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Pilot application of residual insecticides as grain protectants for paddy stored in silos

(Penyemburan racun serangga cecair sebagai pelindung padi semasa penyimpanan di dalam silo)

M. Rahim*, S.H. Ong*, M. Rasali* and Y. Sapuan*

Key words: stored paddy, storage insects, insecticides, chemical residues

Abstract

Trials were conducted to evaluate three insecticide combinations to protect paddy stored in concrete silos from insect infestations. The water-diluted insecticides were sprayed to moving grain on conveyor at the rate of 1 litre/t prior to storage. Application of the grain protectants did not adversely affect grain quality based on grain temperature and moisture content. The efficacy of each treatment was tested by assaying treated grain with six species of grain beetles every 6 weeks for 9 months. The effectiveness was based on the ability of the treatment to induce 100% mortality of the test population and the production of F_1 and F_2 generations.

The combination of pirimiphos-methyl (12.0 mg/kg) + permethrin (1 mg/kg) + piperonyl butoxide (5.0 mg/kg) was the most effective in providing complete protection for at least 4.5 months from all species commonly encountered in stored paddy. Fenitrothion (20.0 mg/kg) + fenvalerate (0.5 mg/kg) + piperonyl butoxide (5.0 mg/kg) provided a minimum 3 months protection from the primary species *Sitophilus oryzae* (L.), *Sitophilus zeamais* (Motsch.) and *Rhyzopertha dominica* (F.) and one of the two prevalent secondary species, *Tribolium castaneum* (Herbst.). The effectiveness of chlorpyrifos-methyl (10.0 mg/kg) + carbaryl (8.0 mg/kg) to control primary species was limited to immediately after treatment, but the protection from the gazetted quarantine species *Trogoderma granarium* (Evert) was 7.5 months. This combination was also effective against *T. castaneum* dan *Oryzaephilus surinamensis* (L.) for 6 months.

The time taken for the residue on paddy to fall to the MRL permitted by FAO/WHO was as follows: carbaryl after 3 months; pirimiphos-methyl before 3 months; chlorpyrifos-methyl and fenitrothion before 1.5 months. Only a small fraction was detected on polished (milled) rice after 1.5−3 months. Synthetic pyrethroids (SPs) were applied at rates below the MRLs. Milling removed significant amount of residues in husks. All the residues that remained in bran were below the MRLs. Only small quantities of residues were found in bran immediately after treatment. The cost of applying grain protectants in bulk grain is estimated at RM1.82/t; 74% of this is the cost of insecticides.

*Rice and Industrial Crops Research Centre, MARDI Headquarters, Serdang, P.O. Box 12301, 50774 Kuala Lumpur, Malaysia
Authors' full names: Ab. Rahim Muda, Ong Seng Hock, Mohd. Rasali Musa and Sapuan Yasir
E-mail: mrahim@mardi.my

Introduction

Paddy is the only grain currently produced on commercial scale in Malaysia. Most of the local paddy and imported cereals (wheat, maize, soybean, barley, etc.) are stored in bulk in concrete and metal silos. These grains are often infested with insects during storage, but insect control is never practised in paddy. Insect species of primary importance (i.e. capable of damaging whole kernel) in stored grain worldwide are the *Sitophilus oryzae* (L.), *Sitophilus zeamais* (Motsch.) and *Rhyzopertha dominica* (F.). Losses in paddy and milled rice in Malaysia due to feeding by these beetles over 3–12 months storage period were estimated at 3–6.8% (Rahim 1985).

Fumigation with phosphine is occasionally conducted for milled rice but only upon discovery of severe infestations during storage. International grain trade and consumer expectation, however, requires product that is pest-free (i.e. nil tolerance to infestation). Switching phosphine gas to insecticides would prevent perpetuation of resistance to the fumigant since resistance to the gas on insects from stored paddy and milled rice were of low levels (Rahim and Sulaiman 1999).

More importantly, fumigation cannot provide protection against future reinfestations, which can be transported over to other localities when these grains are marketed out from the mill. Using insecticides as grain admixture at the earliest point of the postharvest pipeline, for example at port of entry or procurement centres for local grains, can provide longer protection for the raw grain throughout the marketing chain; irrespective of length of storage.

This paper reports trials on the feasibility of using residual insecticides to protect stored paddy from insect infestations. Besides evaluating the effectiveness of the protectants, the other objective of this investigation was to assess the technical feasibility of applying insecticides to paddy intended for long-term storage in terms of practicality of the spraying operation and uniform coverage of the grain. The treatments were assessed on the basis of efficacy against key insect pests and residues remaining on paddy and its milled fractions. These trials were part of a joint research project (1985–1991) between MARDI and Queensland Department of Primary Industries and Fisheries (QDPIF) under the auspices of Australian Centre for International Agriculture Research (ACIAR).

