Potential of pyrethroid-synergised pyrethrum on stored product insects and implications for use as prophylactic sprays

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Abstract The study was conducted to evaluate the synergistic effects of 2% pyrethrum extract with synthetic pyrethroids on the mortality of stored product insects. Contact toxicity was performed at variable concentrations observing mortality at 12, 24 and 48 h durations. The results of the present study indicated that, pyrethrum + deltamethrin combination (25:1 ratio) was effective on the adults of *Rhyzopertha dominica* (F.) and *Tribolium castaneum* (Herbst). On the other hand, pyrethrum + cypermethrin combination proved effective against *Sitophilus oryzae* (L.). The efficacy of the tested combination showed reasonable increase in mortality response in treated insects over increasing exposures. At 48 h, 450 ppm pyrethrum + deltamethrin combination induced 25, 90 and 97% mortalities in *S. oryzae*, *T. castaneum* and *R. dominica* adults; while, pyrethrum-cypermethrin combination recorded 75, 45 and 75% mortalities respectively. On the other hand, it was observed that, among the pyrethrum alone treatments i.e. at 300, 450 and 600 ppm concentrations, maximum mortality (62.5%) was observed in *S. oryzae* exposed to 600 ppm pyrethrum for 48 h. The effective LC₅₀ concentrations for pyrethrum (600 ppm) + deltamethrin combination was estimated to be as 0.1987 and 0.7039 $\mu\text{l}/\text{cm}^2$ for *R. dominica* and *T. castaneum* adults respectively. Contrastingly, for treatments with *S. oryzae*, a

LC₅₀ value of 0.8673 $\mu\text{l}/\text{cm}^2$ was recorded for pyrethrum (600 ppm) + cypermethrin mixture. This investigation strengthens the fact that pyrethrum along with pyrethroids is effective against storage insect pests which can be promisingly a safer insecticidal combination.

Keywords Pyrethrum · Pyrethroids · Stored product insects · Synergism · Prophylaxis

Introduction

Overall post-harvest losses of food grains are estimated to be between 10 and 15% in most of the developing countries as a result of infestation by stored product insect pests (Neethirajan et al. 2007). Insect species such as *Sitophilus oryzae* (L.) (Cotton 1920; Manivannan et al. 2016a), *Rhyzopertha dominica* (F.) (Manivannan et al. 2016b) and *Tribolium castaneum* (Herbst) (Daglish et al. 2015) are considered to be as the major pests leading to qualitative and quantitative losses to stored food grains. Fumigation is a prominent technique administered for reducing the post harvest loss caused by insect pests during storage (Houmayouni et al. 2014; Manivannan 2015). Fumigation is always followed by a prophylactic spray, since it does not offer protection from re-infestations and cross infestations. However, the prolonged use of synthetic pesticides could adversely affect the environment due to their long-term residual effects. Hence, there is a need for reducing the use of synthetic pesticides to minimize risk to users, consumers and also to the environment. Local alternatives for controlling pests involve the use of natural products generally regarded as safe (GRAS) for humans and environment.

Pyrethrum is one of the oldest and widely used insecticides found naturally in *Chrysanthemum cinerifolium*,

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also called the dalmation flower (Klaasen et al. 1996). Pyrethrum has been registered for use in pesticide industry since 1950's, because of its low mammalian toxicity compared to other synthetic insecticides. Esters of chrysanthemic acid and pyrethric acid are known as pyrethrin I and II fractions respectively, which accounts for the kill and knockdown properties with low toxicity to vertebrates (Casida 1980). However, its high photosensitivity and high rate of photo degradation and short knockdown (rapid paralysis on far death) effects, limit its commercial benefits. To enhance the stability of pyrethrum, the addition of synergists or antioxidants in retarding metabolic or photochemical oxidative reactions are the feasible options. Studies have shown that, the addition of piperonyl butoxide (PBO) as synergist to pyrethrum (Young et al. 2006) to suppress the metabolic degradation of pyrethrins thereby inhibiting the natural defense mechanism of the insect. Besides, it is also shown to enhance the penetration rates of pyrethroids through the cuticle of insects (Burt et al. 1971).

