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Toxicity of Black Pepper Oil and Piperine on Egg Hatchability of the European Corn Borer, *Ostrinia nubilalis* (Hubner) and Evaluate their Potential Phytotoxicity

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ABSTRACT

The use of plant-derived oils and extracts and their active constituents as safe alternatives for pest control has been emphasized as an important tool to be incorporated in integrated pest management (IPM) programs that can help to fulfill the efficacy and safety of pest control products. The objective of this work was to determine the ovicidal activity of petroleum ether extract of black pepper fruits and piperine isolated from acetone extract on the egg hatchability of the European corn borer, *Ostrinia nubilalis* (Hubner) in the laboratory. The potential phytotoxic effect of black pepper extracts on the seed germination of the little seed canary grass (*Phalaris minor*), sorghum (Sorghum *bicolor*) and cucumber (*Cucumis sativus*) was also studied. The ovicidal activity of the tested applied concentrations of black pepper oil extract and piperine was increased with the increase in concentrations, and egg mortality (non-hatch) percentage ranged between 20.4 to 100%. Pepper oil extract and piperine showed significant inhibitory activity on seed germination of canary grass at all concentrations tested with good selectivity with sorghum and cucumber at the applied lower concentrations of 500 and 1000 µg/ml. Results suggest that black pepper oil extract and piperine are promising alternative biopesticidal agents for the control of *O. nubilalis* egg masses and reducing the germination of the harmful canary grass in field crops and could be included as a viable component of the IPM system.

INTRODUCTION

Sweet corn (*zea mays* L.) is one of the most important cereal crops in Egypt and many African and European countries. The major insect pest damaging sweet corn is the European corn borer (ECB), *Ostrinia nubilalis* (Hubner). It is a serious insect pest of corn and other cultivated plants in Africa, Europe, Asia and North America (Lee *et al.,* 1999, Boiteau & Noronha, 2007). The ECB has rapidly become one of the most important insect pests of corn because of its ability to adapt to new environments and attack a large number of plants. They are infesting corn plants in the whorl and tassel stage (Ellsworth and Bradley

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1992; Lee *et al.,* 1999). Due to the significant economic value of field corn, and popcorn for the fresh market, Chemical insecticides are employed to control this pest. However, the use of synthetic insecticides on corn fields caused several environmental problems including soil and groundwater contamination, insect resistance and toxicity to non-target organisms. For controlling the ECB, integrated pest management (IPM) has recently gained more attention (Lee *et al.,* 1999, Duke *et al.,* 2010).

Plant-derived essential oils and natural compounds could be used as an alternative to synthetic insecticides (Isman,2015, Isman, 2020). They have many useful biological activities with different modes of action on various target insect pests including Lepidoptera larvae. Essential oils contain plant secondary metabolites especially monoterpenes, and sesquiterpenes which can assist plants as chemical defenses against phytophagous insects. They act as insect repellents, oviposition deterrents and antifeedants as well as killing materials against insects and weeds (Batish *et al.,* 2007, Abdelgaleil *et al.,* 2022). Reduced-risk natural products such as essential oils have a very low impact on human health, low toxicity to non-target organisms, a low potential for groundwater contamination, and very low potential for pest resistance and have the ability for compatibility with IPM programs (Isman, 2006, Isman, 2015, Zhou *et al.,* 2021). The adoption of using plant-derived botanical materials within conventional corn production can result in high yields and low pest densities, equivalent to those obtained with traditional synthetic insecticides, with conserving predator and parasite population densities (Cutler *et al.,* 2006, Gurr *et al.,* 2012). The essential oils contain a mixture of several active compounds with different modes of action on insect pests that make them useful tools for pest control and resistance management strategies. Furthermore, the biological effects of allelopathy and phytotoxicity of natural plant oils might be exploited for weed control especially invasive weeds such *as Phalaris* spp. (Dudai *et al.,* 1999, Chowhan *et al.,* 2011, Tang *et al.,* 2018). This annual weed, native to the Mediterranean region is one of the most harmful crop weeds in the world. Herbicide application is the most effective control for this weed. The use of water or oily extracts of herbicidal (allelopathic) plants can be effective integrated management of herbicide resistance against *P.minor* in maize or wheat for eco-friendly and sustainable weed management (Yasin *et al.,* 2011). The allelopathic activities of secondary compounds of plants from the Piperaceae family and some other plant species, that include monoterpenes, sesquiterpenes, phenolic compounds and amide derivatives can affect seed germination and plant growth (Singh *et al.,* 2003, Batish *et al.,* 2007, De Almeida et al., 2010). The Piperaceae family includes over 1000 species. Many species of the genus Piper, such as black pepper, *Piper nigrum* L. contain biologically active compounds that have insecticidal and medicinal properties (Scott *et al.,* 2008, Pereira *et al.,* 2015).

