The Effectiveness of Household Chemicals in Residue Removal of Methomyl and Carbaryl Pesticides on Chinese-Kale

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ABSTRACT

Leafy Chinese-Kale treated with methomyl and carbaryl was subjected to various chemical aqueous washes to determine the reduction of pesticides. All washing solutions, prepared from chemical stuffs available in household, consisted of 0.9% NaCl, 0.1% NaHCO₃, 0.001% KMnO₄ and 0.1% acetic acid. Filtrated water was used as control. After 15 min of washing procedure, the reduction of pesticide residues were determined by high-pressure liquid chromatography analysis and shown to have a lesser extent in methomyl compared to carbaryl. Methomyl also showed lower level of degradation in the washing solutions (2.6 to 21%) compared to carbaryl (20 to 62%). These may due to the difference of behavior and physiochemical properties between the pesticides. Variations in the degree of reduction in each pesticide were observed with the washing solution treatments. Among the washing solutions, the 0.001% KMnO₄ performed the most effective in residue reduction (48 and 93 % in methomyl and carbaryl, respectively). This may due to the oxidative property of this material. In 0.9% NaCl, 0.1% NaHCO₃ and 0.1% acetic acid washing solutions, the residue reductions were 39, 43 and 43% for methomyl; 91, 91 and 90% for carbaryl, respectively. In water, the reduction of methomyl and carbaryl were 38 and 88%, respectively. The results showed vegetable wash with solutions prepared from the chemical stuffs to be more effective in reducing pesticides than water alone.

Key words: washing solutions, pesticide reduction, vegetable wash

INTRODUCTION

The use of pesticides in agriculture has an important role on farm productivity. However, these uses leave residues on the food supply. Many of these compounds are known as toxins and have adverse effects on human health (Baron, 1991).

Methomyl (S-methyl N-[(methyl-carbamoyl) oxyl thioacetimidate) and carbaryl (1-

napthy methylcarbamate) are members of carbamate group which has pesticidal property on various crops including Chinese-Kale (Permkam and Ngampongsai, 1994). In Thailand, Chinese-Kale has been reported in commodities of high pesticide residue; with 20% of samples examined were found to exceeded the FAO's allowable maximum residue level (Rerkasem, 2004). Therefore, it is desirable to reduce these residues.

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Methomyl is non-ionic (Du Pont de Nemours, 1991) and stable under neutral and slightly acid conditions. However, aqueous solution of methomyl has been reported to decompose rapidly in alkaline media (Howard, 1991). Methomyl is systemic insecticide and when applied to plants, it can move throughout the plant by a process called "translocation". On contrary, carbaryl is contact insecticide and can breakdown through hydrolysis. Carbaryl is stable to acids, but unstable under alkaline conditions. The half-live of carbaryl is about 10 days at neutral pH (Extension Toxicology Network, 1996).

Traditional pratice of washing vegetable to remove debris and dirt prior to consumption has been shown to reduce pesticide residues. Several washing solutions such as chlorine solution, ozonated water and strong acid have shown potential in removal of pesticide residues in a step of commercial crop process (Ong et al., 1996; Archer and Stokes, 1978). However, washing the vegetables with water is widely used by the consumer upon household preparation. Krol et al. (2000) reported the effect of simple household technique of rinsing with tap water on reducing pesticide residues in produce. Not only water washing but also various preparations with chemical stuffs available in household can eliminate the residues. The chemical stuffs recommended for residue removal purposes are salt (NaCl), baking soda (NaHCO₃), distilled vinegar (acetic acid; CH₃COOH) and potassium permanganate (KMnO₄) (Rapee, n.d.). Although the general guideline on preparation of these chemical stuffs for vegetable washing has been given, their effectiveness on carbamate pesticide removal are still unknown.

The purpose of this study was to determine the efficacy of washing solutions, NaCl, NaHCO₃, KMnO₄ and acetic acid, in residue reduction of methomyl and carbaryl pesticides with filtrated water as control. Chinese-kale was used as sample because of its high deposition of

pesticide residue. Because pesticide could be degraded and dissolved during washing procedure, the fates of both pesticides on degradation and dissolution when subjected to the specific washing solution were also included in this study.