Cypermethrin and deltamethrin are α -cyano derivatives belonging to type II synthetic pyrethroid group, known to affect the action of Gamma Amino Butyric Acid (GABA) neurotransmitter (Costa 1997). Pyrethroids type I and II both slow down the nervous system of insects in the nerve cell membrane through sodium ion channels (Davies et al. 2007). Pyrethroids have shown to be the successful alternatives to organophosphorus insecticides, owing to their low toxicity to mammals (Arthur 1996); and their capability to get rapidly metabolized in humans and being renally eliminated (Heudorf and Angerer 2001). Synthetic pyrethroids were developed in order to improve the stability and activity of pyrethrum exhibiting high known down effects. However, very few pyrethroids are currently used against insect pests under storage conditions. The occurrence of insect resistance to conventional insecticides used for the prophylactic sprays especially, malathion resistance (Parkin et al. 1962; Champ and Dyte 1976) has led to the search of new viable alternatives. Therefore, the objective of this study was aimed to investigate the synergistic effects of pyrethrum and synthetic pyrethroids on major insect pests in storage environment.

Materials and methods

Whole wheat and wheat flour purchased from local market were used for rearing insect cultures at the laboratory. 2% pyrethrum (PYR) was purchased from Nitapol Industries, Kolkata. Technical grades of deltamethrin-11% w/w EC (DTM) and cypermethrin-10% EC (CYP) were obtained from Isagro (Asia) Agrochem, Mumbai and Gharde Chem, Mumbai respectively. Piperonylbutoxide (PBO)-90%

technical grade was purchased from Sigma Aldrich, USA. Liquid Teflon (anti-slip) was purchased from BioQuip Products, Inc. USA.

HPLC quantification of pesticides

Standard stock solutions were prepared by dissolving each analytical standard in acetonitrile to make up a final concentration of 1000 $\mu\text{g/ml}$. From the stock solution, serial dilutions were made at 500, 250, 125, and 62.5 $\mu\text{g/ml}$ concentrations for each of the analytical standards. Quantification was carried out using calibration curves and identification was based on the retention time of the pesticide. The chromatographic system used was Waters HPLC (Waters 207000 assembly, Austria) with a photodiode array detector (PDA) set at 220 nm for PYR and 266 nm for CYM and DTM. The conditions used were: Column: Sunfire C18-5 μm (4.6×250 mm); Injector: 20 μl loop; column temperature: ambient temperature (25 $^{\circ}\text{C}$); Mobile phase: acetonitrile/water in 80:20 v/v (degassed) with flow rate: 1 ml/min in isocratic mode. 20 μl of CYM, DTM and PYR taken from the serially diluted solutions were injected to the HPLC system for further quantification.

Rearing of test insects

Three insect species were reared at the laboratory namely, *T. castaneum*, *S. oryzae* and *R. dominica*. 500 g of whole wheat (12–14% moisture) was used as the medium of culture for rearing, *S. oryzae* and *R. dominica*, while, whole wheat flour (500 g) supplemented with 5% yeast was used for rearing *T. castaneum*. All the insect species were reared individually in a glass jar covered with muslin cloth held by rubber rings and were maintained at 30 ± 1 $^{\circ}\text{C}$ and 70 ± 5 % RH. Prior to bioassay experiments, the adults were separated out from the stock culture by sieving the culture contents through appropriate sieves to obtain 7–10 days old adults of all the insect species.

Bioassay

Bioassays were carried out with pyrethrum at three concentrations (300, 450 and 600 ppm) along with PBO in the ratio of 1:4, in combination either with deltamethrin (900 ppm) or cypermethrin (900 ppm). Liquid Teflon (anti-slip) was applied around the inner rim of the petridishes before placing Whatmann No. 1 filter paper to prevent the insects from escaping. Pyrethrum was administered at five different concentrations (1.5, 7.5, 11.5, 15.5 and 19.5 $\mu\text{l/cm}^2$) in each of the combination with four replicates each carried out at different times. Each concentration was diluted with acetone appropriately to ensure

