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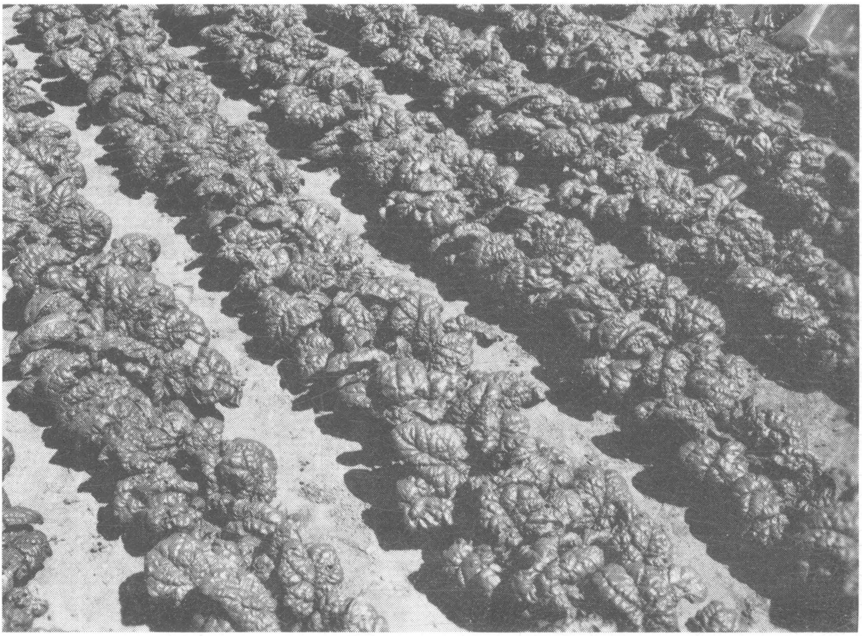
Removal of Malathion from Spinach by Commercial and Home Preparation Procedures

BENITA C. YAO and J. R. GEISMAN



OHIO AGRICULTURAL RESEARCH AND DEVELOPMENT CENTER
WOOSTER, OHIO

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BENITA C. YAO and J. R. GEISMAN

INTRODUCTION

Freedom of the food supply from chemical contamination is of major concern to everyone. Since 1947 there have been no records of illness or disease resulting from the proper use of agricultural chemicals (13). Proper use is defined as application according to label directions.

Both producers and consumers would prefer that the food they eat be free of chemical residues or that these residues be kept at a minimum. The Food and Drug Administration (FDA) conducts constant surveillance programs or market basket studies to assure the safety of the food supply.

Most fruits and vegetables undergo different unit operations for preparing and processing before actual consumption. These unit operations are essential for cleaning, eliminating waste, and making the raw materials into edible or more palatable foods. These procedures remove soil and insect residues. It appeared possible that they might also reduce or remove other undesirable materials, such as pesticide chemicals.

This study was undertaken to determine what effects various unit operations involved in processing a crop might have on chemical residue if the raw products should have such a residue. In order to obtain a residue, malathion was applied according to label recommendations to spinach and the crop was harvested at 1 and 3 days after application. While this time of harvest was before the recommended harvest date, it produced a residue which was reduced or removed by the unit operations in preparing and processing the crop.

Studies have shown the persistence of certain pesticide chemicals, particularly the chlorinated hydrocarbons. The quantities of these chemicals varied according to specific chemicals in different substrates (6). Spinach, with a convolute surface and a large surface area per unit weight ratio, presents a difficult washing problem. However, if a residue could be removed from this commodity, then the methods used would be appropriate to other crops.

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[1] Part of the data presented in this circular was collected by the Senior Author in connection with her Master of Science degree program at The Ohio State University.

REVIEW OF LITERATURE

Pesticide Residues

The use of pesticide chemicals in producing agricultural crops is a common practice. Their use has become indispensable as a means for increasing production, maintaining quality raw products, and lowering prices. However, the availability of a large number of synthetic organic pesticides, plus the extensive and widespread utilization of these chemicals, present the possibility of pesticide residues being incorporated into food products.

A survey of pesticide residues in market food samples conducted by the Food and Drug Administration in the United States over a period of 3 years (1964-66) indicated that half of the samples contained residues, 29% contained more than one kind of pesticide, and 20% had one kind of pesticide residue. About 1.5% of the domestic raw fruits, vegetables, and cereals contained pesticide residues in large quantities (2).

An analysis of residues from *total diet* samples examined each year in five geographic regions and 30 cities covering a period of 3 years indicated that a well-balanced diet in the United States contained the following pesticide chemicals (2):

Chlorinated organic chemicals	0.02 p.p.m.
Organic phosphate chemicals	0.003 p.p.m.
Chlorophenoxy chemicals	0.003 p.p.m.
Carbamate chemicals	0.05 p.p.m.

The average frequencies of the 14 most commonly found chemicals were reported. Malathion ranked sixth on the list. The average level of malathion in the diet was found to be 9 micrograms per day (2).

There are many sources from which raw agricultural commodities can accumulate pesticide residues. The major source is the residue remaining from direct application of pesticides while the crop is in the field. Another source could be from pesticide chemicals in the soil from previously treated crops, in which case the chemicals may migrate and be absorbed by the plants of succeeding crops. Other possible sources are through drift, misuse, and changes in the usual degradation of chemicals which result from weathering.

In the United States, the control of pesticide residues in food began as early as 1906 (3). The Pure Food and Drug Act of 1906 was replaced by the Federal Food, Drug, and Cosmetic Act of 1938. In 1954, the Miller Amendment was added. This law established safe levels of pesticide chemicals in raw agricultural commodities. In 1958, the Food Additive Amendment including the Delaney Clause was added. This included federal control of food additives and pesticide chemicals remaining in foods after processing. Then followed the Colley Amendment, which regulated the use of other chemicals such as plant growth regulators, nematocides, and herbicides (23).

