty samples measuring approximately 51 x 56 cm and weighing approximately 30 g each were cut from each of the 2 types of fabric. All samples were given a "clean-up" wash treatment to remove any traces of starches, gums, or other foreign matter, then tumble dried in an electric dryer, folded and stored in coded plastic bags.

Treatment of the Fabrics With Insecticides. Fabrics of each type were treated with a solution of methyl parathion or toxaphene-DDT-methyl parathion diluted to the strength used for field application. One quart (946 ml) of formulated methyl parathion (4 lb/gal emulsifiable concentrate or 0.48 kg/l) was mixed with 1.75 gal (6624 ml) of tap water for a total volume of 2 gal (7570 ml) or the equivalent of 6.0% of methyl parathion. Similarly, 2 qt (1892 ml) of formulated (4 + 2 + 1 lb/gal or 0.48 + 0.24 + 0.12 kg/l emulsifiable concentrate) toxaphene-DDT-methyl parathion were mixed with 1.5 gal of tap water resulting in a concentration of 12.0, 6.0, and 3.0%, respectively of the pesticides. The fabric samples were thoroughly saturated for each treatment. Each sample was lifted from the solution (using disposable plastic gloves), squeezed lightly by hand, and hung on outdoor drying lines. After 1.5 hr of drying each fabric was folded, placed in a plastic bag, and stored in a refrigerator-freezer at a temperature of -5°C.

Description of Washer. A small electric portable washer, Kenmore^{R} Model 305-73471, equipped with automatic timer, removable aluminum agitator, and stainless steel fittings, was used for washing samples. The interior surface of the tub was porcelain enamel and the water capacity was 19.85 l. The washer was thoroughly cleaned with Tide^{R} and Liqui-Nox^{R}.

The Wash Cycle. An appropriate wash cycle, for the small washer, was established after trial tests using AATCC Test Methods 124-1967, 88B-1969, and 88C-1969 as guides (AATCC). The Standard Detergent of the AATCC, known formulation, type 124 with a pH of 10 was used for this study. Five g of the AATCC detergent was adequate per 1 gal (3785 ml) of water. The complete schedule was, Wash: 14 minutes in distilled water, 60°C using 26.22 g detergent; Deep Rinse: distilled water at room temperature for 10 minute agitation; drain, repeat rinse cycle twice.

Washing and Drying. A wash load was made up of 8 fabrics, including 2 treated cotton, 2 untreated cotton, 2 treated cotton-polyester, and 2 untreated cotton-polyester samples. Sets of fabrics were divided into 3 lots with 2 replicates per lot. The 3 lots of fabrics treated with insecticides were washed in a descending order of 3, 2, and 1 washes. Washing of fabrics

treated in the methyl parathion phase was completed before washing fabrics treated in the toxaphene-DDT-methyl parathion phase. After washing, each sample was lightly squeezed to remove water and the lot was then dried for 60 minutes in an electric dryer at a temperature of 63-68°C. The fabric samples were folded, wrapped with clean aluminum foil, and stored in a freezer at -5°C.

Preliminary Extraction Analysis. A preliminary extraction and analysis of fabrics was done to test the efficiency of the procedure for residue recovery from the samples. When methyl parathion alone was applied the recovery was 91 and 86% from the cotton and cotton-polyester fabrics, respectively. When the toxaphene-DDT-methyl parathion mixture was applied, the % recovery for DDT was 96% from the cotton fabric and 97% from the cotton-polyester fabric. Recovery of methyl parathion in the mixture was 61% from cotton and 75% from the cotton-polyester. Toxaphene recoveries in the mixture were not calculated due to the complexity of the toxaphene reaction. Reagent blanks were tested during the experiment to check purity of reagents, accuracy of procedures, and cleanliness of equipment.

Extraction of Residues. Chemical extraction procedures for recovery of residues from treated fabrics were similar to those used by FINLEY and ROGILLIO (1969).