Several investigators have reported that the amide compounds present in Piper species have insecticidal potential due to their good efficacy and knockdown effects on target insect pests (Scott *et al.,* 2003). Also, *Piper nigrum* amides showed allelopathic activities via their effects on seed germination and plant growth due to its inhibition of photosynthetic rate in many different plant species (Azizi and Fugi, 2005, Siddiqui, 2007, Awojide *et al.,* 2021). Several compounds from black pepper like piperine and piperettine as well as β-pinene may have effects as allelochemicals due to the phenolic properties of amides and the phytotoxic effects of the oxygenated monoterpenes alpha- and β-pinene (Chowhan *et al.,* 2011). Black pepper fruits therefore might be a source of naturally occurring insecticides and herbicides (Su, 1977, Chowhan *et al.,* 2011, Hussain *et al.,* 2017). Several modes of action of plant essential oils and extracts have been reported concerning bioactivities against insect pests including cuticle disruption, reduced fecundity, insect molting inhibition, growth reduction, respiratory inhibition, anti-feeding and repellent activity, and toxic effects

(Hussain *et al.*, 2017, Eldoksch *et.al.*,2012, Elrehawy and Eldoksch, 2022), as well as their potential allelopathic effects on plants (Eldoksch *et al.*, 2001, Singh *et.al.*, 2009).

Although black pepper oil and piperine are toxic to some insects, larvae and adults, their effects on the viability of the ECB eggs and their phytotoxicity to the invasive canary grass weed have not been investigated yet. The aim of this study was to evaluate the ovicidal activity of black pepper oil extract and piperine on the egg hatchability of the ECB, *O.nubilalis* to explore their potential use as a part of a corn-integrated pest management program. The phytotoxic effect of black pepper oil extract and piperine on the seed germination of the harmful weed, canary grass and two of the economic plants, sorghum and cucumber were also investigated.

MATERIALS AND METHODS

Preparation of *Piper nigrum* Oil Extract:

100 g of powdered black pepper, *Piper nigrum* fruits were extracted successively by petroleum ether (40-60) and acetone using a soxhlet extractor apparatus for 4h each. The pet-ether extract obtained was concentrated using a rotary evaporator (45-50 °C) to obtain the crude oily extract.

Preparation of Piperine from Acetone Extract:

The black pepper powder that remained after pet-ether extraction was air dried for 24 h at room temperature and then re-extracted with acetone and the obtained solution was evaporated to 50% volume and kept in the refrigerator for 2 days during which a yellow crystal material was precipitated. Thus after exhaustive washing with distilled water, it was recrystallized with acetone and gave yellowish crystals identified as piperine by using thin layer chromatography analysis using plastic sheets (20x20 cm) pre-coated with silica gel/UV 254 with layer 0.25 mm and gave (R_f value of 0.28) as shown in Table (1), and by infrared, (IR) analysis which indicated that the isolated compound is piperine (Fig.1), and its IR spectrum was found to be identical to piperine IR spectrum available in the literature according to Pouchert 1975; Pungor 1995) and by physical data (mp,128-129 °C).