Insecticides form one of the largest groups of pesticide chemicals. The two main classes of synthetic organic insecticides are the chlorinated hydrocarbons and organophosphorus compounds. Chlorinated hydrocarbons such as DDT, lindane, and dieldrin are characterized by their long persistence in plants and their tendency to accumulate in animal tissues. The organophosphorus compounds such as parathion, TEPP, malathion, and diazinon, on the other hand, decompose more rapidly and have less tendency to accumulate in mammalian tissues. Malathion superseded such compounds as parathion and TEPP because it is safer and has less toxic effect on mammals. For this reason, it has become the most commonly used insecticide in agricultural practices.

Malathion Residues on Plants

Malathion (S-(1,2-dicarboxyethyl) 0,0-dimethyl dithiophosphate) is a broad spectrum, general purpose, contact organophosphate insecticide. It was originally known as compound 4049 or malthon (16). The compound was first introduced for testing in 1950 by the American Cyanamid Company and was accepted for use in agriculture for treating fruits, vegetables, and field crops in 1959 (12).

The chemical and physical properties of malathion are well reviewed in many texts (16, 17). It is available as an emulsifiable concentrate, a wettable powder, dust, or in combination with other chemicals. The tolerance level in most raw agricultural products is 8 p.p.m. (18) and the accepted daily intake is 0.02 mg. per kg. of body weight (2). Malathion is noted for its specific toxicity to insects and its low toxicity to warm-blooded animals.

A number of research investigations on the behavior and degradation of malathion in plant products at field conditions have been undertaken. Residues remaining in fruits and vegetables vary with different formulations and dosages of applications. The rate of disappearance follows an exponential function (21).

Studies on residues in fruits and vegetables have been reported by Westlake and Butler (20). Their studies indicated rapid decomposition of malathion from fruits and vegetables. An initial deposit of 60.9 p.p.m. on spinach was reduced to 4 p.p.m. after 6 days and only a trace remained after 12 days.

Spiller described the behavior of pesticide deposits and residues as follows (16): ". . . there are two characteristic half lives, the shorter measuring the initial rapid dissipation of insecticide while it remains on the surface subject to physical and chemical diminution by weathering, the second and longer half life resulting from penetration of insecticide into the plant tissue where decrease in concentration is principally due to metabolic attack and to increase in plant size."

The persistence of residues in plants is related to many factors. First, it depends on the chemical properties of the pesticide chemicals. Weather conditions such as wind, rain, amount and intensity of light, air temperature, and humidity play an important role, too. Finally, the rate of growth, the rate of metabolism, the enzyme systems, as well as the shape and position of the edible portion of the plant have marked effects on the dissipation of pesticide residues (8). The influence of these factors on the rate of disappearance of malathion in fruits and vegetables has been thoroughly investigated by Koivistoinen (12).

Fruits and vegetables are usually trimmed, washed, and may be cooked before eating. These preparations reduce the residue content of the foods. The removal of residues from broccoli, snap beans, and other vegetables by washing has been reported by Smith (14). Residues were reduced as the amount of washing increased.

Washing, blanching, canning, and freezing can lower the residues originally present in raw products (1, 9, 19, 22). A recent report on tomatoes and green beans treated with malathion, DDT, and carbaryl indicated that the amount of each residue decreased considerably after commercial and home preparation procedures (4, 5).

The effects of unit operations in the preparation and processing of spinach treated with malathion had not been investigated prior to this study. The objective of this investigation was to determine the fate of malathion residues in spinach by chemical analysis.

EXPERIMENTAL MATERIALS AND METHODS

A. Preliminary Studies

Recovery Experiments

Malathion standards (98.6%) dissolved in absolute ethyl alcohol at different known concentrations were added to spinach samples before extraction. Both the treated and untreated samples were analyzed for residues, using the method described by Koevistoinen (12), which was the official method for malathion residue analysis.

Studies were also made on the stability of malathion in spinach substrates at a storage temperature of —22° F. The experiment was conducted to determine the possibility of keeping malathion-treated samples at this temperature for a certain period of time before extraction of residues.

Spinach samples weighing 500 grams each were placed in cheesecloth and dipped in 10 liters of a malathion solution for 1 minute. The concentration of the solution was 0.25% (active ingredient) of malathion wettable powder. After treatment, the samples were allowed to drain and then were air dried overnight at room temperatures. Two replicate treated and untreated samples were analyzed for initial deposits. The remaining samples were sealed in plastic bags and stored in the freezer. Duplicate samples were analyzed for malathion deposits once a week for 4 weeks.

B. Preparation of Samples

The spinach (cultivar American Long Standing) used in this investigation was grown at The Ohio State University Horticulture Farm in spring 1968. The crop was planted in eight parallel rows. Rows were 200 feet long and 2 feet apart. Cultural practices recommended for this area were followed except for pesticide application. The crops were not treated with pesticide until near to the harvest date. A water suspension of malathion insecticide prepared from 25% commercial wettable powder formulation (Stouffer Chemical Company, N. Y.) was applied to the plants at a recommended rate of 1 lb. per acre. A Hudson Sprayer (No. 9 SE. 2 U. S. gallon capacity) was used for spraying.

The outside rows were not sprayed with the insecticide. These were used as control samples. One hour after treatment or when the solution had dried, triplicate samples were collected and analyzed for initial deposit of residue at 0 day. The sprayed solution was also sampled for the determination of malathion concentration.