Fig. 1 Chromatograms of pesticide standards and technical samples used for the toxicity studies (**i** Cypermethrin standard, **ii** Deltamethrin standard, **iii** Pyrethrum Pestanal standard, **iv** Cypermethrin test

sample, **v** Deltamethrin test sample, **vi** Pyrethrum extract), where **1**— isomers of cypermethrin, **2**—deltamethrin, **3a** and **3b**—Pyrethrins II and I respectively

Table 1 Effect of pyrethrum + PBO mixture with cypermethrin on the adults of three stored product insect species

Pyrethrum–PBO + cypermethrin	<i>R. dominica</i>		<i>S. oryzae</i>		<i>T. castaneum</i>	
	LC ₅₀ (μl/cm ²)	LC ₉₀ (μl/cm ²)	LC ₅₀ (μl/cm ²)	LC ₉₀ (μl/cm ²)	LC ₅₀ (μl/cm ²)	LC ₉₀ (μl/cm ²)
300 ppm						
12 h	**	**	**	**	**	**
24 h	38.68	63.85	25.90	35.02	##	##
48 h	31.75	62.14	18.78	26.36	32.83	46.53
450 ppm						
12 h	28.32	39.28	**	**	**	**
24 h	21.72	29.88	24.80	34.45	##	##
48 h	16.43	23.63	15.44	21.81	21.52	29.85
600 ppm						
12 h	20.58	23.93	**	**	30.80	41.84
24 h	13.32	26.56	21.37	32.24	20.21	27.25
48 h	9.679	30.21	13.64	19.74	17.58	23.51

**—No mortality observed; ##—LC₅₀ values could not be estimated. The LC₅₀ and LC₉₀ values correspond to the pyrethrum concentrations synergized with fixed volume of cypermethrin

that the spread of the test concentration was uniform onto a 90 mm diameter Whatmann No. 1 filter paper. After evaporation of the solvent, 20 adults of the *S. oryzae* were released onto the filter paper. The treatment involving only PBO, deltamethrin, cypermethrin, acetone and untreated filter circles served as separate controls. The synergistic effect of the above-tested combinations was compared with the 2% pyrethrum extract at 300, 450 and 600 ppm concentrations. The data on mortality of the adults was recorded at 12, 24 and 48 h of durations. The same procedure was repeated for the bioassays on *R. dominica* and *T. castaneum*.

Statistical analysis

The % mortality was calculated from three replicates and was corrected using Abbott's formula (Abbott 1925) and was subjected to probit analysis using Stat plus, 2009 professional software, Analyst Soft Inc, 2009 for the determination of LC₅₀ and LC₉₀ values. Graphical representation of the data and the significance between the groups were generated using Graph pad Prism 5 (GraphPad Software, Inc., La Jolla, CA, USA).

Results

HPLC results

The retention times for the standards—Pyrethrin-II, Pyrethrin-I and DTM were 7.23, 14.05, and 17.93 min, respectively; and for the CYM isomers (3 peaks combined)

were 16.11 for trans- and 16.92 and 17.39 min for cis-isomers. The calibration curves were obtained by plotting the peak area against pesticide concentration. The obtained correlation coefficients (r^2) for CYM, DTM and pyrethrins (Pyrethrin-I and Pyrethrin-II combined) were 0.999, 0.988 and 0.999 respectively. Typical chromatograms obtained for the pesticides used in the experiment and standards using HPLC equipped with PDA detector are presented in Fig. 1. Pyrethrin-II elutes earlier than Pyrethrin-I because Pyrethrin-II is more hydrophilic than Pyrethrin-I and showed that no interferences were encountered between the two peaks as indicated from their different retention times (Antonius et al. 1997). From the regression equation of each analyte, the estimated concentration of CYM, DTM and pyrethrins were found to be 107.52 µg/mg (10.75%), 113.83 µg/mg (11.38%) and 23.47 µg/mg (2.35%) of sample respectively. The HPLC analysis revealed that the, technical quality of the pesticides was on par with obtained commercial pesticide standards.