In Australia, laboratory studies of individual insecticides (including those evaluated in the present study) as protectants of paddy and other grain species under equivalent tropical temperature regime in the tropics (30 °C) had shown potential candidates and indicative effective rates for field application (Samson and Parker 1989; Samson et al. 1989). The chemicals showed greater loss of efficacy during the first 6 weeks, possibly due to inactivation of the residues. The relative efficacy of the protectants varies with the species of targeted insect and grain (Bengston et al. 1983; Samson and Parker 1989; Samson et al. 1989). Field application however requires the chemicals to be applied as mixture of insecticides, and therefore the application rates would be different. The assessment of the protection afforded as conducted here on paddy must be repeated, as various factors affecting the success of the control measure (the condition of the equipment, application technique, hygiene of the grain and storage facilities) are also known to influence its effectiveness (Desmarchelier 1985).

Materials and methods

Storage of grain and treatment

The experiments were carried out at Padiberas Nasional (BERNAS) paddy procurement and processing mill at Sekincan, Selangor in December 1985 to January 1986. The mill at Sekincan was equipped with six concrete silos, each with a capacity of 750 t. Emulsifiable (liquid) concentrates were used in the case of organophosphorous (OP), synthetic pyrethroid (SP) and piperonyl butoxide (synergist to SP insecticides); and water-dispersed colloids in the case of carbaryl (a carbamate group of insecticides). Details of the treatments are given in *Table 1*. The insecticide sprayer (*Eluteg*, imported from Australia) consists of four basic components: (i) a compressor to draw the diluted insecticide from the insecticide tank to the nozzle; (ii) a flowmeter that regulates the flow of the liquid (measured in cm^3/min) to the speed of movement of grain on conveyor; (iii) a primary valve to control the flow of the liquid to the nozzle as regulated by the flowmeter; (iv) a secondary valve that drains excess chemical back to the insecticide container; and (v) automatic switcher to control timing of spraying (pending the start or termination of grain movement on conveyor). The sprayer can be assembled from locally made components.

The OPs were used to control all commonly occurring storage insects known to disinfest stored grains except for *R. dominica* (Bengston et al. 1975, 1984). Control of this insect requires a mixture with either a synergised SP or carbaryl (Davies and Desmarcheliar 1981; Bengston et al. 1983). Each insecticide is diluted with water in a tank, and then combined with the companion insecticide in a vat. The diluted insecticides were sprayed into the grain stream on a belt conveyor at the rate of 1 litre/t during loading of dried paddy (11.7–12.7% m.c.) into the silo at the start of storage period. All paddy stored in silos at the mill were aerated daily from 0900 to 1800 hours at ambient air flow rate of 30 m/min/t. Aeration prevents occurrence of discoloured (yellowing) rice – the phenomena related to overheated grain in specific areas of the grain mass (‘hotspots’). Aerated grain is also an advantage in pest control as it encourages insect dispersion, thus better chance of exposure to the insecticide. In unaerated silos, the insects tend to aggregate in the localised ‘hotspots’.

Due to market demand and shortage of storage space during peak harvest, the storage time of the treated grains in the first treatment trial (Silo No. 1) was shortened to 6 months. For similar reasons, the storage period was limited to 3 months for second and third treatments (Silo No. 2 & 3). To complete the storage period (9 months), 0.9 t of the grain from each treatment was loaded in small 0.3-t bins after storage in silo was terminated.

Sampling of grain

The paddy used in the experiments was the long grain type. The sampling of the grain for bioassay and chemical residue analysis was done at two locations: (i) on conveyor

Table 1. Summary of treatments on application of insecticides for protection against insect infestations for paddy to be stored in concrete silos at the BERNAS Paddy Processing Complex at Sekincan, Selangor

	Paddy treated (tonnes)	Insecticide mixture	Insecticide application rate		
			Active ingredient (a.i) (mg/kg)	Diluent spray rate	
				lit/h	lit/t
Silo 1	732	Pirimiphos-methyl	12.0	19.0	1.0
		+ permethrin	1.0		
		+ piperonyl butoxide	5.0		
Silo 2	755	Fenitrothion	20.0	18.9	1.0
		+ Fenvalerate	0.5		
		+ Piperonyl butoxide	5.0		
Silo 3	775	Chlorpyrifos-methyl	10.0	18.9	0.9
		+ Carbaryl	8.0		

belt during loading into silo before and after treatment (the latter designated as ‘0’ month sample); and (ii) from 1.5 m below surface at 1.5 months interval during the 9 months storage. The pre-treatment samples were taken from the conveyor at 15 min intervals as checks for the purpose of comparing grain moisture and temperature during treatment, and for storage in the 0.3-t bins to be used for bioassays. Samples from the grain surface were taken using a vacuum pump at 12 sampling points in each silo, bulked, subdivided and sealed in sample tins. Samples were kept in freezer at –10 °C pending chemical residue analysis.

