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Full Length Research Paper

Insecticide activity of piperine: Toxicity to eggs of *Spodoptera frugiperda* (Lepidoptera: Noctuidae) and *Diatraea saccharalis* (Lepidoptera: Pyralidae) and phytotoxicity on several vegetables

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Products made from piperine are important for the management of pests, but allelopathic studies for these products are also relevant. The aims of the study were: First, to evaluate the effect of piperine on *Spodoptera frugipera* and *Diatraea saccharalis* and as second objective, to evaluate the allelopathic and phytotoxicity effects of this compound in the germination and growth of several vegetables. The piperine was applied on eggs laid by both lepidopteran species and on sand used as a substrate for the seeds plants. The hatching in recently laid eggs of *S. frugiperda* and *D. saccharalis* with piperine was lower than older eggs. *Allium cepa* presented a higher percentage of germination at 0.5 and at 5.0 mg/Kg and *Lactuca sativa* the lowest. The highest length of seedling was for the *A. cepa*. The piperine shows biological impact on eggs of *S. frugiperda* and *D. saccharalis* as well as in the germination and growth of plants tested.

Key words: Allelopathic, extracts, hatching, lepidoptera, phytotoxicity, piperine, vegetables.

INTRODUCTION

Insecticides from medicinal plants are an attractive alternative for pest management because they pose low threat to the environment or to human health compared to synthetic insecticides (Moreira et al., 2007). Studies on bioactivity of plant derivatives for pest control continue to increase, but few of them have potential as botanical insecticides. Chemical instability, regulatory barriers and the availability of new cost-effective and relatively safe synthetic products compared to traditional insecticides are hindering the success of botanicals products. These insecticides are best suited for organic food production but they have potential to protect food in developing countries (Isman, 2006).

The harmful impact of allelopathy can be exploited for pest and weed control (Xuan et al., 2005). The potential allelopathic effects of secondary metabolites of plants from Piperaceae family, which include phenolic compound and amide derivatives in Capsicum leachings on germination and plant growth, have been shown by different studies (Kato-Noguchi and Tanaka, 2003). However, the allelopathy of black piper, an important crop in north region of Brazil, is poorly studied (Siddiqui, 2007).

Extracts of black pepper *Piper nigrum* L. (Piperaceae) and other species of this family are toxic to Lepidoptera

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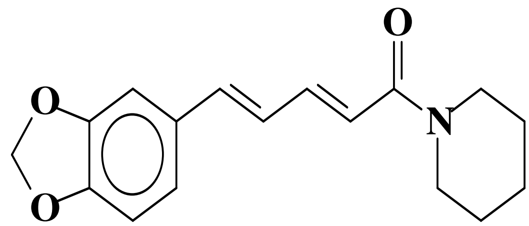

Figure 1. Structural formula of piperine.

insects, including *Ascia monuste orseis* Latreile, 1819 (Lepidoptera: Pieridae) (Paula et al., 2000) and *Spodoptera frugiperda* Smith, 1797 (Lepidoptera: Noctuidae) (Batista-Pereira et al., 2006). They were also repellents and caused morphogenic effects in the fifth instar of *Dysdercus cingulatus* F., 1775 (Heteroptera: Pyrrhocoridae) and in the last instar of *Achoea janata* L., 1758 (Lepidoptera: Noctuidae) (Osmani et al., 1987).

Species of the family Piperaceae are rich in amides as the major secondary metabolites and responsible for the insecticidal properties of these plants (Parmar et al., 1997). Piperine (Figure 1) ([(*E*,*E*)-1[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine]) is the main compound of black pepper extracts with insecticidal properties (Su and Horvat, 1981). Piperine and structural analogues are toxic to *S. frugiperda* larvae (Batista-Pereira et al., 2006), but the effect of piperine on viability of Lepidoptera eggs and its phytotoxicity activities have not been studied yet.

The aims of this work were to evaluate the effects of piperine on the hatching of *S. frugiperda* and *Diatraea saccharalis* F., 1974 (Lepidoptera: Pyralidae) and as a second part the potential phytotoxicity of this amide in the germination and growth of seedlings of broomstick *Bidens pilosa* L., lettuce *Lactuca sativa* L. (Asteraceae), onion *Allium cepa* L. (Alliaceae) and cucumber *Cucumis sativus* L. (Cucurbitaceae).