MATERIALS AND METHODS

Washing solutions preparation

All washing solution treatments were prepared from the ingredients available in common household kitchen. These consisted of 0.9% NaCl (Prung Thip; Thai Refined Salt Co., Ltd, Thailand.), 0.1% NaHCO₃ (Best Foods Baking powder; Unilever Bestfoods (Thailand) Limited, Thailand.), 0.001% KMnO₄ (Saengsawangtrakankoaw Limited, Thailand.) and 0.1% acetic acid (5% Distilled vinegar; Thai Q.P. Co., Ltd, Thailand.). The filtrated water generated from Green plus: ultraviolet water purifier (Elect Aqua, USA.) was used as control and diluents for all the solution treatments. All washing solutions were freshly prepared and measured for pH and ORP (oxidative reduction potential) prior to and after washing with a dual-scale pH meter (Model S-47, Mettler-Toledo GmbH, Schwerzenbach, Switzerland) equipped with pH and ORP electrodes.

Studies on degradation of methomyl and carbaryl in washing solutions

Methomyl (Lannate 40% SP, DuPontTM, Thailand) and carbaryl (Sevin 85% WP, Bayer Crop Science, Thailand) were diluted with filtrated water according to the product label recommendation to the concentration of 0.06% and 1.7% active ingredient. The pesticides were mixed with the washing solutions in 1:9 ratio and allowed to stand for 15 min. Then 0.5 ml of the mixture was taken and extracted immediately to determine the effect of the washing solutions on the pesticide degradation.

Studies on reduction and dissolution of methomyl and carbaryl in washing solutions

Chinese-Kale (Brassica oleracea var. alboglabra) samples were collected from local markets in Kamphaeng Phet province, Thailand. The samples were equally divided into sub samples by hand and each was soaked in either methomyl or carbaryl solution for 5 min to simulate the deposition of pesticides on them. The treated samples were then air-dried for 1 hr in the shading area. Samples from each pesticide treated pesticide vegetable were collected and analyzed for the pesticides. Each of the samples left was then divided into five groups assigned for the specific washing solution tests. The washing solution treatments were freshly prepared and used for fifteen times the volume of the pesticide treated vegetable as has been described in the method of Izumi (1999). The pesticide-treated samples were soaked in the washing solutions for 15 min, with the initial 15s gentle rotation by hand. We believed that this method mimicked actual household vegetable washing procedure. Following the washing process, the vegetable samples were picked up and air-dried for 1 hr. Vegetable samples and the after-washed solutions were then collected for pesticide residue analyses.

Sample extraction

The pesticides were extracted from treated-samples and after-washed solutions using the procedure modified from Ahmad *et al.* (1995). The extracts were analyzed by HPLC with a scanning UV-visible photodiode array detector.

Recovery study

To evaluate performance of the analytical procedures, recovery studies were performed at the 1.0 ppm fortification level of each pesticide for three extractions. The samples of pesticide-free Chinese-Kale were prepared by adding a stock solution of standard methomyl and standard carbaryl (Accu standard, USA; P-0835 and 2-Ca-

P-0838, respectively) at known amount to 20.0g of the samples before extraction. The extractions were performed as described earlier. Percent recovery was derived from the equation; % recovery = (amount of pesticide obtained / amount of pesticide added) x 100

Statistical analysis

All treatments were replicated 3 times. Values are shown as means \pm S.D. The effects of washing solutions on the % degradation, % reduction and % dissolvability of pesticides were analyzed by ANOVA as a split-plot design, with the two pesticides served as main effects and the five washing solutions served as minor effects. Significant differences between treatment effects and interactions were determined following a significant F-test (P<0.05) by using the all pairwise comparisons of treatment means with DUNCAN'S option (SAS Inst. Inc., Cary, NC). The pH and ORP of each washing solution prior to and after washing were also tested for the difference by ANOVA using the CRD model.