Measurement of storage moisture and temperature

Grain moisture was measured from pre-and post-treatment grain, and at the grain surface in the silo at each sampling date. Readings at trial site using a *Protimeter* moisture meter (manufactured by Cole Palmer, USA) were later adjusted as oven-dried based on the drying curve of the paddy used in each silo. Grain temperature was measured using a portable electronic recorder (*YSIR* by Cole Palmer). The grain temperature during treatment was measured from the sampling tin, while the grain in the silo was measured by probing the sensor 1.5 m below the surface.

Insect bioassays

Laboratory bioassays were carried out on 6 species of malathion-resistant storage beetles, namely rice weevil *(S. oryzae)*, maize weevil *(S. zeamais)*, lesser grain borer *(R. dominica)*, red-rust flour beetle (*Tribolium castaneum,* Herbst), sawtoothed grain beetle (*Oryzaephilus surinamensis,* L.) and khapra beetle (*Trogoderma granarium,* Everts). *Sitophilus zeamais* and *T. granarium* are not found in stored paddy in Malaysia. A total of 50 unsexed adult beetles (or *T. granarium* larvae) of <1 week old were assayed in 3 replicates each containing 83 g or 167 g *(T. castaneum)* of treated or untreated (control) grain. Mortality of test insects was recorded at 3 days and 26 days, and progeny production was recorded for the arbitrary F_1 and F_2 generations after 8 weeks and 16 weeks respectively. The intervals during which no progeny were produced were calculated for the F_1 and F_2 generations.

Insecticide deposition on grain and residues in milled fractions

Samples of treated paddy (250–350 g) were milled to obtain husks, bran and polished rice. Fenitrothion, pirimiphos-methyl and chlorpyrifos-methyl deposits on paddy and residues in the milled fractions were extracted with methanol (200 ml) using a modification of the method developed by Desmarchelier et al. (1977). The sample weights taken for extraction were 40 g each for paddy and polished rice and 10 g each for husks and brans. The contents were first shaken with hand for 5–10 min and allowed to stand in a refrigerator. For this study, an extraction time of 72 ± 12 h was employed instead of 40 ± 12 h. The longer time gave higher recovery of the applied insecticide (Desmarchelier et al. 1977). An aliquot of each methanolic extract was quantitatively analysed by a Varian 2100 gas-liquid chromatograph (glc) using a glass column packed with 2% OV-101 + 6% OV-210 Chrome WP, 80-100 mesh, and an alkali flame ionisation detector (AFID). The glc was operated under the following conditions: detector temperature, 220 °C; injector temperature, 200 °C; oven temperature, 130–180 °C; nitrogen flow-rate, 18 divisions on flowmeter; air-flow rate, 240 ml/min and hydrogen flow-rate, 35 ml/min.

Fenvalerate and carbaryl deposits on paddy grain and residues in the milled fractions were extracted by shaking each sample with 200 ml of methanol in a conical flask for 3–4 h (Bengston et al. 1983) using a mechanical shaker. About 40 samples of paddy and polished rice and 10 g samples of bran and husks were used.

For fenvalerate analysis, aliquots of 10, 40, 20 and 10 ml of the methanolic extracts of paddy, polished rice, bran and husks, respectively, transferred to a graduated tube placed in a hot water-bath at 45–50 °C and evaporated almost to dryness. The residue was dissolved in 1 ml of dichloromethane and 0.5 ml was transferred into a *SEP-PAK* Florisil cartridge to be eluted with a 5% acetone in n-hexane mixture to obtain 50 ml of eluate. The eluate was concentrated slowly with nitrogen gas to 5 ml. The final extract (5 μl) was injected to determine the amount of fenvalerate present. The glc operating conditions were: detector temperature, 300 °C; injector temperature, 290 °C; column temperature 275 °C; nitrogen flow-rate-rate of 18 divisions on flowmeter. A 3% OV-101 Varaport 30, 100–120 mesh packed column was used to separate the fenvalerate residues for detection with a ^{63} Ni electron-capture detector. In bran analysis, a small amount of a 7.0% deactivated aluminium oxide 90 (acidic, 70–230 mesh ASTM, activity 1) was placed at the sample end of the cartridge to remove fats from bran extracts.