MATERIALS AND METHODS

Piperine

Piperine was isolated from dried fruits of black pepper. Milled fruits (30 g) were extracted with ethanol (100 mL, analytical grade) under reflux by 30 min. The solution obtained was concentrated in an evaporative bath under vacuum and a cold solution of NaOH (10%, w/w) was added, which precipitated a yellow solid. Thus, after exhaustive washing with water, it was recrystallized with acetone, and it yielded yellowish crystals characterized as piperine by comparison of spectroscopic (FTIR, MS and NMR) and physical data (mp = 128°C) that were obtained (Ahn et al., 1992).

Insects and extracts

The trial was carried out at the Laboratory of Insects Rearing (LACRI) in a climatized room (25 ± 1°C, 70 ± 10% of RH and 12 h of photoperiod) at Embrapa Maize and Sorghum in Sete Lagoas, Minas Gerais State, Brazil. *S. frugiperda* and *D. saccharalis* were reared on artificial diets (Sen et al., 1993; Tavares et al., 2009). Piperine was diluted in ethanol at 10 g/L (1%) or 20 g/L (2%) and its liquid extract (solution) was applied with micropipette on eggs (recently laid, one- and two-days-old) of *S. frugiperda* and *D. saccharalis*. The control group was treated with ethanol alone.

Larvae hatching

Groups of pieces of paper used for oviposition and with recently laid or one- or two-days-old eggs of *S. frugiperda* or *D. saccharalis* were cut and 20 eggs per group were left on them. Each group was put into tubes individually (2 cm in diameter × 10 cm in height). The piperine was diluted in ethanol at 10g/L (1%) or 20 g/L (2%) and 0,04 ml of the solution of this product was applied to each group of eggs and were left for 30 min at room temperature in order to dry. Hence they were sealed with PVC film.

The evaluation of the hatching of the group of eggs of *S. frugiperda* and *D. saccharalis* occurred after 5 and 8 days from the application of piperine extract. This period is enough for the larvae hatching to occur under normal laboratory conditions (Sen et al., 1993; Tavares et al., 2009).

The design was completely casualized with three replications, each of them with a group of 20 eggs. Data were corrected (Abbott, 1925) and the data set of the hatching were subjected to variance analysis (ANOVA) and the averages compared with the Tukey test ($P < 0.05$) with the software MSTAT-C, version 2.1 (Russel, 1989).

Phytotoxicity bioassays

The phytogrowth inhibitory activity of piperine was evaluated on seeds of *A. cepa*, *B. pilosa*, *C. sativus* and *L. sativa* using a Petri dish bioassay with a modified methodology (Macías et al., 2000). Piperine was dissolved in acetone (50 ml, analytical grade) and impregnated in sand (30 to 60 mesh) with micropipette to the final concentration of 0.5 and 5.0 mg/kg after evaporation. Pure acetone was added on sand in the control.

Three Petri dishes with 20 seeds were used per treatment. Each one was performed in BOD (Biochemical Oxygen Demand) incubator with a photoperiod of 12 h during 10 days. The number of seeds germinated per dish and the total length of seedlings were evaluated after this time. The results were analyzed by the Kruskal-Wallis test ($P < 0.05$). Ethanol or acetone was used by the higher solubility in the substrate.

RESULTS

The piperine at 1% caused a higher mortality of recently laid eggs (88.80%) than of those with one- (36.30%) or two-days-old (15.00%) of *S. frugiperda* (Table 1). The hatching of larvae of this insect was similar with piperine at 2% (12.50, recently laid; 23.70, one-day-old and 20.00%, two-days-old) (Table 1).

The hatching of larvae of one- or two-days-old of *S. frugiperda* was higher with piperine at 2% (76.30 and 80.00%, respectively) than with this product at 1% (36.30 and 15.00%, respectively) (Table 1). The piperine at 1% caused higher mortality of recently laid (98.90%) or one-day-old eggs (96.90%) than of those of two-days-old *D. saccharalis* eggs (93.00%), but the mortality of eggs of

Table 1. Hatching (mean ± standard error) of *S. frugiperda* (Lepidoptera: Noctuidae) according to development age of egg after five days of application of the ethanolic extract of piperine in laboratory[1].