The spinach crop was harvested for processing on the first and third days after the treatment. These dates were earlier than the recommended date for harvesting which is 1 week after the application of pesticide. The purpose of early harvest was to insure the presence of pesticide residues.

The crops were harvested by the use of a wheeled cutter, which was pushed manually along each row. This device cuts the plants at soil level. The leaves were gathered by hand and placed in wooden baskets. About 15 baskets, each weighing approximately 18 lb., were sampled for each harvest.

Analyses of residues were also performed for samples taken at intervals of 2 days until no residue was found in the crop.

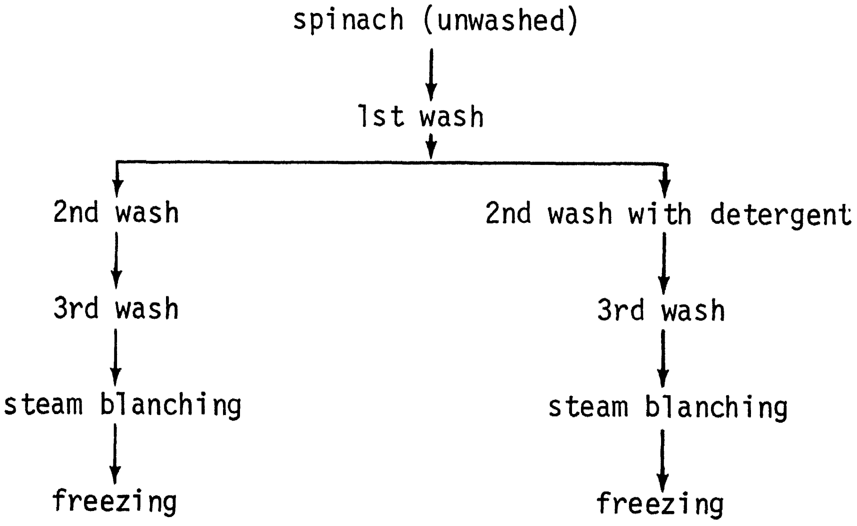
Pilot Plant Scale Processing

The harvested crop was brought to The Ohio State University Department of Horticulture Fruit and Vegetable Processing and Technology Pilot Plant immediately after harvest for processing. The following procedure was performed at the Pilot Plant (7).

Before processing, triplicate random samples weighing 500 grams each were collected in plastic bags, secured with rubber bands, and kept for analyses of residues.

1. First wash: Spinach leaves were trimmed and sorted. Yellow and defective leaves and weeds were removed. The leaves were soaked in a tank of water for 10 minutes to loosen soil adhering to the leaves. Good leaves were separated for further washing. The ratio of water and spinach was approximately 10 to 1.
2. Second wash: The leaves from the first washing were divided into two equal portions. One portion was washed for 3 minutes in warm water (100° F.). A swirling motion by hand-simulating agitation was performed to facilitate efficient washing.
3. Third wash: The washed spinach from above was then subjected to a third washing for similar amount of time and performance.
4. The clean spinach was steam blanched for 3 minutes in the blancher equipped with a slow-moving, stainless steel, screened belt. The products were cooled with a spray rinse.
5. Spinach was then packed in plastic bags for quick freezing at —22° F..
6. The other portion was washed in the same way as the plain washing described above except for the addition of 2500 p.p.m. of Klenzade fruit and vegetable washing compound at the second washing operation. It was then steam blanched as the other portion.

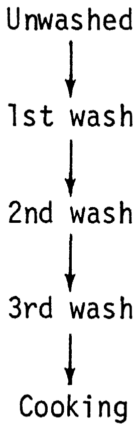
The diagram below shows the unit operations and the sampling points for residue analyses.



Triplicate samples were taken from each step for residue analyses. Samples were placed in sealed plastic bags and frozen. Residues from the unwashed, control, and steam-blanched samples were extracted in solvents on the same day of processing without freezing.

Home Preparation

Two baskets of spinach were kept in the cold room (40° F.) after harvest for home preparation on the next day. The diagram below shows the steps performed and the samples taken for residue analyses:



The preparation procedure was that of Hughes (11). It included the following operations:

1. Sorting of leaves.
2. Spinach leaves weighing approximately 500 grams were dropped into a small sink filled with cold water. The leaves were lifted in and out of the water and washed for 1 minute and sampled for residue analyses.
3. The second and third samples were washed three times and then placed in a 4-quart saucepan containing boiling water. The ratio of water to spinach was 4 to 1. The spinach was cooked for 6 to 7 minutes.

Triplicate samples were collected in each operation for residue determination. All samples except the control, unwashed, and cooked samples were frozen until extraction.

C. Residue Analyses

Residue determinations on the samples thus obtained were performed using chemical analysis (12). The method had been tested for reproducibility and amount of recovery. The procedure used was as follows:

Extraction

1. Five-hundred ml. of purified benzene were added to 500 grams of sample in a 1½ gallon wide-mouth, glass bottle. Equal amounts of $Na_2\ SO_4$ (anhydrous) and NaCl weighing 500 grams were then added. The bottle was sealed with a plastic cover lined with aluminum foil.
2. The mixture was placed in a drum tumbler stripper and agitated for 1 hour at a speed of 44 r.p.m.
3. After agitation, the mixture was strained through cheesecloth into a separatory funnel. The solid materials were separated from the liquid, with manual squeezing used to remove most of the extract.
4. The liquid was allowed to stand until two phases separated. The lower aqueous layer was drained and discarded.
5. The benzene extract was measured and transferred to a smaller glass jar for clean-up.