IR absorption spectrum of piperine (Fig.1) showed the following absorption bands: Several bands of 760, 800 and 850 cm^{-1} attributed to the stretching of aromatic C-H. Bands of 1000, 1020 and 1030 cm^{-1} are attributed to conjugated C=C. A band of strong intensity at 930 cm^{-1} is probably related to C-O stretching, a weak Overton of this band appeared at 1860 cm^{-1}. Several bands of strong intensities characteristic of C-O-C stretching vibration at 1250 and 1100 cm^{-1}. Two bands of strong intensities at 1450 and 1370 cm^{-1} due to C-H banding of methylene. Two bands of medium and strong intensities at 1590 and 1505 cm^{-1} respectively, characteristic of aromatics. A band of medium intensity at 1680-1672 was attributed to C=O stretching vibration and a band of strong intensity at 2950 cm^{-1} was attributed to the stretching of aromatic and aliphatic C-H.

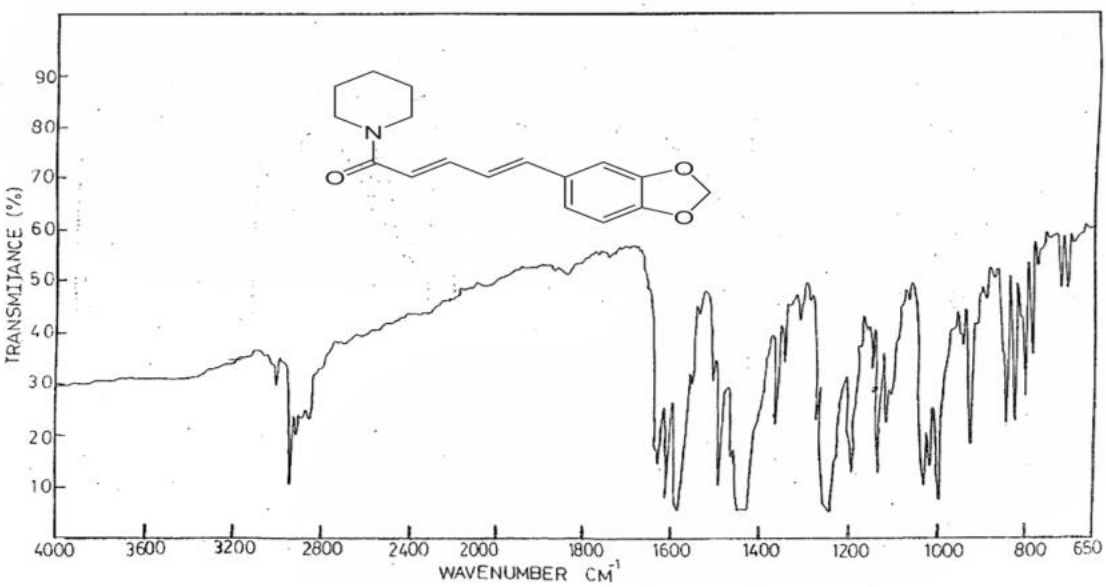

Fig. 1. Chemical structure of piperine and its IR spectrum

Bioassay of Ovicidal Activity:

The ovicidal activity was evaluated using corn foliage containing egg masses attached 1-2 days old that were collected early in the season from an untreated sweet corn field located in Abee's district, Alexandria Governorate during the 2022 summer season and transferred to the laboratory for bioassay. Aqueous emulsion series of concentrations for each test material (black pepper oil extract and piperine) were prepared (0, 1000, 4000, and 8000 µg/ml). Egg masses each containing about 30-40 eggs were used for each replicate. At least 120 eggs were used for each concentration of plant material and four replicates were made for each concentration. Egg masses were dipped for 5 seconds in each test solution. The untreated eggs were dipped in water plus 0.1% Tween- 80 was served as control. Preparing emulsions for black pepper oil and piperine required the addition of 0.1% Tween-80 to emulsify the plant material in water. The oil of black pepper was dissolved in ethanol and diluted with distilled water to the final concentration required using Tween- 80, 0.1%. Piperine was dissolved in acetone and diluted with distilled water to the required concentration using Tween- 80 solution, 0.1%. After treatments, the egg masses were allowed to dry at room temperature and then placed in tubes covered with a muslin cloth. The daily examination was made for two days after the hatching of water-treated controls. The percentage of hatching was determined by counting the number of hatched larvae in treated and untreated eggs and calculating the percentage of ovicidal activity (mortality %) as described by Sangha et al. (2017):