Methanolic extracts containing carbaryl were passed through an anhydrous sodium sulphate column. About 20–30 ml of each dried extract was filtered through a Whatman GF/B glass microfibre filter (1.0 μm). The volume was finally filtered through an Anotop filter (0.2 μm). The filteration removed particles that might block the high-performance liquid chromatograph (hplc) column. The filtered extract (20 ml) was injected into a hplc equipped with an UV-detector operated at 222 nm. The hplc unit employed for the analysis of carbaryl comprised 2 Millipore Waters Model 501 pumps, a Millipore Waters Model 680 Automated Gradient Controller, a Millipore Waters Model 481 variable wavelength spectrophotometer detector and a Millipore Waters 745 Data Module. A Waters reversed-phase μBondapak C18 stainless steel column (7.8 mm x 30 cm) was employed for carbaryl separation. The column used a mobile phase of acetonitrile-water (60:40 v/v) under isocratic conditions at a flow-rate of 0.5 ml/min. Detection was conducted at 0.05 AUFS.

The permethrin residue on paddy was conducted by Wellcome Foundation in United Kingdom but the analysis method was not made available to the authors.

Results and discussion

Accuracy of application rates

The accuracy of application rates, based on comparison of applied rate and the calculated residue of the insecticides on paddy grain measured immediately after treatment (0 week) and at 1.5 monthly intervals for 6 months are shown in *Tables 1* and *2*. In spite of satisfactory spraying operation whereby the chemical delivery rate varies between 0.9–1.0 litre/t *(Table 1)*, the actual insecticides on the paddy grain indicated substantial variations from the targeted dosage. For example, pirimiphos-methyl was overdosed by 16.9% while fenitrothion, fenvalerate, permethrin, chloropyrifos-methyl and carbaryl were under-dosed by 31.7, 30.0, 49.0, 25.0 and 37.5% respectively *(Table 2)*. The variations were likely due to unevenness of coverage of the insecticides during spraying. Another possible reason was the presence of dust derived from abraded cuticle and epidermal tissues, which according to Bengston et al. (1984) is normally present in appreciable quantities in commercial grain. Residue loss usually occurs when insecticide protectants are applied to raw commodities in field trials (Bengston et al. 1983, 1984; Thomas et al. 1987, Arthur et al. 1992). Nevertheless in the underdosed treatments, the effectiveness would not be adversely affected. Minett and Williams (1971) and Webley (1985) showed similar efficacy irrespective of whether 1% or 100% of the grain is targeted provided the same (effective) quantity of the insecticide (active ingredient per tonne) is delivered for the whole treatment. Data on the diluent spray

Table 2. Deposits of insecticide residues on treated paddy

	Insecticide mixture					
	Fenitrothion (20 mg/kg) + Fenvalerate (0.5 mg/kg) + Piperonyl butoxide (5 mg/kg)		Pirimiphos-methyl (12 mg/kg) + Permethrin (1 mg/kg) + Piperonyl butoxide (5 mg/kg)		Chlorpyrifos-methyl (10 mg/kg) + Carbaryl (8 mg/kg)	
	Residues (mg/kg)					
Storage period (month)	Fenitrothion	Fenvalerate	Pirimiphos -methyl	Permethrin	Chlorpyrifos -methyl	Carbaryl
0	13.65	0.35	14.03	0.51	7.5	5
1.5	7.0	0.28	13.32	0.61	3.16	4
3.0	3.09	0.12	1.69	0.48	0.99	3
4.5	3.89	0.24	1.97	0.6	0.94	2
6.0	3.35	0.22	2.07	NA	0.8	2

NA = not available

rate (0.9–1.0 litre/t) indicated the targeted dosage was satisfactorily delivered.

Storage conditions

The grain temperatures and moisture contents immediately after treatment and during storage are shown in *Table 3*. The application of insecticides did not influence the grain temperature during storage. Increase in temperature can affect the rate of degradation of insecticides on grain by shortening the half-life of the chemicals (Desmarchelier 1978). In this study the result showed the grain temperature before and after treatment did not change. In Silo 1 the treatment appeared to have an effect when the mean temperature at initial storage (immediately after treatment to 1.5 months) showed an increase from 28.8 °C to 32 °C for the rest of the storage period (3 months onwards). It was however not the same in Silo 2 & 3 as the temperature at initial storage instead showed a decrease from 32–33 °C to 30.5 °C for the later period of the storage. The temperature of the treated grain during storage in the three silos was quite comparable to the mean 30 °C in untreated control. The slight variations within silo and between silos could possibly reflect the influence of the ambient air temperature which fluctuates daily at 26–34 °C.