RESULTS

Recovery study

Mean recoveries of the standard methomyl and carbaryl added to the pesticide-free Chinese-Kale at 1 ppm level were 100.96 ± 8.50 and 93.06 ± 0.37 %, respectively. The recovery percentages in this study were higher than 80%, which indicated good and valid analytical procedure. The requirement, regarding the processes, for the quantitative analysis of pesticide residue is generally considered satisfactory if the recovery is over 80% with the coefficient of variation of 10% for repeatability (Ambrus and Their, 1986).

Pesticide degradation study

Effects of the washing solution treatments on the pesticide degradation are shown

| Item | Pesticide degradation (%) | | Effect ² | <i>P</i> -Value |
|-------------------------|------------------------------|------------------------------|---------------------|-----------------|
| | Methomyl | Carbaryl | | |
| | $(\overline{X} \pm S.D.)$ | $(\overline{X} \pm S.D.)$ | | |
| Water | 2.60 ± 0.59^{ax} | 20.13 ± 1.05^{ay} | | |
| 0.9% NaCl | 9.13 ± 2.38 bx | 20.50 ± 1.60^{ay} | T | < 0.001 |
| 0.1% NaHCO ₃ | 7.19 ± 2.38^{abx} | 20.49 ± 1.86^{ay} | W | < 0.001 |
| $0.001\%~\text{KMnO}_4$ | $20.49 \pm 2.70^{\text{cx}}$ | $62.22 \pm 8.79^{\text{by}}$ | T x W | < 0.001 |
| 0.1% Acetic acid | $21.11 \pm 3.40^{\circ}$ | 20.57 ± 1.25^{a} | | |

Table 1 Effect of washing solutions on methomyl and carbaryl degradations¹.

in Table 1. After 15 min in the washing solutions, both pesticides showed different degree of degradations. With the exception of 0.1% acetic acid solution, carbaryl was found to be more degradable than methomyl (P<0.01). The washing solution explicitly the most effective in degradation of carbaryl was 0.001% KMnO₄ washing solution (62.22 ± 8.79 %), which presented the higher (3 times) value of degradation than did other washing solutions.

On the contrary, methomyl had different levels of degradation. In water, methomyl was rather stable with only small amount of the substance degraded (2.60%). Higher degrees of degradation were observed in 0.9% NaCl and 0.1% NaHCO $_3$ washing solutions. The most effective washing solutions for methomyl degradation were 0.001% KMnO $_4$ and 0.1% acetic acid (20.49 and

21.11%, respectively). The extents of degradation indicated that methomyl was more resistant to degrade in the washing solution treatments than was carbaryl.

Pesticide reduction study

The percent reductions of methomyl and carbaryl in Chinese-Kale are presented in Table 2. To determine the percent of pesticide reductions, concentration of each wash-treatment was compared to that of the no-wash treatment. In the no wash samples, the total concentration of methomyl and carbarly were 58.72 ± 0.45 and 363.82 ± 10.84 ppm, respectively. After the washing process, the reductions of pesticides were observed, the percent reductions in carbaryl treated-sample were shown to be superior to methomyl treated sample. Washing the carbaryl

| Table 2 | Effect | of w | ashino | solutions of | n methomyl | and | carbaryl | reduction | s in | leafy | Chinese-K | ale |
|---------|--------|------|--------|--------------|------------|-----|----------|-----------|------|-------|-----------|-----|
| | | | | | | | | | | | | |

| Item | Pesticide deg | Effect ¹ | P-Value | |
|-------------------------|---------------------------|------------------------------|---------|---------|
| | Methomyl | Carbaryl | | |
| | $(\overline{X} \pm S.D.)$ | $(\overline{X} \pm S.D.)$ | | |
| Water | 37.90 ± 1.96^{ax} | 88.77 ± 0.28^{ay} | | |
| 0.9% NaCl | 39.33 ± 2.45^{ax} | $91.18 \pm 0.77^{\text{by}}$ | T | < 0.001 |
| 0.1% NaHCO ₃ | 43.19 ± 0.47^{bx} | $91.24 \pm 0.28^{\text{by}}$ | W | < 0.001 |
| $0.001\%~KMnO_4$ | 47.57 ± 1.05^{cx} | 93.50 ± 0.48^{cy} | T x W | < 0.006 |
| 0.1% Acetic acid | 43.40 ± 2.10^{bx} | 90.46 ± 0.33^{aby} | | |

¹ T = pesticide effect; W = washing solution effect; T x W = pesticide x washing solution interaction.