$$\textbf{OAP (\%)} = (\text{EUC} - \text{EUT}/\ 100\text{- EUC}) \times 100$$

where OAP is ovicidal activity percentage, EUC is un-hatched eggs in the control, and EUT is un-hatched eggs in treatments. The average percentage of ovicidal activity was corrected using Abbott's formula (1925) and the data set of the hatching were subjected to analysis of variance (ANOVA) and the means were compared with Duncan's multiple range test (P=0.05).

Plant Seeds, Dose-Response Studies and Phytotoxicity Evaluation:

Seeds of sorghum (*Sorghum bicolor* L., Family Poaceae), cucumber (*Cucumis sativus* L., Fam. Cucurbitaceae) and the weed little seed canary grass (*phalaris minor* Retz, Fam. Poaceae) were purchased from supermarket and surface sterilized in 70% ethanol for 10 min, then 2% sodium hypochlorite for 5 min, and washed two times with distilled water. To test the inhibitory effect of pepper oil and piperine, different concentrations of each tested plant materials were prepared (0, 500, 1000, 4000 and 8000 µg/ml) and their effect was studied on the germination rate of tested weed and crop plants. Twenty healthy seeds of each plant type were placed in a Petri dish (9cm diameter) containing three layers of Whatman No.1 filter circle moistened with 7ml of different concentrations of each plant material solution. A similar treatment with water containing 0.1% Tween- 80 is served as control. For each concentration, four, replication was made. All the Petri plates were kept in a growth chamber maintained at 16/8h light/dark period, 26 ± 2 °C temperature, and 80% relative humidity. After 14 days, germinated seeds that gave healthy seedlings were counted. The experiment was conducted by using a completely randomized block design with 4 replicates.

Statistical Analysis:

All data were subjected to analysis of variance (ANOVA) and the least significant Difference Test (LSD) was employed to compare the treatment means (P=0.05), means were compared by Duncan's multiple range test using the SAS Program version 9.1 (SAS Institute, 2010).

RESULTS AND DISCUSSION

Thin Layer Chromatography (TLC) analysis:

Piperine in crystal form was separated from acetone extract of black pepper powder with high purity as indicated by TLC analysis that showed one spot with R_f value (0.28) with yellow color using anisaldehyde reagent. Also, TLC analysis of black pepper oil extract was applied using Carbon tetrachloride: Ethyl acetate: Chloroform (8:1:1) as a solvent system for developing the spots, and the air-dried chromatoplates were sprayed with an anisaldehyde reagent for revealing the spots, the results exhibited 8 spots (compounds) with R_f values (0.15, 0.28, 0.42,0.48, 0.51, 0.72, 0.81 and 0.88) as indicated in Table (1).

Table 1: Thin Layer Chromatography of petroleum ether oily extract of black pepper, *Piper nigrum* and piperine**.**

Plant extracts	Solvent system	No. of spots	R_f values
Pet-ether oily extract	CCl_4: ETOAC: $CHCl_3$ (8: 1: 1)	8	(0.15, brown), (0.28, yellow), (0.42, violet), (0.48,violet),(0.51,blue), (0.72, red),(0.81, brown) , (0.88, violet)
Piperine	CCl_4: ETOAC: $CHCl_3$ (8: 1: 1)	1	(0.28, yellow)

A solvent system was used for developing the spots (compounds), and an anisaldehyde reagent for revealing the spots.