Clean-up or Purification

1. Ten grams of a mixture of 50% activated charcoal (Nuchar-C), 25% Hyflo super cel (Kensington Scientific Corp.), and 25% anhydrous Na_2SO_4 were added to 250 ml. of benzene extract.
2. The mixture was immediately agitated for 5 minutes in the drum tumbler stripper at 44 r.p.m.
3. It was then passed through filter paper. The purified extract was collected and kept in an air-tight amber bottle at 40° F.

Determination

1. Fifty ml. of the purified extract (containing ca. 50-700 micrograms of malathion) were placed in a 250 ml. separator funnel which contained 15 ml. of ethyl alcohol (absolute). One ml. of 6N NaOH was added. (Note: The extract was diluted to 50 ml. when the concentration of malathion was too high. The extract was concentrated under reduced pressure when the residue concentration was too low.)
2. The mixture was shaken for 1 minute.
3. Thirty ml. of Na_2SO_4 solution (90 grams in 1 liter solution) at 15° C. were added to the mixture, which was again shaken for 1 minute.
4. Twenty-five ml. of CCl_4 were then added and the mixture was again shaken for 15 seconds.
5. The mixture was neutralized with HCl, using phenolphthalein as indicator.

6. The mixture was allowed to stand. The benzene-CCl_4 layer was discarded.

7. One ml. of 5% $FeCl_3$ in 0.1 N HCl solution was added.

8. The solution was washed with 15 ml. of CCl_4 by shaking vigorously for 15 seconds. This was repeated three times.

9. The CCl_4 layer was drained out each time.

10. Exactly 10 ml. of CCl_4 and 1 ml. of $CuSO_4$ reagent were added to the mixture and the mixture was shaken.

11. The CCl_4 was drained and filtered through glass wool into a 1 cm. cuvette.

12. The absorbency was measured at 418 mm. in a Beckman DU spectrophotometer against a pure CCl_4 blank.

13. The absorbency was compared with a standard curve (Figure 1) and the concentration of malathion in the sample was determined using the following formula:

μg = micrograms of malathion obtained from standard curve

$$\mu g \times \frac{\text{vol. of solvent used for extraction}}{\text{vol. of extract used for determination}} \times \frac{1}{500 \text{ gm.}} = \text{p.p.m.}$$

Statistical Analyses

Analyses of variance were calculated according to the methods of Snedecor and Cockran (15). Student's t test was also utilized in determining differences (15).

RESULTS AND DISCUSSION

Recovery Tests

The data for recoveries from fortified spinach samples are shown in Table 1. The values of residues recovered were corrected from values of control samples. The average of two determinations was used for the calculation of percentage recovery. The analyses at different time intervals of residues remaining in treated spinach samples stored at —22° F. are presented in Table 2.

The recovery results of residues from fortified spinach samples of known malathion concentrations shown in Table 1 were satisfactory. The range of recovery was from 90% at 0.2 p.p.m. to 101% at 0.5 p.p.m. This recovery test indicated the reproducibility of the procedure.

The effect of storage temperature on the stability of malathion residues in plant substrates varied according to temperature. In general, a reduction of residues occurred as the storage temperature increased; previous studies of malathion residues in tomatoes at 55° F. showed a decrease of 30% at storage intervals of 7 days (5).

Since many samples were obtained for analyses at one time, a study of their behavior at a storage temperature of —22° F. was important with regard to values obtained in subsequent analyses. The amounts of residue recovered after different storage periods, shown in Table 2, indicated erratic results. The average concentration of malathion after 3 days of storage was higher than the initial deposits. This was probably due to a slight change in moisture content. 92% and 87% were recovered after 7 days and 14 days of storage, respectively.

TABLE 1.—Recoveries of Malathion from Fortified Spinach.

Malathion Added (p.p.m.)	Amount Recovered (p.p.m.)	Average (p.p.m.)	Percent Recovered
0.2	0.17	0.18	90
0.2	0.20		
0.5	0.50	0.51	101
0.5	0.51		
1.0	0.98	0.98	98
	0.97		
6.0	5.50	5.80	97
	6.00		
30.0	29.00	28.50	95
	28.00		

TABLE 2.—Recoveries of Malathion Residues from Fortified Spinach Stored at —22° F.

Days After Application	Concentration (p.p.m.)	Average (p.p.m.)	Percent Recovered
Initial Deposit	100	115	—
	130		
3	125	117	101
	110		
7	100	106	92
	112		
14	107	100	87
	94		
21	65	68	59
	70		
28	25	19	16
	12		

Three of the samples had concentrations higher than one of the samples at 0 day. This may be attributed to the variability of initial deposits among samples. Reductions to 59% and 16% were noted after 21 and 28 days of storage. This observation suggested that malathion in spinach might be stable for a 2-week storage period at —22° F.