Application of grain protectants did not affect the moisture content of the grain both immediately upon treatment, and during storage. The grain during storage in Silo 1 stabilised at just below 12% m.c., approximately the same as before treatment. The moisture of grain at pre-treatment and during initial storage (up to 3 months) in Silo 2 and 3 (mean 12.2 and 12.8% respectively) were only slightly higher than grain in Silo 1. Grain in Silo 2 and 3 and untreated control was observed to gain moisture for the duration of storage after 4.5 months, where the mean grain moisture 13.2, 13.6 and 14.3% respectively hovered around the 14% optimum limit for safe storage of grain (Christensen 1972).

As discussed later, the moisture gain was an indicative of insect activity. The difference in m.c. between silos reflected relative efficacy of insecticide treatments in controlling insect infestations. The substantially high moisture gain in untreated grain was due to metabolic activity (respiratory) of observed large natural infestations. It is known that the efficacy of

Table 3. Temperature and moisture content of grain during treatment and storage

Silo No.	Insecticide mixtures	Temperature, t (0 °C) and Moisture content, mc (%)	Mean grain temperature and moisture content during loading		Mean grain temperature and moisture content during storage (month)					
			Before treatment	After treatment	0	1.5	3	4.5	6	7.5
1	Pirimiphos-methyl + Permethrin +piperonyl butoxide	t	29.1	29.2	28.8	26	33	32	32	32
		mc	11.7	11.6	11.5	11.1	11.3	11.7	11.6	11.5
2	Fenitrothion + Fenvalerate + Piperonyl butoxide	t	34.9	34.7	32.8	31	30	30	30	32
		mc	12.7	12.8	12.2	12.3	11.6	12.2	14.1	13.3
3	Chlorpyrifos-methyl + Carbaryl	t	33.6	33.9	34.8	31	30	30	33	29
		mc	12.3	12.1	14.4	12.7	12.1	13.9	13.9	13
4	Control	t	NA	NA	30	30	30	30	33	29
		mc	NA	NA	12.3	12.2	12.6	14.3	14.4	14.3

NA = not available

OP insecticides decreases as grain moisture content increases (Samson et al. 1987, 1988). Increase in grain moisture content can cause a dilution effect and accelerate residue degradation (Arthur 1992). Grain moisture does not appear to have an influence on the insecticides, as the high degradation period occurs within 1.5 months after treatment.

Efficacy

Data on mortality and reduction of progeny are given in *Table 4.* The efficacy of the three treatment combinations varied with insect species.

Generally, the combination of pirimiphos-methyl + permethrin + piperonyl butoxide was the most effective among the three treatments tested. A minimum of 4.5 months protection was achieved against all insect species commonly encountered in stored paddy (*S. oryzae*, *R. dominica*, *T. castaneum* and *O. surinamensis*). Complete mortality of parent population and production of new generation (based on prevention of F_2 generation) was obtained for that storage period. Against *T. granarium,* the period of protection was shorter (1.5 months) but this gazetted quarantine insect is not yet known to infest paddy. *Sitophilus zeamais* is presently only found in milled rice. In future, cross-infestation could possibly lead to the species thriving on paddy since milling is done within the same facility. The *Sitophilus* species were the most difficult to control as reflected by the longer exposure (26 days) to induce total mortality for the adult (parent) beetles by all insecticide combinations tested. This resulted in some reproduction (as shown by the shorter 1.5-months suppression of F_1 generation achieved). Nevertheless, F_1 production by the surviving parent that infests the treated grain after 1.5 months could be prevented for at least 4.5 months.

A longer duration of protection was obtained for *R. dominica*, *T. castaneum* and *O. surinamensis* with treatment combination

Table 4. Periods (months) of complete mortality or progeny suppression of F_1 and F_2 generations of test insect achieved by different insecticide mixtures for the control of stored product beetles in treated paddy

Treatment	Period of effectiveness (months)				
	Species	100% Mortality		100% progeny suppression	
		3-day	26-day	F_1	F_2
Pirimiphos-methyl	SO	1.5	4.5	1.5	4.5
(12 mg/kg)	SZ	4.5	4.5	1.5	6.0
+ Permethrin	RD	9.0	9.0	9.0	6.0
(1 mg/kg)	TC	4.5	4.5	9.0	6.0
+ Piperonyl butoxide	OS	9.0	9.0	7.5	6.0
(5 mg/kg)	TG	0*	0	0	1.5
Fenitrothion	SO	1.5	7.5	3.0	4.5
(20 mg/kg)	SZ	1.5	4.5	4.5	6.0
+ Fenvalerate	RD	1.5	6.0	1.5	3.0
(0.5 mg/kg)	TC	0	1.5	0	6.0
+ Piperonyl butoxide	OS	0	0	0	0
(5 mg/kg)	TG	0	0	1.5	0
Chlorpyrifos-methyl	SO	1.5	6.0	1.5	0
(10 mg/kg)	SZ	0	4.5	1.5	0
+ Carbaryl	RD	0	0	0	0
(8 mg/kg)	TC	0	1.5	0	6.0
	OS	0	0	0	6.0
	TG	0	0	1.5	7.5