Ovicidal Activity Evaluation:

The ovicidal activity of essential oil of black pepper and piperine isolated from acetone extract were tested on 1–2 days old eggs of a field strain of the European corn borer, *Ostrinia nubilalis* (Hubner) is shown in Table (2). The results clearly indicate that the tested plant extracts of black pepper oil and piperine varied in their ovicidal activity which may be due to the differences in plant components and chemical structures, physical and chemical properties and mode of action of each plant material applied. Concerning black pepper oil, the high concentration tested (8000 µg/ml) caused complete inhibition of egg hatch giving (100%) egg mortality and a significant difference with piperine at the same concentration with egg mortality (81.3%). The data also indicated that black pepper oil was more effective against eggs than piperine at all concentrations tested with a significant difference from the control. Generally, for each plant extract the increase in concentration led to an increase in egg mortality percentage (non-hatch) as shown in Table (2). Adamski et al., (2009) demonstrated that fenitrothion insecticide can cause deformation in the shell of *Spodoptera exigua* (Hubner), (Lepidoptera: Noctuidae) eggs and interfere in the reproduction and population growth of this insect. Extracts of black pepper *P.nigrum* L. (Piperaceae) and other species of this family are toxic to Lepidoptera insects (Paula *et al.,* 2000; Pereira *et al.*, 2006) and species of the family Piperaceae are rich in amides as the major secondary metabolites which responsible for the insecticidal properties of this plants. Tavares *et al.* (2011) isolated piperine from dried fruits of black pepper using extraction by ethanol. They reported that piperine at 1% caused a higher mortality of eggs laid by *Spodoptera frugiperda* (Lepidoptera: Noctuidae) and *Diatraea saccharalis* (Lepidoptera: Pyralidae). Vinturelle *et al.,* (2017) indicated that *P.nigrum* essential oil inhibited egg-laying by up to 96% in a concentration-dependent manner suggesting it reduced the tick *Rhipicephalus microplus* (*Boophilus*) fecundity.

Table 2: Mortality (%) of *Ostrinia. nubilalis* eggs attached to sweet corn leaflets treated with black pepper oil and piperine[2]

Treatment	**Mortality (%) at indicated concentrations**			
	Control	1000 (µg/ml)	4000 (µg/ml)	8000 (µg/ml)
Pepper oil	0e	36.8 ± 3.6c	96.5 ± 2.2a	100a
Piperine	0e	20.4 ± 3.3d	74.7 ± 3.8b	81.3 ± 5.2b

Mortality values (mean ± standard error), LSD 5%, (8.3).
Piperine compound isolated from acetone extract of black pepper fruit powder.

Phytotoxicity Effect of Black Pepper Oil and Piperine:

Results in Table (3) indicated that all the applied concentrations of black pepper, *P.nigrum* oil on canary grass, *P.minor* seeds caused a significant ($P<0.05$) reduction in germination compared with the control. The detected reductions were directly proportional to the applied concentrations. It was observed that the higher concentrations (8000 and 4000 µg/ml) completely inhibited the seed germination of both *P.minor* and C. sativus with 100% inhibition. The lower concentration of (500 µg/ml) gave 62.2% inhibition of seed germination of *P.minor* with good selectivity with *S. bicolor* and *C. sativus* with 4.1% and 6.5% inhibition respectively.