Toxicity results

The mortality of the tested insects showed dose-time dependent response. Pyrethrum along with pyrethroids (deltamethrin and cypermethrin) and PBO resulted in higher mortality than pyrethrum alone and the mortality was directly correlated with the concentration (Tables 1, 2). Linear reduction in the LC₅₀ values was observed with increasing concentration and exposure in all the tested insect species. Almost all the combined doses offered synergistic effects on treated insects irrespective of the combination administered. No adult mortality was observed in any of the

Table 2 Effect of pyrethrum–PBO mixture with deltamethrin on the adults of three stored product insect species

Treatment with pyrethrum + deltamethrin + PBO	<i>R. dominica</i>		<i>S. oryzae</i>		<i>T. castaneum</i>	
	LC ₅₀ (µl/ cm ²)	LC ₉₀ (µl/ cm ²)	LC ₅₀ (µl/ cm ²)	LC ₉₀ (µl/ cm ²)	LC ₅₀ (µl/ cm ²)	LC ₉₀ (µl/ cm ²)
300 ppm						
12 h	51.81	80.00	**	**	32.37	46.04
24 h	23.26	36.16	34.44	50.96	24.80	34.45
48 h	21.13	35.06	26.74	47.34	23.01	35.95
450 ppm						
12 h	22.68	33.80	**	**	18.75	26.10
24 h	12.89	23.60	28.78	39.86	17.25	23.94
48 h	9.86	24.68	24.35	34.09	13.64	19.74
600 ppm						
12 h	27.36	45.95	**	**	20.57	33.84
24 h	12.52	26.84	25.61	35.56	14.84	20.78
48 h	3.12	17.57	16.21	26.23	11.07	15.48

**—No mortality observed, The LC₅₀ and LC₉₀ values correspond to the pyrethrum concentrations synergized with fixed volume of deltamethrin

tested insect species when pyrethrum alone was tested at 300 and 450 ppm concentrations. The data on the mortality response of the adults to pyrethrum + pyrethroid (deltamethrin/cypermethrin) combination showed species-specific variation. The general order of susceptibility of tested insects to various synergistic combinations was found to be as pyrethrum + deltamethrin > pyrethrum + cypermethrin > pyrethrum alone.

Among the tested insect species, the adults of *R. dominica* responded well to all the pyrethrum concentrations (300, 450, and 600 ppm) when combined with synthetic pyrethroids, deltamethrin or cypermethrin (Fig. 2). It was observed that with increase in pyrethrum concentration and exposure time, the LC₅₀ values decreased by approximately 55–90% (i.e.) when combined with deltamethrin and 40–53% with

cypermethrin combinations respectively. The observed adult mortality in *T. castaneum* was comparatively low (Fig. 3) for PYR + cypermethrin combination than other insect species tested (Table 1). The estimated LC₅₀ values for *T. castaneum* exposed to pyrethrum + cypermethrin combination for 48 h were found to be as 3.283, 2.152, and 1.758 $\mu\text{l}/\text{cm}^2$ for 300, 450 and 600 ppm respectively. No mortality was recorded until 24 h treatment period, when 300 and 450 ppm concentrations of pyrethrum + cypermethrin combinations were tested. For pyrethrum + deltamethrin combination, at 300 ppm concentration recorded an LC₅₀ value of 3.237 $\mu\text{l}/\text{cm}^2$ over 12 h exposure which came down to 2.301 $\mu\text{l}/\text{cm}^2$ over 48 h extended period (Table 2). Interestingly, in contrast to *T. castaneum* (Fig. 3) and *R. dominica*, the adults of *S. oryzae* were more susceptible to pyrethrum + cypermethrin

Fig. 2 Mortality responses of *R. dominica* to different PYR concentrations with synthetic pyrethroids. (a, b, c PYR:CYM + PBO combinations at 300, 450 and 600 ppm concentrations respectively, while d, e, f PYR:DTM + PBO combinations at 300, 450 and

600 ppm concentrations respectively). The means followed by the same letter in the graph columns are not significantly different at ($P < 0.05$) according to Tukey's test at degrees of freedom = 15