¹ Mixture of pesticide and washing solution was in 1:9 ratio.

² T = pesticide effect; W = washing solution effect; T x W = pesticide x washing solution interaction.

x,y Within a row, means without a common superscript letter differ (influence of the pesticide).

a,b,c Within a column, means without a common superscript letter differ (influence of the washing solution).

x,y Within a row, means without a common superscript letter differ (influence of the pesticide).

a.b.c Within a column, means without a common superscript letter differ (influence of the washing solution).

treated-samples showed reduction between 88 to 93%, while washing the methomyl treated-sample caused the decrease between 37 to 47%. This indicated the carbaryl was easier to remove by the washing procedure than was the methomyl.

Reduction in the pesticide of the samples was also influenced by the specification of washing solution treatments. Almost 48% of methomyl and 93% of carbaryl were removed from the treated samples with the 0.001% KMnO₄ wash, while washing with water alone resulted in the reductions of 37% and 88% of methomyl and carbaryl in treated-sample, respectively (Table 2). Though 0.9% NaCl, 0.1%NaHCO3 and 0.1% acetic acid were not as effective as the 0.001% KMnO₄ solution, they were more effective than the water wash. This indicated that the chemical substances used in this study could provide benefit over water on removal of pesticide residues, and 0.001% KMnO₄ washing solution was the most effective treatment for both pesticides.

Pesticide dissolving ability study

The dissolution percentages of pesticides from the treated samples in the washing solutions were examined by using the amounts of pesticides left in the solutions after washing process compared to the total amount of pesticides in the treated samples. Results showed that the carbaryl in treated sample was more dissolved than was the methomyl (47 to 74% vs 25 to 31% for carbaryl

and methomyl, respectively, Table 3).

There were also differences in pesticide dissolution percentage among the washing solutions. For carbaryl treated samples, the water and 0.1% acetic acid wash showed more pesticide dissolvability percentages than did the others. The similar pattern was also observed in methomyl treated-samples, eventhough the extents of percent dissolution were smaller when compared to the carbaryl.

pH and ORP of washing solution study

Results of the measurements on pH and ORP of washing solution are showed in Table 4. Prior to washing process, the water, 0.9% NaCl, 0.1% NaHCO₃ and 0.001% KMnO₄ washing solutions showed neutral condition (pH 6.49 to 7.18). In 0.1% acetic acid washing solution, the lower pH of 3.74 indicated acidity of the solution. After the washing process, the pH of all washing solutions was not significantly different from the pH at the beginning (P>0.05).

Before the washing process, the ORP values were high in 0.001% KMnO₄ and 0.1% acetic acid washing solution (553 and 617 mV, respectively.). This indicated the high redox potential (ORP) in these two washing solutions compared to the others. However, after the washing, the ORP value in 0.1% acetic acid washing solution decreased significantly (P< 0.05). This indicated the instability of ORP

Table 3 Effect of washing solutions on methomyl and carbaryl dissolutions in leafy Chinese-Kale.

| Item | Pesticide deg | radation (%) | Effect ¹ | <i>P</i> -Value |
|--------------------------|-------------------------------|------------------------------|---------------------|-----------------|
| | Methomyl | Carbaryl | | |
| | $(\overline{X} \pm S.D.)$ | $(\overline{X} \pm S.D.)$ | | |
| Water | 28.20 ± 2.01^{abx} | 71.71 ± 2.05^{ay} | | |
| 0.9% NaCl | 31.05 ± 1.26^{ax} | $47.93 \pm 4.23^{\text{by}}$ | T | < 0.001 |
| 0.1% NaHCO ₃ | $25.38 \pm 0.22^{\text{bcx}}$ | $46.34 \pm 0.76^{\text{by}}$ | W | < 0.001 |
| 0.001% KMnO ₄ | 24.28 ± 1.04^{cx} | $46.23 \pm 2.52^{\text{by}}$ | T x W | < 0.001 |
| 0.1% Acetic acid | 30.50 ± 2.14^{ax} | 72.70 ± 2.11^{ay} | | |