Age of egg	Hatching (%) of *S. frugiperda*[2]		
	1% Piperine extract	2% Piperine extract	Control
Recently laid	11.20 ± 3.34[Aa]	12.50 ± 3.53[Aa]	66.20 ± 8.13[Ab]
One-day-old	63.70 ± 7.98[Bb]	23.70 ± 4.86[Aa]	70.00 ± 8.36[Ab]
two-days-old	75.00 ± 8.66[Bb]	20.00 ± 4.47[Aa]	80.00 ± 8.94[Ab]
VC		25.62%	

[1]Three replications (20 eggs per group). [2]Abbott (1925). Means followed by the same capital letter per column or small letter per line did not differ by the Tukey test ($P < 0.05$). VC: Variation coefficient.

Table 2. Hatching (mean ± standard error) of *D. saccharalis* (Lepidoptera: Pyralidae) according to development age of egg after eight days of application of the ethanolic extract of piperine in laboratory[1].

Age of egg	Hatching (%) of *D. saccharalis*[2]		
	1% Piperine extract	2% Piperine extract	Control
Recently laid	1.10 ± 0.04[Aa]	0.00 ± 0.00[Aa]	6.80 ± 1.60[Ab]
One-day-old	3.10 ± 0.76[Ab]	0.00 ± 0.00[Aa]	12.40 ± 3.52[Bc]
Two-days-old	7.00 ± 1.64[Bb]	0.00 ± 0.00[Aa]	17.70 ± 4.20[Cc]
VC		8.81%	

[1]Three replications (20 eggs per group). [2]Abbott (1925). Means followed by the same capital letter per column or small letter per line did not differ by the Tukey test ($P < 0.05$). VC: Variation coefficient.

Table 3. Germination (%) of *B. pilosa*, *L. sativa* (Asteraceae), *C. sativus* (Cucurbitaceae) and *A. cepa* (Alliaceae) treated with piperine in two concentrations (0.5 and 5.0 mg/kg) in laboratory.

Concentrations	*B. pilosa*	*L. sativa*	*C. sativus*	*A. cepa*
0.5 mg/kg	-4.80	-12.82	+8.33	+13.04
5.0 mg/kg	-12.82	-12.82	-12.82	+30.43

Values expressed as percentage of control germinated seeds. Values (+) indicate stimulus and (-) indicate inhibition of the germination compared to the control.

this insect was similar with piperine at 2% (0.00%, respectively) (Table 2).

The mortality of eggs of one- or two-days-old of *D. saccharalis* was higher with piperine at 2% (100.00%) than with this product at 1% (96.90 and 93.00%, respectively) (Table 2). The seeds germination varied among the species and the piperine concentration (Table 3). The inhibition in the germination was of 12.82% to 5.0 mg/kg in the dicotyledons (*B. pilosa*, *C. sativus* and *L. sativa*), whereas at 0.5 mg/kg it inhibited only 4.8%, of the germination of *B. pilosa* and 12.82% of the *L. sativa* and it increased by 8.33% the germination of *C. sativus* (Table 3). The monocotyledon *A. cepa* had, in both concentrations, higher germination compared to the control (13.04% at 0.5 mg/kg and 30.43% at 5.0 mg/kg) (Table 3). The length of the dicotyledoneous seedling (*B. pilosa*, *C. sativus* and *L. sativa*) decreased with the increase of piperine concentration, and this was not detected in the treatments with *A. cepa* (Table 4). The lengths of *B. pilosa*, *C. sativus* and *L. sativa* in the two concentrations of piperine were shorter, which shows that piperine in low concentrations affects the growth of these plants (Table 4).

DISCUSSION

The extraction procedure piperine yields at 5.7% (w/w) with high purity (> 99% by GC-MS) with similar results to those of 3 to 7% (w/w) for this compound (Ribeiro et al.,

Table 4. Length (mm) of the seedling of *B. pilosa*, *L. sativa* (Asteraceae), *C. sativus* (Cucurbitaceae) and *A. cepa* (Alliaceae) treated with piperine in two concentrations (0.5 and 5.0 mg/kg) in laboratory.