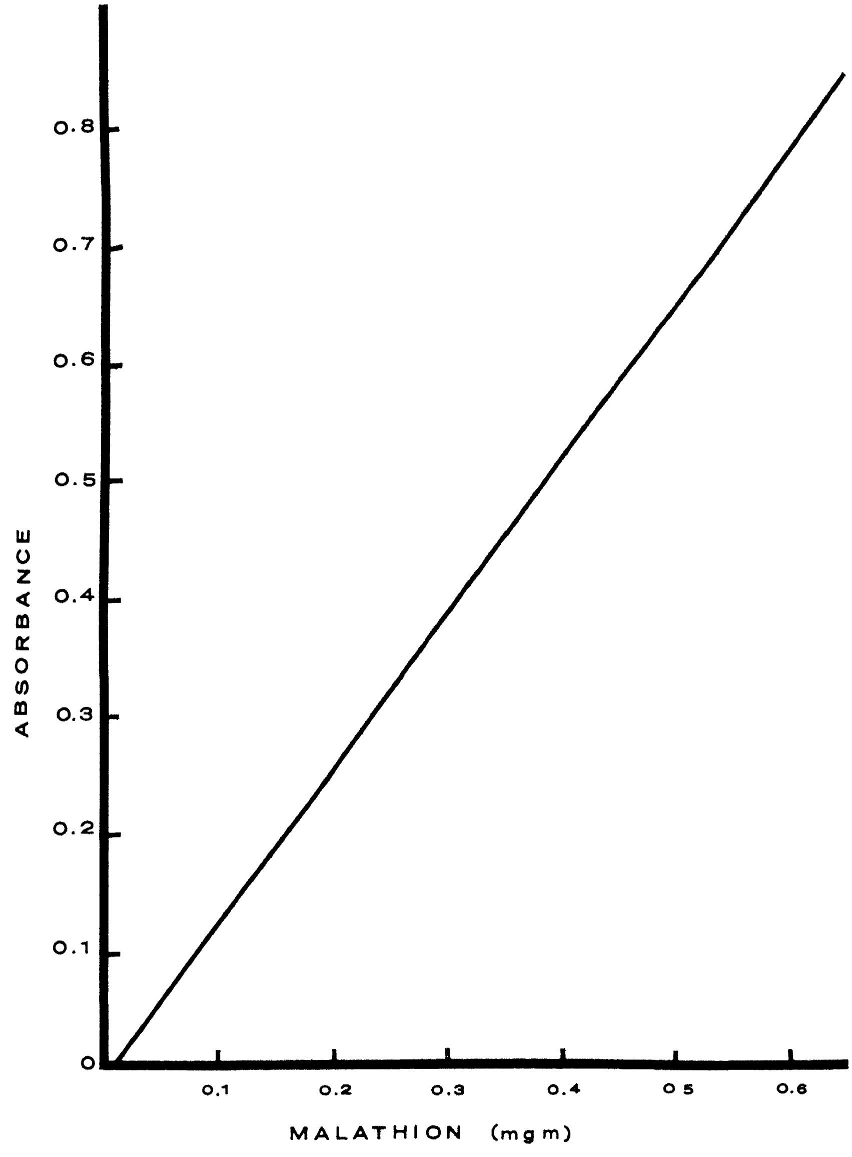


FIG. 1.—Standard curve for malathion.

Degradation of Malathion in Spinach Under Field Conditions

Data on residues of malathion in spinach at 2-day intervals after application were collected and are presented in Table 3. Mean values of residues were corrected from the mean values of control samples. The upper and lower confidence limits were arbitrarily set at the 5% level. Percent reductions of residues from the initial deposit for each determination were also calculated.

Variations among individual samples were noted. This was to be expected because the total area of leaves treated with the chemical in a given weight sample differed widely from sample to sample. The concentrations of residues at each time interval were not linear, as shown in Figure 2. There was a rapid decrease of residues 1 day after application, then the degradation process slowed. No residues were found on the 11th day after application.

There was no rainfall in the period of investigation which followed the application of malathion. Temperatures ranged from a low of 53° F. to a high of 90° F. Relative humidity ranged from 69% to 84%. It must be observed that the rate of degradation did not change as rapidly as expected after the first day of application. This may be explained by the high relative humidity, which tends to retard the loss of residues from leaf surfaces as the residues stabilize on the surface (11).

Removal of Malathion Residues by Preparation and Processing Procedures

Pilot Plant

Data concerning the removal of malathion residues in the pilot plant processing of spinach are presented in Tables 4 and 5. Percent

TABLE 3.—Recoveries of Malathion Residues on Spinach After Application.

Days After Application	Average Residues (p.p.m.)	Confidence Limits*	Percent Reduction
0	45.00	±33.22	------
1	10.25	± 7.89	77.2
3	2.26	± 1.59	94.3
5	1.66	± 2.05	96.3
7	0.57	± 0.25	98.7
9	0.21	± 0.53	99.3
11	-------	0.00	100.0

*95% Confidence limits = SD (Student's t (0.05))/ N.