 $[\]overline{}$ T = pesticide effect; W = washing solution effect; T x W = pesticide x washing solution interaction.

x,y Within a row, means without a common superscript letter differ (influence of the pesticide).

a,b,c Within a column, means without a common superscript letter differ (influence of the washing solution).

| Table 4 | Measurements of pH and oxidative-reduced | ction potential (ORP,mV) on t | the washing solutions |
|---------|--|-------------------------------|-----------------------|
| | at before and after washing process. | | |
| _ | | | |

| Item | Before-washing | After- | After-washing | | |
|---------------------------|---------------------------|--------------------------------------|--------------------------------------|-------|--|
| | | methomyl | carbaryl | | |
| | $(\overline{X} \pm S.D.)$ | $\overline{(\overline{X} \pm S.D.)}$ | $\overline{(\overline{X} \pm S.D.)}$ | | |
| | | рН | | | |
| Water | 6.80 ± 0.48 | 7.30 ± 0.06 | 7.37 ± 0.16 | 0.194 | |
| 0.9% NaCl | 6.49 ± 0.09 | 6.46 ± 0.10 | 6.24 ± 0.09 | 0.069 | |
| 0.1% NaHCO ₃ | 7.18 ± 0.05 | 7.25 ± 0.18 | 7.32 ± 0.22 | 0.696 | |
| $0.001\%~\mathrm{KMnO_4}$ | 7.12 ± 0.36 | 6.82 ± 0.24 | 6.86 ± 0.65 | 0.776 | |
| 0.1% acetic acid | 3.74 ± 0.09 | 3.80 ± 0.18 | 3.70 ± 0.13 | 0.783 | |
| | | ORP | | | |
| Water | 284 ± 28 | 341 ± 45 | 308 ± 5 | 0.154 | |
| 0.9% NaCl | 291 ± 36 | 329 ± 53 | 288 ± 15 | 0.060 | |
| 0.1% NaHCO ₃ | 293 ± 42 | 318 ± 38 | 271 ± 26 | 0.330 | |
| $0.001\%~\mathrm{KMnO_4}$ | 553 ± 20 | 567 ± 16 | 552 ± 16 | 0.512 | |
| 0.1% Acetic acid | 617 ± 15^{x} | 510 ± 64^{y} | 488 ± 23^{y} | 0.016 | |

x,y Within a row, means without a common superscript letter differ.

value in acetic acid washing solution. In contrast, the ORP value in 0.001% KMnO₄ washing solution did not show the difference between before and after the washing process.

DISCUSSION

Effect of household washing preparations on the reduction of pesticide residues from the produce can be seen in this study. There were differences in the degree of pesticides removal. Methomyl-treated samples explicitly showed the lesser extent in residue removal than that of carbaryl. This may probably due to the difference in behavior properties between these two pesticides. Methomyl has been reported as a systemic pesticide (Du Pont de Nemours, 1991). Systemic pesticide is defined as a pesticide that is translocated to other parts of the plant than those of which originally applied (Meister, 1999). Thus, it is possible that methomyl pesticide was incorporated into the plant tissue while the vegetable samples were soaked in the pesticide solution. This mechanism made the removal of methomyl difficult. On the contrary, carbaryl is a contact pesticide and non-systemic, which resided on the surface of plant (Extension Toxicology Network, 1996). So it would be reasonable to assume that removing the carbaryl from the plant would be easier. Data on the dissolution of both pesticides in the solutions after washes, which carbaryl was found to be more dissolvable than methomyl, also supported this suggestion.

Degradation of pesticide was found considerably related to the residue reduction. Methomyl demonstrated lower level of degradation in the washing solution compared to carbaryl. This may due to the physiochemical property of this pesticide. Methomyl is non-ionic (Du Pont de Nemours, 1991). This could prevent much of methomyl from reaction with other compounds existed in the solutions. The lower degree of degradation could result in the lesser extent of reduction. Thus, the lower degree of methomyl degradation may be another reason for the difficulty in methomyl removal.