Parameter	*B. pilosa*	*L. sativa*	*C. sativus*	*A. cepa*
Control	54.54 ± 8.35^{a}	49.80 ± 7.40^{a}	67.67 ± 23.93^{a}	47.54 ± 12.35^{a}
0.5 mg/kg	49.18 ± 8.03^{b}	36.65 ± 6.83^{b}	48.92 ± 10.69^{b}	50.89 ± 9.41^{a}
5.0 mg/kg	45.91 ± 7.46^{b}	33.85 ± 7.32^{b}	39.29 ± 14.66^{b}	50.29 ± 13.41^{a}

Same letters indicate that the averages do not differ by the Kruskal-Wallis test ($P < 0.05$).

2004). Ethanolic extracts of Asteraceae species were toxic to *S. frugiperda* and the yield of these crude extracts varied from 2.08 to 5.80% (w/w) (Tavares et al., 2009). This shows that this method of isolation and purification of the piperine from *P. nigrum* fruits was efficacious, which would make its utilization in large scale viable. The higher mortality of recently laid *S. frugiperda* eggs than those of one- or two-days-old of this insect with piperine at 10 g/L (1%) confirms that the older eggs are more resistant to plants extracts due to its higher maturity and because they hinder the entrance of external products (Tavares et al., 2010). This was proven by the mortality of 97.7% of one-day-old *S. frugiperda* eggs with extracts of *Lychnophora ericoides* Mart. or *Trichogonia villosa* Sch. Bip. Former Baker (Asteraceae) and of 72.0% for two-days-old eggs of this insect with *Lepidaploa lilacina* (Mart. Former DC.) H. Rob. (Asteraceae) (Tavares et al., 2009). Insecticides can cause deformation in the shell of *Spodoptera exigua* Hübner, 1808 (Lepidoptera: Noctuidae) eggs and interfere in reproduction and population growth of this insect (Adamski et al., 2009). Young and new-born *Leptinotarsa decemlineata* Say, 1824 (Coleoptera: Chrysomelidae) larvae were more susceptible to *Piper tuberculatum* Jacq. (Piperaceae) extracts than those with four-days-old, whereas the *P. nigrum* extract reduced the larval survival of this insect in up to 70% (Scott et al., 2003). This confirms that younger insects are more susceptible to botanical extracts than older ones.

The higher mortality of eggs with one- or two-days-old of *S. frugiperda* with piperine at 20 g/L (2%) than with this product at 10 g/L (1%) shows the higher efficiency of the former concentration to control *S. frugiperda* eggs. On the other hand, *Piper* spp. (Piperaceae) extracts, in concentrations lower than 1 g/L (0.l%) were considered effective in potato *Solanum tuberosum* L. (Solanaceae) plantations attacked by *L. decemlineata* (Scott et al., 2003).

The poisonous effect of products derived from Piperaceae, such as the synthetic amide [3-(3’,4’-methylenedioxyphenyl)-2-(*E*)-propenoyl]piperidine, caused higher mortality of *S. frugiperda* larvae (Batista-Pereira et al., 2006). The larvae mortality in the fourth instar of *Spodoptera litura* F., 1775 (Lepidoptera: Noctuidae) was higher with artificial diets with amides of Piperaceae after six days of feeding. On the other hand, one of these amides caused the 100% mortality of *Chilo suppressalis* Walker, 1863 (Lepidoptera: Pyralidae) at 500 mg/kg and did not affect *S. litura* (Miyakado et al., 1985).