Analysis of Residues. Analyses of the extracts were made by electron capture gas chromatography using a Varian-Aerograph Model 1200 instrument. The column was packed with 15% QF-1-10% DC-200 on Gas Chrom Q, operated at 185°C with nitrogen as the carrier gas. Standard solutions of toxaphene, DDT, and methyl parathion were injected after each test sample and analyzed to determine the sensitivity of the gas chromatograph at the time each test sample was injected. The standard check injection for all solutions was 5 µl. Extracts were diluted for injection into the gas chromatograph to adjust peak heights of the pesticides for close agreement with those of the standards. Methyl parathion and DDT were calculated using peak height measurements while toxaphene was calculated using area measurements.

The Statistical Analyses of Data. The data were analyzed statistically in 2 parts. A 2 x 4 factorial arrangement of treatments (2 fabrics x 4 levels of washing) in a completely randomized design, with 2 observations per treatment combination, was used for the toxaphene-DDT-methyl parathion and the methyl parathion phases. Three orthogonal comparisons (linear, quadratic, and cubic) were made to evaluate contamination levels by washing for each lot of the 2 fabrics. A 2 x 2 factorial arrangement of treatments was used to compare transfer samples with treated samples, for the 2 fabrics in a completely randomized design, with 2 observations per treatment combination.

Biological Assay. Drosophila melanogaster Meigen were used to determine if insecticide residues present in the fabrics after washing and extraction were biologically active. The sensitivity of Drosophila in bioassay experiments has been cited by HOSKINS et al. (1959); NAGASAWA (1959); and WYLIE (1956). The bottom and lid sections of disposable petri dishes, 100 x 20 mm, were lined with circles of fabric. The lids were pierced to provide ventilation.

Before placing the Drosophila in the petri dishes, approximately one-half teaspoon of canned pumpkin, freshly opened, was pressed and smoothed against the inside wall of each petri dish to provide food and moisture for the Drosophila during the 24-hour test period. Ten 2-day-old Drosophila were placed in each petri dish and the lid was securely taped. The petri dishes containing the Drosophila were held in an incubator at 27°C.

Tests were made to determine the concentration of methyl parathion and toxaphene-DDT-methyl parathion mixture which would kill 50% of the Drosophila. Circles of clean cotton and cotton-polyester fabric were immersed in solutions of methyl parathion ranging in concentrations from 60 to 0.0001 mg/ml, and dried overnight. The following morning petri dishes were lined with the insecticide-treated fabric and a test with Drosophila was begun within 1-2 hr. The estimated LD_{50} for methyl parathion on the cotton fabric under these conditions was 0.0024 mg/ml while the LD_{50} for cotton-polyester fabric was 0.0012 mg/ml. Similar tests with the toxaphene-DDT-methyl parathion mixture revealed an estimated LD_{50} 0.0024 - 0.0012 - 0.0006 mg/ml for both fabrics. Drosophila were not killed when placed on clean untreated fabrics and fabrics saturated with 65% acetonitrile and 35% water.

RESULTS AND DISCUSSION

Washing greatly reduced residues of toxaphene, DDT, and methyl parathion present in both cotton and cotton-polyester fabrics as shown in Table 1. However, there was no apparent difference between the 2 fabrics in the effectiveness of washing in removing these residues.

The removal of methyl parathion from the fabrics by the washing procedure may be explained in part by the alkalinity of the detergent (pH 10) since methyl parathion is hydrolyzed to p-nitrophenol and other materials in an alkaline medium.

Methyl parathion applied alone was much more effectively removed by 3 washings (8-60 fold more) than methyl parathion applied in a mixture with toxaphene and DDT. BRADLEY et al. (1972) demonstrated that toxaphene when applied with DDT to cotton reduced DDT residues in natural water run off by over 50% compared to residues for DDT when applied alone. Toxaphene apparently exhibited

TABLE 1

MEAN TOXAPHENE, DDT, AND METHYL PARATHION RESIDUES (PERCENT) IN ALL-COTTON AND COTTON-POLYESTER FABRICS BEFORE AND AFTER WASHING