The variation in each pesticide reduction was dependent upon the specificity of washing solution. In methomyl, the 0.001% KMnO₄ revealed the greatest effect on residue reduction.

This could be partially explained by the degradation effect of this solution. Methomyl was found highly degraded in the 0.001% KMnO₄ solution. Methomyl can be broken down by oxidation. KMnO₄ is a strong oxidizing agent and the solution resulted in the high redox potential when used as the active ingredient for a given concentration. Oxidative compound such as hypochloric acid in strong acids electrolyzed water has been shown to be most effective in reducing dimethoate concentrations by oxidation (Lin *et al.*, 2006). Thus, the oxidative property of KMnO₄ could also have a significant effect on degradation and contributed to the reduction of methomyl after washing.

Methomyl has been reported to be stable under neutral and slightly acid solution (pH5) (Du Pont de Nemours, 1991). However, it was found in this study that methomyl was degraded with the 0.1% acetic acid in the similar extent to the 0.001% KMnO₄ solution. This may probably due to the high acidity and/or high redox potential of this solution. The pH and ORP of the 0.1% acetic acid had shown to be 3.74 and 617 mV, respectively. Thus, it is possible that methomyl may be degraded partially in this environment.

Like in the methomyl, the degrees of reduction in carbaryl treated sample varied and depended on the specific washing solution. Results from the washes showed that the washing solutions prepared from the chemical stuffs were more effective in reducing carbaryl than was water alone. Again, the 0.001% KMnO₄ wash revealed the highest carbaryl reduction efficiency. Examination of the effect of washing solutions on carbaryl degradation had also shown the high degrading potential of the 0.001% KMnO₄. Thus, the oxidative mechanical action by 0.001% KMnO₄ washing solution was likely responsible for the removal of carbaryl residue.

In contrast to the 0.001% KMnO₄ solution, the degradation of carbaryl with the rest of solutions did not have statistical difference from

water, but their results on the residue removal demonstrated greater value than that of water. So, the differences in carbaryl dissolution may be possibly attributed to the variation in the reduction. Unfortunately, data on dissolvability percentage of carbaryl in each washing solution did not support the suggestion. It was found that carbaryl in the treated sample was more dissolved in the water than was in the 0.9% NaCl, 0.1% NaHCO₃ and 0.001% KMnO₄ washing solutions. The lower dissolution of carbaryl in 0.001% KMnO₄ could be explained by the high degradation of carbaryl pesticide in this solution. However, this explanation could not apply to the 0.9% NaCl and 0.1% NaHCO₃ as the degradability of carbaryl in these solutions did not exceed that in the water. The cause and effect on the reduction in 0.9% NaCl and 0.1% NaHCO₃ washing solution are still not known in this study and need further investigation.

In the current study, the degradation of pesticide in the washing solution had confounded effect on the percent dissolution, as had been seen in the 0.001% KMnO₄ solution, so the percent dissolution of pesticide in the washing solution may not accurately be used in estimating the reduction of pesticide in the vegetable.

In conclusion, pesticide residues in Chinese-kale were reduced to various degrees, depending on the behavior and physiochemical properties of pesticides and also the specificity of chemical stuffs used for washing. The removal of pesticide residues from vegetable sample washes showed methomyl was more difficult to be removed than was carbaryl in the same condition. The percentages of degradations in methomyl were also less than in carbaryl. Washing vegetable with the chemical stuffs reported herein enhances the removal of pesticide residues from produce above that of washing with water alone. Among the washing solution treatments, the 0.001% KMnO₄ washing solution was found to be the most effective treatment in reducing the pesticides residues.

IMPLICATIONS

Due to the difficulty in removal of methomyl by washing procedure, the crop applied with the pesticide should have enough waiting period (defined as the time required to decrease the residue below the tolerance level) before its product could be harvested. And even though the 0.001% KMnO₄ showed the most benefit on pesticide reduction under household washing condition, one should be aware of potential risks that may be present from the material. It has also been suggested that the vegetable may want repeated rinses or washes with water if the chemical washing technique is used.

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