Results illustrated in Table (4) showed that all the tested concentrations of piperine on canary grass seeds caused a significant ($p<0.05$) reduction in seed germination which was directly proportional to the concentrations tested. The reduction in seed germination percentage reached 100% in response to the high concentration (8000 µg/ml) while giving a 51.9% reduction at the lower concentration (500 µg/ml) with a significant difference from the control. The data also indicated that at lower concentrations of (1000 and 500 µg/ml) *P. minor* weed seeds were more sensitive to piperine treatment than *S. bicolor* and *C. sativus* seeds with promising selective action (Table 4). The mode of action of the inhibitory effect of black pepper oil and piperine is unknown, it might be due to that pepper oil contains a mixture of amides, monoterpenes and sisquiterpenes and other different compounds such as β-cariophyllene (23.5%), 3-carene (22.2%), d-limonene (18.4%), β-pinene (8.9%) and alpha-pinene (4%) (Liu *et al.*,2007) that might lead to inhibition of the mitotic activity of growing cells. Abrahim *et al.*, (2000) indicated the inhibitory effects of four monoterpenes namely camphor, limonene, eucalyptol and alpha-pinene on the seed germination, primary root growth and mitochondrial respiration of maize (*Zea mays*). Awojide *et al.*, (2021) indicated the phytotoxicity of black pepper essential oil against *Solanum lycopersicum* L., *Zea mays* L., and *Vigna unguiculata* L. and found that essential oil formulation exhibited phytotoxicity and high inhibitory activity on seed germination and root and shoot growth. β-pinene is a principal component of essential oils of several aromatic plants including black pepper which are involved in many ecological interactions including allelopathy (Chowhan *et al.*, 2011). They also indicated that β-pinene significantly reduced the root and coleoptile length of rice, and exposure of seeds and seedlings to β-pinene reduced germination and total chlorophyll content in rice coleoptiles suggesting a negative impact on photosynthesis. Also, Singh *et al.* (2009) reported that the essential oil of *Artemisia scoparia* inhibited plant growth by generating reactive oxygen species and causing oxidative damage. In conclusion, from the obtained results we suggest the possibility of utilizing products with black pepper essential oil or piperine base in the control of European corn borer egg masses as safe alternative natural products and also can be incorporated as a viable component in integrated insect and weed management of cereal crops.

Table 3: Effect of different concentrations of Black pepper essential oil on germination (%) of canary grass, sorghum and cucumber.

Concentration µg/ml	**Germination (%)**		
	Canary grass	**Sorghum**	**Cucumber**
8000	0e	0e	0e
4000	0e	14.2d	0e
1000	12.6d	38.5c	63.8b
500	37.8c	95.9a	93.5a
0	100a	100a	100a
LSD 5%	(8.4)		

Oil of black pepper is dissolved in ethanol and diluted by 0.1% Tween-80 to the required concentrations.

Table 4: Effect of different concentrations of piperine on germination (%) of canary grass, sorghum and cucumber.

Piperine µg/ml	**Germination (%)**		
	Canary grass	**Sorghum**	**Cucumber**
8000	0.0d	0.0d	0.0d
4000	8.6c	45.5b	18.2c
1000	20.7c	88.3a	37.5b
500	48.1b	88.7a	91.4a
0	100a	100a	100a
LSD 5%	(12.8)		

Piperine is dissolved in acetone and diluted by 0.1% Tween-80 to the required concentrations.

REFERENCES

Abbott, W. S. (1925). A method of computing the effectiveness of an insecticide. *Journal of Economic Entomology*, 18(2), 265-267.

Abdelgaleil, S. A., Al-Nagar, N., Abou-Taleb, H. K., & Shawir, M. S. (2022). Effect of monoterpenes, phenylpropenes and sesquiterpenes on development, fecundity and fertility of *Spodoptera littoralis* (Boisduval). *International Journal of Tropical Insect Science*, 42(1), 245-253.

Abrahim, D., Braguini, W. L., Kelmer-Bracht, A. M., & Ishii-Iwamoto, E. L. (2000). Effects of four monoterpenes on germination, primary root growth, and mitochondrial respiration of maize. *Journal of Chemical Ecology*, 26(3), 611-624.

Adamski, Z., Machalska, K., Chorostkowska, K., Niewadzi, M., Ziemnicki, K., & Hirsch, H. V. (2009). Effects of sublethal concentrations of fenitrothion on beet armyworm (Lepidoptera: Noctuidae) development and reproduction. *Pesticide Biochemistry and Physiology*, 94(2-3), 73-78.

Azizi, M., & Fuji, Y. (2005,). Allelopathic effect of some medicinal plant substances on seed germination of *Amaranthus retroflexus* and *Portulaca oleraceae*. *In International Symposium on Improving the Performance of Supply Chains in the Transitional Economies* 699 (pp. 61-68).

Batish, D. R., Singh, H. P., Setia, N., Kohli, R. K., Kaur, S., & Yadav, S. S. (2007). Alternative control of littleseed canary grass using eucalypt oil. *Agronomy for Sustainable Development*, 27(3), 171-177.