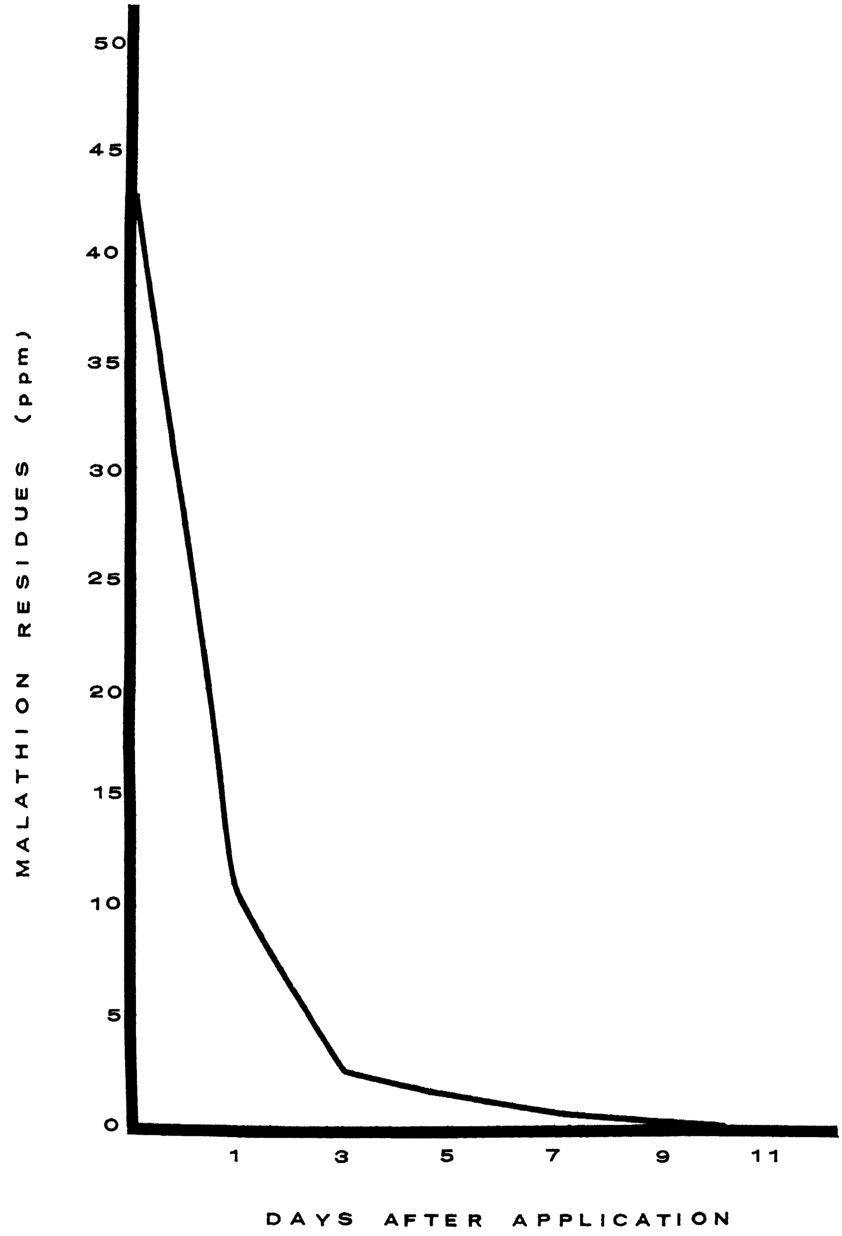


FIG. 2.—Degradation of malathion under field conditions.

reductions using unwashed spinach samples as the base were calculated and also appear in Tables 4 and 5.

Data in Table 4 indicate that unwashed spinach harvested at 1 day after application contained an average of 10.26 p.p.m. residues with

TABLE 4.—Removal of Malathion Residues in Spinach by Pilot Plant Processing at 1 Day After Application.

Treatments	Average Residues (p.p.m.)	Confidence Limits*	Percent Reduction
Unwashed	10.26	±1.89	
1st wash	2.06	±1.07	80.0
2nd wash	0.53	±0.18	94.8
2nd wash with detergent	0.25	±0.24	97.6
3rd wash	0.38	±0.09	96.3
3rd wash with detergent	0.07	±0.10	99.3
Steam blanch	0.14	±0.10	98.6
Steam blanch with detergent	0.06	±0.02	99.4
Freezing	0.15	±0.19	98.5
Freezing with detergent	0.07	±0.06	99.3

*95% Confidence limits = SD (t (0.05))/ N.

TABLE 5.—Removal of Malathion Residues in Spinach by Pilot Plant Processing Method at 3 Days After Application.

Treatments	Average Residues (p.p.m.)	Confidence Limits*	Percent Reduction
Unwashed	2.57	±1.60	
1st wash	0.17	±0.09	93.4
2nd wash	0.09	±0.11	96.5
2nd wash with detergent	0.07	±0.06	97.3
3rd wash	0.06	±0.02	97.7
3rd wash with detergent	trace†		
Steam blanch	trace†		
Steam blanch with detergent	0	0	100.0
Freezing	trace†	0	100.0
Freezing with detergent	0	0	100.0

*95% Confidence limits = SD (t (0.05))/N.
†Residue is below 0.05 p.p.m.

confidence limits of ± 7.89 p.p.m. at 5% level of significance. This amount was in excess of the tolerance level (8 p.p.m.) established by the Food and Drug Administration.

Plain washing removed from 80% to 96.3% of the residues present in raw spinach. The degree of removal of residues increased with the number of washings. Washing with detergent removed 99.3% of the residues after rinsing.

The residue content of spinach leaves which were washed in detergent was significantly lower than that of water-washed leaves. Hence, the addition of detergent in wash water improved the efficiency of washing by increasing the removal of chemical residues. With detergent washing, 15% more residues were removed after the third washing. The residues decreased by 98.6% from the original unwashed samples.

Spinach washed with detergent had only a slight decrease in residues after steam blanching. There was a slight increase in residues after freezing. This may be due to variations in individual samples. The differences in the amount of residue reduction from washing with and without detergent and subsequent steam blanching and freezing were apparent as illustrated in Figure 3.

Unwashed spinach at 3 days after application contained 2.57 p.p.m. of malathion (Table 5). This amount was far below the FDA tolerance. Similar effects were observed on the removal of residues after washing and processing procedures as previously indicated. More residues were removed after the first washing in the second crop. Only a trace of residue was detected after rinsing and after the third washing. No residue was found after steam blanching or freezing. This result indicated complete disappearance of malathion residues from the product after preparing and processing procedures. The removal of residues in the spinach harvested at 3 days after application is illustrated in Figure 4.

Plain washing in water procedures reduces malathion residue levels 96% and 97%. Detergent washing removes 99% or almost all of the residues present in raw spinach. The removal of residues in washing represented reduction of surface residues. Steam blanching caused volatilization of malathion. There was a slight decrease of residues after steam blanching. Freezing had little or no effect on the removal of residues.