*'0' month = effectiveness limited to immediately after treatment. SO = *Sitophilus oryzae*; SZ = *Sitophilus zeamais*; RD = *Rhyzopertha dominica*; TC = *Tribolium castaneum*; OS = *Oryzaephilus surinamensis*; TG = *Trogoderma granarium*

of pirimiphos-methyl + permethrin + piperonyl butoxide. There appears to be an anomaly in the period of protection for these three species, whereby the control for F_1 generation was 9, 9 and 7.5 months respectively. The corresponding results for the F_2 progeny were 6 months for all three species. Similar anomalies occur for the other two treatments. Perhaps these were caused by the slow developing F_1 progeny that were missed during the F_1 count.

The combination of fenitrothion + fenvalerate + piperonyl butoxide provided at least 3 months protection from 3 of the 4 species found in stored paddy. On the basis of period of protection (prevention of F_2 generation) this combination was comparable to the preceding treatment against *S. oryzae* and *T. castaneum*, but the control against *R. dominica* was limited to 3 months. Nevertheless, the effectiveness was only limited to immediately after treatment for *O. surinamensis*, which is one of the two most prevalent secondary species in all stored cereals in Malaysia (the other being *T. castaneum*). That limitation was also observed in the control *T. granarium*. The action of fenitrothion + fenvalerate was also slower than treatment with pirimiphos-methyl + permethrin in controlling most species found in paddy as shown by the limited period of protection of (parent) adult mortality at short exposure (3 days) and their immediate (F_1 generation) progeny.

Chlorpyrifos-methyl + carbaryl was the least effective of the three treatments tested. Control against the more important primary pests (*S. oryzae* and *R. dominica*) was limited to post treatment, even though a longer period (6 months) was attainable for the two prevalent secondary species. This treatment combination was, however, particularly effective against *T. granarium*, where the residual activity of the protectants was effective in preventing F_2 generation for 7.5 months.

The relative efficacy of the insecticides studied here was quite comparable to the findings obtained in Australia by Samson et al. (1989). In their laboratory investigation, they compared the relative efficacy of individual insecticide on the basis of minimum effective application rates for control of *S. oryzae* and *R. dominica* on paddy. They found that fenitrothion lost biological activity more rapidly than chlorpyrifos-methyl and carbaryl, which in turn lost activity more rapidly than pirimiphos-methyl, permethrin and fenvalerate. They also observed the greatest loss of activity occurred during the first 6 weeks after treatment. They suggested the residues were probably inactivated during this early period, which is confirmed in the present study. Against *S. oryzae* their studies however showed chlorpyrifos-methyl and pirimiphos-methyl provided comparable period of protection, and both chemicals were more effective than fenitrothion. Against *R. dominica*, the order was permethrin and fenvalerate > carbaryl, which is quite similar to these findings except permethrin was more effective than fenvalerate.

Chemical residues

Deposits of fenitrothion and chlorpyrifos-methyl on paddy dropped drastically to below the maximum residue limits (MRLs) for cereal grain after 1.5 months but the time taken was longer (3 months) for pirimiphos-methyl and carbaryl (*Tables 5* and *6*). Residue loss after 1.5 months was 57, 48, 20, 20, 5 and 0% for chlorpyrifos-methyl, fenitrothion, fenvalerate, carbaryl, pirimiphos-methyl and permethrin respectively. All SPs' applied were below their respective MRL. For all insecticides the residue level on paddy tended to stabilize after 3 months.

The fast degradation of fenitrothion and chlorpyrifos-methyl during initial months of storage was similarly observed in