Fabric (Washes)	Fabrics							
	Cotton				Cotton-Polyester			
	Mixture [1]			MP	Mixture [1]			MP
	Tox.	DDT	MP	Alone [2]	Tox.	DDT	MP	Alone [2]
0	15.65	4.62	2.28	3.19	18.10	7.12	1.92	4.31
1	4.00	1.71	0.49	0.59	5.74	2.41	0.58	0.31
2	4.98	1.85	0.21	0.04	4.07	1.38	0.07	0.01
3	4.46	1.49	0.07	0.001	3.21	0.91	0.04	0.005

[1] Fabric dipped in solution containing 0.25 lb methyl parathion, 0.5 lb DDT, and 1 lb toxaphene/gal of water (3.0, 6.0, and 12.0%, respectively).
[2] Fabric dipped in solution containing 0.5 lb methyl parathion/gal of water (6.0%).

similar activity on the 2 fabrics under study. A further indication of the difficulty in removing toxaphene residues was the fact that toxaphene residue levels in both fabrics decreased only slightly after the first washing (Table 1). DDT residues were also very difficult to remove from the 2 fabrics after the initial washing.

Residues of the 3 insecticides were transferred to "clean" cotton and cotton-polyester fabrics when they were washed with cotton and cotton-polyester contaminated fabrics (Table 2).

TABLE 2

MEAN RESIDUES (PERCENT) OF TOXAPHENE, DDT, AND METHYL PARATHION TRANSFERRED FROM CONTAMINATED FABRICS TO CLEAN FABRICS DURING WASHING

Fabric	Mixture			MP Alone
	Tox.	DDT	MP	
All-Cotton				
Contaminated	4.00	1.71	0.49	0.59
Transfer	0.09	0.04	0.01	0.04
Cotton-Polyester				
Contaminated	5.74	2.41	0.58	0.31
Transfer	3.66	1.71	0.31	0.03

When methyl parathion was applied alone to either fabric, there was no difference in the amount of residue detected in the cotton and cotton-polyester transfer fabric. The residue level in the transfer

fabrics amounted to about 10% of that in the contaminated fabrics. However, transfer of methyl parathion to cotton-polyester fabric was greatly enhanced by the presence of toxaphene and DDT. Methyl parathion residues extracted from the cotton-polyester transfer fabric were approximately 50% of those detected in the contaminated fabric. The residues detected in the cotton transfer fabric were similar to those found when methyl parathion was applied alone.

The residues of toxaphene and DDT detected in the cotton-polyester transfer fabric were about 67% of the levels in the contaminated fabric. However, residues of toxaphene and DDT detected in the cotton transfer fabrics were only about 2.5% of those found in the contaminated fabrics (Table 2).

Bioassays with Drosophila on insecticide-contaminated fabrics before and after each of the 3 washings showed that all flies confined on both fabrics contaminated with toxaphene-DDT-methyl parathion were killed in 24 hours.

All flies confined to fabrics treated with methyl parathion alone were killed after 2 washings on cotton fabric and after 1 washing on cotton-polyester fabric. Forty % of the Drosophila were alive after 24 hr of exposure to cotton fabric after 3 washings and cotton-polyester fabric after 2 washings. Sixty-five % of the flies survived the test period on the cotton-polyester fabric washed 3 times.

Bioassays of both "transfer" fabrics washed with fabrics treated with methyl parathion alone resulted in 100% mortality of flies. All of the flies exposed to the cotton-polyester "transfer" fabrics washed with the toxaphene-DDT-methyl parathion mixture treated fabrics were killed while none died when exposed to the cotton "transfer" fabric treated similarly.

Generally speaking, the data gathered in this study indicate 1) that laundering greatly reduces the amounts of insecticide residues present, 2) that insecticide-contaminated clothing should not be laundered with uncontaminated clothing, 3) that even 3 launderings were not effective in removing all residues of the 3 insecticides studied, 4) that residues of the chemicals studied remaining in clothing after 3 launderings are biologically active, and 5) that the bioassay data were in general agreement with the residue analyses using GLC. It is not known how many launderings would be required to remove all residues or if they could even be removed by laundering. Similarly, the optimum laundering procedure and materials for insecticide residue removal remains to be determined.

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