Fig. 3 Mortality responses of *S. oryzae* to different PYR concentrations with synthetic pyrethroids. (a, b, c PYR:CYM + PBO combinations at 300, 450 and 600 ppm concentrations respectively, while d, e, f denotes PYR:DTM + PBO combinations at 300, 450 and

than with pyrethrum + deltamethrin combination (Fig. 4). In the case of *S. oryzae*, 12 h treatment did not induce any mortality and no significant variation in their response was apparent in both the tested combinations over further extended periods. At the maximum tested concentration (600 ppm), pyrethrum + cypermethrin treatment recorded a LC_{50} value of $1.364 \mu\text{l}/\text{cm}^2$ while for deltamethrin combination the estimated LC_{50} value was $1.621 \mu\text{l}/\text{cm}^2$ over 48 h treatment period.

Discussion

In the present study, we investigated the insecticidal activities of pyrethrum alone and pyrethrum: synthetic pyrethroid combinations on three major stored product

600 ppm concentrations respectively). The means followed by the same letter in the graph columns are not significantly different at ($P < 0.05$) according to Tukey's test at degrees of freedom = 15

insects. Our studies with pyrethrum alone treatments at 300, 450 and 600 ppm test concentrations exhibited significantly lower mortality in the test insects compared to the synergistic treatments due to their high photosensitivity and less stability. To support this view, Zito et al. 1983 reported that, pyrethrum is highly photosensitive, less stable and can easily degrade. Further, we observed that the adults exposed to higher test concentrations developed narcosis/paralysis within 10 h of exposure, as both pyrethrins and pyrethroids are known to enter the insect system through the cuticle and target the central and peripheral nervous systems of insects. Similar to the present observation, Davies et al. (2007) also reported that, pyrethrins excite the nerve cells and induce the production of repetitive discharges resulting in paralysis. In general, pyrethrum is known to influence the transmission of nerve

are in agreement with Arthur (1996), who indicated *S. oryzae* as one of the most resistant stored product insect species to pyrethroids. In addition, Athanassiou et al. 2004 reported that the synergised cypermethrin was more effective against *S. oryzae*. Similarly, the present study reports the potential toxicity of pyrethrum-cypermethrin combination on *S. oryzae*. Furthermore, Samson and Parker (1989) and Arthur (1992, 1994, 1999) reported that *S. oryzae* could not be controlled by normal application rates of deltamethrin, bioresmethrin and cyfluthrin applicable for other insect species, like *R. dominica* (F.) and *T. castaneum* (Herbst). These studies further suggested the tolerance/resistance trait in *S. oryzae* to deltamethrin in general. Contrastingly, Bengston et al. (1987) reported that, *S. oryzae* was less susceptible compared to *R. dominica* treated with synergized cypermethrin as grain protectant. More interestingly, the present report indicate the susceptibility of *R. dominica* population to all the three tested concentrations (300, 450 and 600 ppm) of pyrethrum extract in combination with deltamethrin or cypermethrin. To support this view, the lowest LC₅₀ value (3.12 µl/cm²) in the entire experiment was recorded when pyrethrum was used along with deltamethrin. Hence it is imperative that, *R. dominica* can be managed effectively at lower concentrations than *S. oryzae*, which is in agreement with the earlier reports by Arthur (2002); Halliday et al. (1992). As pointed out by Athanassiou et al. (2004), residual sprays may probably continue to play a vital role in stored product food protection. But it is equally important to comply with the local standards where the pesticide residues in stored food products have stringent regulations. Hence, considering them for prophylactic sprays will preclude the chances of cross/re-infestation in storage environment especially reducing the extensive load of synthetic pesticides which are currently in use.

Conclusion

In the current context of decreasing number of insecticides and increasing number of control failures due development of insect resistance to conventional insecticides, it is inevitable to consider newer insecticides for effective Integrated Pest Management. The present study is a similar approach towards Integrated Pest Management, which invariably suggests the potential use of pyrethrum-deltamethrin combination as a prophylactic spray at food storage depots to effectively manage stored product insects. However, further studies are required to evaluate the persistence of this combination at different storage environments and determine their long-term efficacy.

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