Table 5. Residue of insecticides (mg/kg) in milled fractions of paddy

Insecticide mixture	Fenitrothion (20 mg/kg) + Fenvalerate (0.5 mg/kg) + Piperonyl butoxide (5 mg/kg)						Pirimiphos-methyl (12 mg/kg) + Permethrin (1 mg/kg) + Piperonyl butoxide (5 mg/kg)						Chlorpyrifos-methyl (10 mg/kg) + Carbaryl (8 mg/kg)					
	Fenitrothion			Fenvalerate			Pirimiphos-methyl			Permethrin			Chlorpyrifos-methyl			Carbaryl		
Storage period (month)	Milled rice	Bran	Husk	Milled rice	Bran	Husk	Milled rice	Bran	Husk	Milled rice	Bran	Husk	Milled rice	Bran	Husk	Milled rice	Bran	Husk
0	0.1	5.83	69.2	<0.003	0.28	0.94	0.11	11.2	53.8	NA	NA	NA	0.05	4.1	26.7	ND	3.0	18.4
1.5	0.12	3.39	33.4	<0.003	0.27	0.74	0.13	10.4	46.0	NA	NA	NA	0.03	2.93	14.1	ND	2.0	14.9
3.0	0.06	1.58	12.7	<0.003	0.20	0.65	0.07	7.21	9.75	NA	NA	NA	0.03	1.86	5.9	ND	1.05	14.6
4.5	0.05	2.93	16.2	<0.003	0.24	0.8	0.03	4.83	11.3	NA	NA	NA	0.02	1.73	4.6	ND	1.15	11.2
6.0	ND	1.16	13.0	ND	0.22	0.62	0.05	6.02	11.8	NA	NA	NA	0.02	1.4	4.29	ND	0.9	10.8

NA = not available; ND = not detectable

commercial treatments on sorghum and wheat (Bengston et al. 1980, 1984; Arthur et al. 1992). In wheat and sorghum the residue levels of the two chemicals were also shown to be less persistent than fenvalerate, permethrin and pirimiphos-methyl (Bengston et al. 1980, 1984). Laboratory studies on paddy by Anuttarakul et al. (1985) showed lower degradation of pirimiphos-methyl than chlorpyrifos-methyl. The showed loss of pirimiphos-methyl residues was 17, 26 and 47% of the applied rate compared to 44.4, 89.0 and 90.2% for chlorpyrifos-methyl after 1.5, 3.0 and 5.0 months respectively.

In the present study when paddy was milled, most of the insecticides (76.3–98.4%) were removed in the husks *(Table 5)*. A small proportion was found in the bran (4.1–22.8%). A very small proportion of the insecticide deposits found their way to the polished rice. The detectable pirimiphos-methyl and chlorpyrifos-methyl residues after 6 months were 0.4% and 0.2% of the applied dose respectively. At comparable storage period Anuttarakul et al. (1985) found the residue level on milled rice after 5 months to be 4% and 1% of the application rate of pirimiphos-methyl and chlorpyrifos-methyl respectively. The higher level of residues in the milled fraction in their studies could possibly be due to less variability during spraying and lower dust level in the grain from the laboratory trials.

By 1.5–3.0 months after application of treatment, most insecticide deposits in milled fractions had dropped below the MRLs (*Tables 5* and *6*) except carbaryl. If the Malaysian MRL of 3.0 mg/kg is followed, carbaryl-treated paddy has to be stored for a period of 4.5 months before the deposition level could drop below the MRL level. The MRL of 5.0 mg/kg for carbaryl set by FAO/WHO would be more practical from a logistic point of view. The milling data indicated that residues occurring in bran are generally below the MRLs set by both Malaysia and FAO/WHO. The same conclusion cannot be drawn for permethrin because analyses were not carried out on

Table 6. Malaysian and FAO/WHO maximum residue limits (MRL) for insecticides used as a grain protectant (mg/kg)

Organisation/ Food Regulation	MRL (mg/kg)																	
	Fenitrothion			Fenvalerate			Pirimiphos-methyl			Permethrin			Chlorpyrifos-methyl			Carbaryl		
	CG	Bran	MR	CG	Bran	MR	CG	Bran	MR	CG	Bran	MR	CG	Bran	MR	CG	Bran	MR
Malaysian Food Regulations, 1995	10	NA	0.1	1.5	NA	NA	10	20 (W)	1	5	NA	NA	10 (M,S,W)	20	0.1	3	NA	NA
FAO/WHO	10	20 (W)	1.0	5.0	10	NA	10	20	1	2	10	NA	10	20	NA	5	20	NA

CG = cereal grain; MR = milled rice; W = Wheat; M = maize; S = sorghum; NA = not available milled fractions. The MRLs (Malaysian and FAO/WHO) are not available for milled rice. Nevertheless the applied concentration was below the MRL for unmilled grain.

General discussion

The chemical residues in milled rice and bran were very much below MRL set by both FAO/WHO and Malaysia. Most of the residues of insecticide protectants were inactivated on paddy during the first few weeks of storage. The chemical deposits stabilize thereafter and thereby available for protection of the grain throughout the storage period. The efficacy of the protectants was also dependent on insect species, with *T. granarium* and *Sitophilus* spps. most difficult to control than *R. dominica*, *T. castaneum* and *O. surinamensis*. The application of grain protectants has no adverse effect on the temperature and moisture content of the stored grain. The silo's temperature and interchange of moisture between the grain and their environment did not appear to influence the degradation and biological activity of the protectants.