Home Preparation

Residue data for the home preparation procedures are presented in Tables 6 and 7. Residues in unwashed spinach were 7.5 p.p.m. and 3.35 p.p.m. at 1 day and 3 days after application, respectively. Cold water washing removed from 95% to 98% of the residues in the crop harvested 1 day after application. Practically all of the residues present in unwashed spinach were removed after the third washing in the crop harvested at 3 days after application. No detectable residue was found after cooking.

Home preparation procedures seemed to remove a greater amount

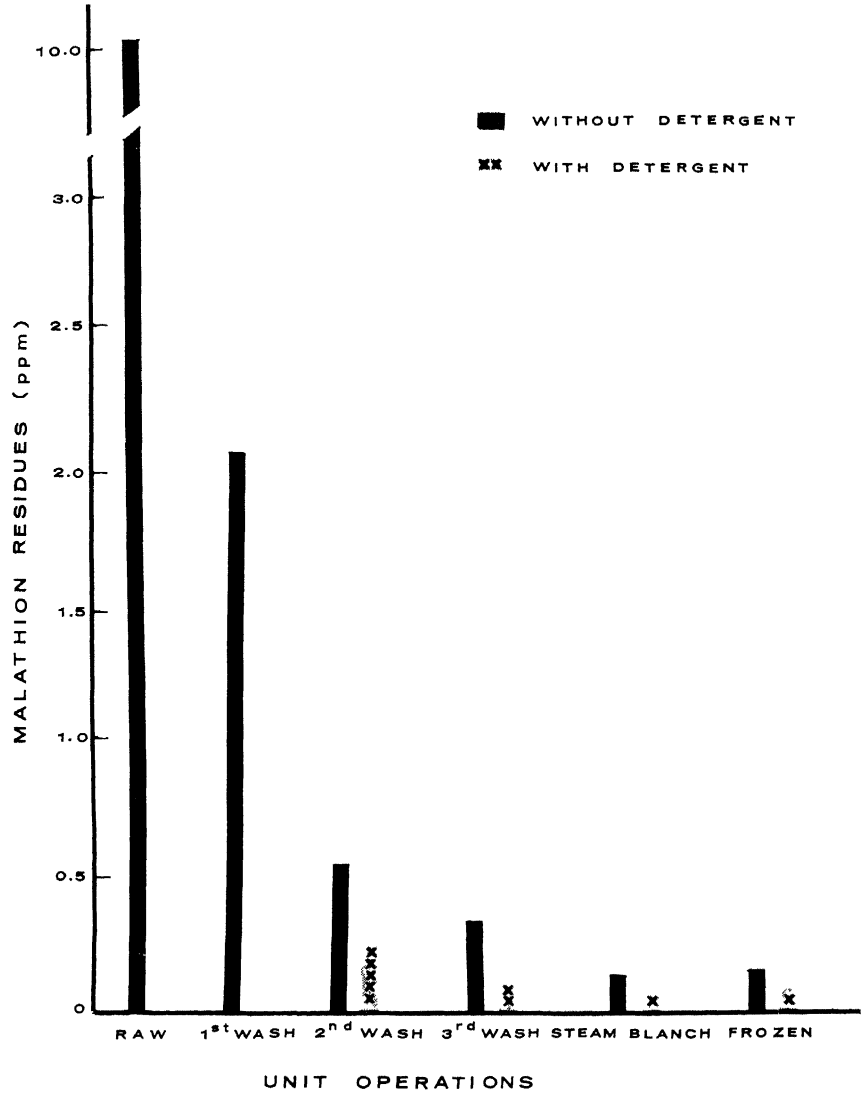


FIG. 3.—Malathion residues remaining in spinach harvested at 1 day after application by pilot plant unit operations.

of residues than was done when dealing with a small sample because it was easier to handle. Boiling or cooking washed samples in water for 6 to 7 minutes caused decomposition of the malathion residues. There was no significant difference in the percentage of residue removed from the two crops.

