

## Removal of Malathion Residues on Lettuce by Washing

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Malathion (S-1,2-bis(ethoxycarbonyl)ethyl O,O-dimethyl phosphorodithioate), a broad-spectrum insecticide, was used in California to eradicate infestations of the Mediterranean fruitfly (Medfly-*Ceratitis capitata*) (Segawa 1991). Under the eradication program, malathion in a protein-sugar bait was applied by helicopters over extensive agricultural and residential areas where many of the over 260 host plants susceptible to Medfly infestation could be found (Foote 1993).

Homeowners were concerned about consuming fruits and vegetables grown in home gardens that were exposed to the bait sprays. Water rinsing or washing has been reported to remove malathion residues from vegetables (Smith et al. 1955, Wallis et al. 1957). The extent to which pesticide residues could be removed from treated produce by washing may be influenced by a variety of factors such as the chemical properties and formulation type of the pesticide, the nature of the commodity, the length of time that the residue has been on the commodity's surface, and the rinsing time and rinsing agents used (Smith et al. 1955).

Hence, this study was conducted to compare the efficiency in removing malathion bait residues on lettuce a) by using two washes, water or a surfactant, and b) for two holding periods 12-hr or 36-hr after the bait was applied.

### MATERIALS AND METHODS

Malathion bait was applied to 40 heads of leaf lettuce, *Lactuca sativa* var. *Crispa* L., on a July evening to simulate the conditions under which malathion is aerially applied during an eradication program. Prior to application, each lettuce head was weighed and placed vertically in a 10.2 cm x 25.4 cm, 3-L glass jar. The 40 lettuce heads ranged in weight from 139.5 g to 231.8 g with an average value of 184.52 g. Malathion (Clean Crop Malathion ULV® 94.9% purity, Platte Chemical Co.) was combined with Nu-Lure® bait (1:4/v:v), a protein-sugar based attractant, making a mixture containing 232.4 g of malathion active ingredient (ai) per liter of bait. In order to simulate the malathion deposition from an aerial application of 23.8 mg ai/m<sup>2</sup> (Segawa 1991), each lettuce head, estimated to cover 930 cm<sup>2</sup> of land surface area, received 9 uL (2,092 ug ai) of malathion bait. The bait was applied in 1-uL droplets to the lettuce leaf surfaces using an electronic pipette.

(EP-100, Rainin Instruments Co., Woburn, MA). The malathion bait was continuously agitated on a stir plate throughout the application. After application, the 3-L jars containing the lettuce heads were placed outdoors on blue ice in open ice chests.

Washing solutions were deionized water or 1 ml of 2% Sur-Ten®, a surfactant, (American Cyanamid, New Jersey) in 1.8-L deionized water, to simulate washing with water or a detergent, respectively. Twelve hrs after application, 10 lettuce heads were randomly selected for washing with de-ionized water and 10 heads for washing with Sur-Ten® water. The 20 remaining lettuce heads were stored in a refrigerator at 10°C for 36 hrs and then rinse using the two procedures (10 heads for each rinse solution). Since in Medfly eradication program the bait is always applied in late evening, the 12-hr samples simulated residues that remain on lettuce the morning after an aerial application, and the 36-hr samples simulated the storage before consumption. Washing solution (1.8 L) was added to the 3-L container with the treated lettuce and the jar was capped and gently inverted 12 times for a 15-second period. This time period simulates the average rinsing time used by homemakers (Far-row et al. 1972). Immediately, the rinsate was transferred to an empty 2-L glass jar. The rinsate was extracted and analyzed for dislodgeable malathion and malaoxon residues. The whole washed lettuce head was chopped and blended with dry ice, then extracted and analyzed for non-dislodgeable malathion and malaoxon residues. All heads were analyzed for percent moisture prior to determination of non-dislodgeable residues.

For the residues analyses, the rinsate was extracted with methylene chloride and the lettuce slurry was extracted with ethyl acetate. Malathion and malaoxon were determined by gas chromatography (Hewlett Packard Model 5890 GC) with an HP-1 10m x 0.53mm x 2.0 um column, final column temperature at 250°C, helium carrier gas flow rate of 10 mL/min and a flame-photometric detector, at 250°C. Results were reported in ug/g (wet weight basis). The method detection limits for both malathion and malaoxon were 0.02 ug/g for lettuce and 0.002 ug/mL for rinsate, and recovery of fortified samples were about 98%. The data were analyzed using SAS General Linear Model procedures to compare log-transformed mean malathion concentrations at each level (SAS 1987). Significant effects were tested on malathion concentrations with two-way analyses of variance (ANOVA).

## RESULTS AND DISCUSSION

There were no significant differences in malathion concentrations of rinsate between the Sur-Ten® and water-rinse procedures at 12- or 36-hrs posttreatment. The percentage malathion removed was calculated by dividing ug/g dislodgeable by ug/g applied. Consequently, 12 hrs after application, 30.8% of applied malathion was removed with either rinsing method. Thirty-six hrs after application, 29.5% of applied malathion was removed with either procedure. In contrast, Smith et al. (1955) reported that 88% of the residues was washed off; albeit, they used a 30-second tap water wash at one hr after application, and they also used a

**Table 1.** Concentrations ( $\pm$ SD) of dislodgeable and non-dislodgeable malathion residues in lettuce heads rinsed with water or Sur-Ten®, a surfactant, at 12 or 36 hours after application of a malathion bait.

Holding Time	Plant Weight (g)	Dislodgeable malathion (ug/g)	Non-dislodgeable malathion (ug/g)
<b>12 Hours</b>			
Water wash	195 $\pm$ 20	3.49 $\pm$ 0.12 <sup>a</sup>	4.15 $\pm$ 0.13 <sup>a</sup>
Surfactant wash	175 $\pm$ 15	3.50 $\pm$ 0.17 <sup>a</sup>	4.22 $\pm$ 0.17 <sup>a</sup>
<b>36 Hours</b>			
Water wash	170 $\pm$ 25	3.35 $\pm$ 0.23 <sup>a</sup>	5.82 $\pm$ 1.07 <sup>b</sup>
Surfactant wash	199 $\pm$ 26	3.33 $\pm$ 0.32 <sup>a</sup>	5.97 $\pm$ 1.61 <sup>b</sup>

<sup>a,b</sup> Same letter within a column are not significantly different at  $p < 0.05$  of dislodgeable

dust, emulsifiable concentrate, and wettable powder formulations of malathion. The average non-dislodgeable malathion levels increased significantly for both rinse methods from 12 to 36 hrs holding time. The cause of this effect was unknown but not due to wilting (water loss) because before the chemical analysis the percent moisture for both 12- and 36-hr samples was approximately the same at 95%. Malaoxon were not detected in any of the rinsate or lettuce samples. We conclude that there was no difference between the use of surfactant or water for removing malathion residues from treated lettuce; water alone could be used to remove about 30% of the dislodgeable malathion residue.

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