The lower hatching of larvae of two-days-old than those recently laid or with One-day-old of *D. saccharalis* with piperine at 10 g/L (1%) confirms that older eggs are more resistant to plant extracts (Tavares et al., 2010). The higher hatching of larvae with one- or two-days-old of *D. saccharalis* with piperine at 20 g/L (2%) than at 10 g/L (1%) points out that the former concentration was more efficient to control the eggs of this insect. The mortality of larvae at fourth stage of *D. saccharalis* was higher in treatments with dichloromethane-methanol extract of the *P. tuberculatum* seeds and two isobutyl amides (4,5-dihydropiperlonguminine and pellitorine) after 24, 48 and 72 h of application of the insecticides (Debonsi et al., 2009). These results showed the possibility of utilizing products with piperine base in the control of *D. saccharalis* eggs, due the capacity of these products of adhering to the eggs of these insects and also to fowls (Terners and Kraüse, 2002). The instar of egg of the *D. saccharalis* is crucial for the effective control of this pest because after the larvae penetrate the stem of the corn *Zea mays* L. or sugarcane *Saccharum* spp. L. (Poaceae) the control would be more difficult (Kumar and Mihm, 2002). The attempt to control *D. saccharalis* with synthetic insecticides causes environmental contamination (Rodriguez et al., 2001). *Piper aduncum*, *P. tuberculatum* and *Piper decurrens* Miq. (Piperaceae) and compounds of these species caused mortality and deterrent effect on *Ostrinia nubilalis* Hübner, 1796 (Lepidoptera: Pyralidae) (Bernard et al., 1995) and Piperaceae extracts on *Diprion similis* Hartig, 1837 (Hymenoptera: Diprionidae), *Malacosoma disstria* Hübner, 1820 (Lepidoptera: Lasiocampidae), *Lymantria dispar* L., 1759 (Lepidoptera: Lymantriidae) and *Choristoneura fumiferana* Clemens, 1865 (Lepidoptera: Torticidae) (Scott et al., 2007). The efficiency of botanical products on insects may vary with UV radiation, temperature, solvent used or concentration of extract (Moreira et al., 2007). The genetic of the plant, the extraction time of the compounds (Sidhu et al., 2004) and the part of the plant used for producing the extracts further influence the activity of some compounds againstinsects (Siskos et al., 2007).

The increase in the germination of *C. sativus* in the concentration of 0.5 mg/kg may be understood due to the fact that this species has higher energetic reserve in the seeds, in relation to other dicotyledons. This makes it less sensitive to poisonous effects of external compounds, especially in low concentrations (Macías et al., 2000). On the other hand, secondary metabolites secreted by plants with allelopathic action, inhibit the germination, growth and enzyme activity of plants (Veiga et al., 2007).

The germination inhibition of the seeds of dicotyledons was higher at 5.0 mg/kg than at 0.5 mg/kg, suggesting the higher inhibition capacity of the piperine at higher concentration. On the other hand, this was not detected in the *A. cepa*. The *Piper guineense* Schumach and Thonn. (Piperaceae) essential oil inhibited the lettuce growth, only in higher concentration (> 400 mg/kg) (Oguntimein and Elakovick, 1991) and the *Ottonia martiana* Miq. (Piperaceae) essential oil and of the amides piperovatine and isopiperlonguminine caused lower effect of the tested fractions amides, on the lettuce germination, even in higher concentrations (from 100 to 800 mg/kg) (Cunico et al., 2006).

The lower length of the seedlings of dicotyledoneous treated with the piperine shows that this compound, even in low concentrations, affects their development. This is also shown by the higher effect of *P. nigrum* leachates in the germination, growth, production and accumulation of chlorophylls in *Vigna mungo* L. Hepper (Fabaceae) inhibiting the enzymes responsible for the conversion of precursors of porphyrins into chlorophylls, thus decreasing the concentration of this pigment in the plant. Even with the phytotoxic potential of *P. nigrum*, phytochemical researches for the evaluation of the compounds responsible for this activity were not done (Siddiqui, 2007). This corroborates the reports that the piperine is the main compound of the *P. nigrum* fruits and responsible for the allelopathic effect of this plant (Siddiqui, 2007). On the other hand, other amines may cause allelopathic effect in plants, such as the capsaicin isolated from *Capsicum annuum* L. (Solanaceae), which showed substantial differences in the inhibition of the germination and growth of the plants, such as alfalfa *Medicago sativa* L., crabgrass *Digitaria sanguinalis* L. (Leguminosae), cress *Lepidium sativum* L. (Brassicaceae), timothy *Phleum pretense* L., ryegrass *Lolium multiflorum* Lam. (Poaceae) and *L. sativa*, suggesting that this amine has allelopathic effect on some of them (Kato-Noguchi and Tanaka, 2003). Piperine caused lower hatching in *D. saccharalis* than of *S. frugiperda* and this compound at 20 g/L (2%) concentration showed higher possibility to be utilized to control these insects. The low persistence and high degradability of botanical extracts in field conditions are some obstacles to be solved. Further studies should be done under semi-field or field to confirm these results. Piperine was selective for the germination and growth of mono and dicotyledons, but it had higher impact in the development than in the germination of these species. Therefore, further studies should be directed to elucidate these effects under more realistic (field) and its potential impact on plant development and production. Therefore, possibility to utilize piperine in the organic agriculture in programs of management of pests and weeds is suggested.

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