In this study, the adoption of the grain spraying technology at the BERNAS Sekincan Mill is practical (installation and operation of the sprayer) without any need and costs in terms of additional infrastructure, modification, or manpower. Most rice mills in the country are highly mechanized. These entail the whole operation from receival of wet paddy, drying, pre-milling storage, milling, and finally packing the milled rice. Product quality assurance is best adopted at the initial point of the rice-marketing pipeline. Protecting paddy from insect infestations is important towards achieving zero tolerance to insect in rice packing at consumer outlets. From the three treatments tested, the combination of pirimiphos-methyl + permethrin + piperonyl butoxide is the best choice for complete protection against all common insects found in stored paddy for at least 4.5 months. The cost of applying insecticides as grain protectants is estimated at RM1.82 (US 0.45 per tonne). The insecticides and labour cost contributed 74% and 23% of the total cost in protecting paddy grain in silos, respectively.

There is a need, however, to look for longer complete protection through grain admixture treatments, involving other combinations of OPs with synergised SPs, or using less toxic pesticides such as insect growth regulators and biopesticides. There is also the need for longer insect protection for paddy than the current practice which is 1–3 months. A longer protection from infestations is required when the emphasis of storage reverts from milled rice to paddy. Present paddy storage practice is more of interim handling process during peak harvest and milling period. Rice is transported out of the mill for storage in warehouses in urban areas. Storage as paddy is more economic and strategic in terms of food security and quality assurance. The infrastructure, maintenance and quality assurance for storing rice is more expensive. Pest control of bag-stack commodity, particularly rice in plastic packing is difficult. It costs less to store paddy in the rural areas. The grain can be stored in bag or bulk form within a simple infrastructure, since the protective husk is less susceptible to damage caused by changes in storage climates and pest infestation as compared to storing milled rice.

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Abstrak

Tiga kombinasi campuran racun serangga telah diuji untuk melindungi padi daripada serangan serangga semasa penyimpanan di dalam silo konkrit. Padi disembur dengan racun emulsi, yang telah dilarut dengan air, pada kadar 1 liter/t semasa bergerak di atas lungsur pengangkut sebelum padi disimpan. Berasaskan suhu dan kandungan air bijirin, rawatan racun cecair tidak menjejaskan mutu padi. Keupayaan pengawalan oleh setiap rawatan diuji secara menyaring sampel padi dengan enam jenis kumbang bijirin setiap 6 minggu selama 9 bulan. Keberkesanan adalah berasaskan kematian 100% populasi serangga uji kaji serta keupayaannya membiakkan generasi F_1 dan F_2.

Kombinasi pirimiphos-methyl (12.0 mg/kg) + permethrin (1.0 mg/kg) + piperonyl butoxide (5.0 mg/kg) paling berkesan mengawal semua spesies yang biasa terdapat pada padi untuk paling minimum 4.5 bulan. Fenitrothion (20.0 mg/kg) + fenvalerate (0.5 mg/kg) + piperonyl butoxide (5.0 mg/kg) berkesan mengawal perosak primer, *Sitophilus oryzae* (L.), *Sitophilus zeamais* (Motsch.) dan *Rhyzopertha dominica* (F.) dan salah satu [*Tribolium castaneum* (Herbst.)] daripada dua spesies sekunder utama untuk 3 bulan. Keberkesanan mengawal spesies primer dengan chlorpyrifos-methyl (10.0 mg/kg) + carbaryl (8.0 mg/kg) terhad semasa rawatan sahaja tetapi amat berkesan mengawal *Trogoderma granarium* (Evert), iaitu satu spesies yang diwarta sebagai perosak terkawal (kuarantin) selama 7.5 bulan. Rawatan ini juga berkesan mengawal *T. castaneum* dan *Oryzaephilus surinamensis* (L.) selama 6 bulan.

Kandungan sisa yang terdapat pada padi menurun kurang daripada MRL yang dibenarkan oleh FAO/WHO dalam masa yang berikut: carbaryl selepas 3 bulan; pirimiphos-methyl sebelum 3 bulan; chlorpyrifos-methyl dan fenitrothion sebelum 1.5 bulan. Hanya sedikit sisa kimia tersebut dikesan pada beras putih selepas 1.5–3 bulan. Dos racun kelas sintetik piretroid yang digunakan adalah lebih rendah daripada had yang dibenarkan (MRL). Jumlah sisa yang signifikan terdapat pada hampa padi yang di asingkan semasa proses pengilangan. Sisa di dalam dedak juga di bawah MRL selepas semburan. Kos rawatan dianggarkan RM1.82/t, dengan 74% merupakan kos kimia.

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