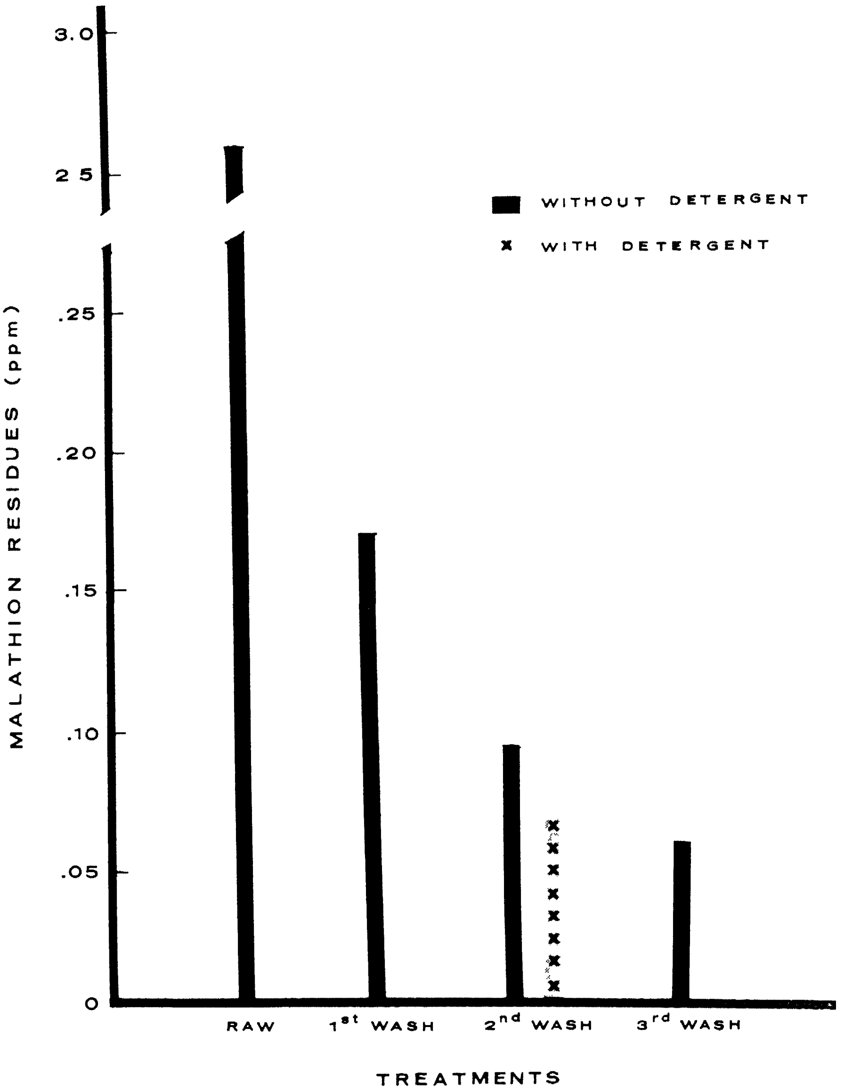


FIG. 4.—Malathion residues remaining in spinach harvested at 3 days after application by treatments.

TABLE 6.—Removal of Malathion Residues in Stored Spinach by Home Preparation Method at 1 Day After Application.

Treatment	Residue	Confidence Limits*	Percent Reduction
Unwashed	7.50	4.36	
Wash I	0.35	0.37	95.3
Wash II	0.20	0.10	97.3
Wash III	0.10	0.19	98.7
Boiling	trace		99.6

*95 % Confidence limits = SD (t (0.05))/N.

TABLE 7.—Reduction of Malathion Residues in Spinach by Home Preparation Method at 3 Days After Application.

Treatment	Residue	Confidence Limits*	Percent Reduction
Unwashed	3.35	±3.33	
Wash I	0.27	±0.29	91.9
Wash II	0.08	±0.05	97.6
Wash III	trace	trace	
Boiling	not detected		100

*95 % Confidence limits = SD (t (0.05))/N.

SUMMARY AND CONCLUSIONS

The objective of this study was to evaluate the effects of unit operations in preparing and processing spinach on the removal or reduction of malathion residues. Recovery studies were carried out to determine the reliability of the chemical method for detecting malathion residues.

Spinach used in this study was grown on The Ohio State University Horticultural Farm in spring 1968. Ordinary cultural practices were followed except for the pesticide application. The crop was sprayed with malathion wettable powder at the recommended rate (1 lb. per acre) close to harvest time in order to insure the presence of a residue. Samples were taken to determine the initial deposits.

On the next day and third day after application, the spinach crop was harvested and brought to the Department of Horticulture Fruit and Vegetable Processing and Technology Pilot Plant for processing. Pilot plant scale procedures simulating commercial processing and home preparation procedures were performed.

The unit operations involved in the pilot plant were: trimming, sorting and soaking, plain washing and detergent washing in warm water (100° F.), steam blanching, followed by cooling and quick freezing. Home preparation methods included: trimming and sorting, washing in cold water, and then cooking in boiling water for 6 to 7 minutes. Triplicate samples were taken at each unit operation for residue analyses.

Samples were collected at intervals of 2 days from the field and analyzed until no residues were found. This happened on the 11th day after the application of malathion.

The following conclusions were drawn from the data obtained:

1. Rapid decomposition of malathion deposits was observed in field conditions. A fast rate of decomposition occurred on the first day after application and the decomposition slowed considerably afterwards.

2. Washing procedures aided removal of malathion residues in raw products. Washing with addition of detergent greatly improved the removal of residues. The percent reduction by using detergent in the washing treatment was 97 to 99 percent.

3. Blanching and cooking reduced or completely removed the trace amount of residues remaining after washing.

4. Freezing caused no reduction of residues.

5. Statistical analyses indicated that, except for the first washing, the initial concentration of residues in the spinach crop does not significantly affect the percentage of residues removed.

6. The data reported in this study indicated that if by some accident spinach was obtained with a malathion residue, the residue could

be significantly reduced or removed by the unit operations involved in preparation and processing. In most cases, a single washing reduced the malathion residue below the FDA tolerance. This means that the processor has a means for reducing residues and assuring the consumer of a wholesome food supply.

LITERATURE CITED

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Ohio's major soil types and climatic conditions are represented at the Research Center's 13 locations. Thus, Center scientists can make field tests under conditions similar to those encountered by Ohio farmers.

Research is conducted by 15 departments on more than 6500 acres at Center headquarters in Wooster, nine branches, Green Springs Crops Research Unit, Pomerene Forest Laboratory, and The Ohio State University.

Center Headquarters, Wooster, Wayne County: 1953 acres
Eastern Ohio Resource Development Center, Caldwell, Noble County: 2053 acres
Green Springs Crops Research Unit, Green Springs, Sandusky County: 26 acres
Jackson Branch, Jackson, Jackson County: 344 acres
Mahoning County Farm, Canfield: 275 acres
Muck Crops Branch, Willard, Huron County: 15 acres
North Central Branch, Vickery, Erie County: 335 acres
Northwestern Branch, Hoytville, Wood County: 247 acres
Pomerene Forest Laboratory, Keene Township, Coshocton County: 227 acres
Southeastern Branch, Carpenter, Meigs County: 330 acres
Southern Branch, Ripley, Brown County: 275 acres
Western Branch, South Charleston, Clark County: 428 acres