Boiteau, G., & Noronha, C. (2007). Topical, residual and ovicidal contact toxicity of three reduced-risk insecticides against the European corn borer, *Ostrinia nubilalis* (Lepidoptera: Crambidae), on potato. *Pest Management Science: formerly Pesticide Science*, 63(12), 1230-1238.

Chowhan, N., Singh, H. P., Batish, D. R., & Kohli, R. K. (2011). Phytotoxic effects of β-pinene on early growth and associated biochemical changes in rice. *Acta Physiologiae Plantarum*, 33(6), 2369-2376.

Cutler, G. C., Scott-Dupree, C. D., Tolman, J. H., & Harris, C. R. (2006). Toxicity of the insect growth regulator novaluron to the non-target predatory bug *Podisus maculiventris* (Heteroptera: Pentatomidae). *Biological Control*, 38(2), 196-204.

De Almeida, L. F. R., Frei, F., Mancini, E., De Martino, L., & De Feo, V. (2010). Phytotoxic activities of Mediterranean essential oils. *Molecules*, 15(6), 4309-4323.

Dudai, N., Poljakoff-Mayber, A., Mayer, A. M., Putievsky, E., & Lerner, H. R. (1999). Essential oils as allelochemicals and their potential use as bioherbicides. *Journal of Chemical Ecology*, 25(5), 1079-1089.

Duke, S.O., Cantrell, C. L., Meepagala, K. M., Wedge, D. E., Tabanca, N., & Schrader, K. K. (2010). Natural toxins for use in pest management. *Toxins*, 2(8), 1943-1962.

Eldoksch, H, Houssien, A., Mansy,A. (2001) Plant growth response to caraway, sage and radish essential oils and their combination with mitribuzin. *Alexandria Science Exchange Journal*, 22(4):333-344.

Eldoksch, H.A., Dewer,Y.& Kenawy,A. (2012).Fumigant toxic action and repellent effects of plant essential oils against the two spotted spider mite *Tetranychus urticae* Kock (Acari: Tetrachidae). *Alexandria Science Exchange Journal*,33(4):245-260.

Ellsworth, P. C., & Bradley Jr, J. R. (1992). Comparative damage potential and feeding dynamics of the European corn borer (Lepidoptera: Pyralidae) and cotton bollworm (Lepidoptera: Noctuidae) on cotton bolls. *Journal of Economic Entomology*, 85(2), 402-410.

Elrehawy, E.S. & Eldoksch, H.A. (2022). The insecticidal and antifeedant activity of *Calotropis procera* latex and foliar extracts against the red palm weevil, *Rhynchophorus ferrugineus* (Olivier). *Egyptian Academic Journal of Biolgical Sciences (F. Toxicology and Pest Control)*,14(2): 25-33.

Gurr, G. M., Wratten, S. D., & Snyder, W. E. (Eds.). (2012). Biodiversity and insect pests: key issues for sustainable management. John Wiley & Sons.

Hussain, A., Rizwan-ul-Haq, M., Al-Ayedh, H., & Aljabr, A. M. (2017). Toxicity and detoxification mechanism of black pepper and its major constituent in controlling

*Rhynchophorus ferr*ugineus Olivier (Curculionidae: Coleoptera). *Neotropical Entomology*, 46(6), 685-693.

Isman, M. B. (2006). Botanical insecticides, deterrents, and repellents in modern agriculture and an increasingly regulated world. *Annul Review of Entomology,* 51, 45-66.

Isman, M. B. (2015). A renaissance for botanical insecticides? *Pest Management Science,* 71(12), 1587-1590.

Isman, M. B. (2020). Commercial development of plant essential oils and their constituents as active ingredients in bioinsecticides. *Phytochemistry reviews*, 19(2), 235-241.

Lee, S., Tsao, R., & Coats, J. R. (1999). Influence of dietary applied monoterpenoids and derivatives on survival and growth of the European corn borer (Lepidoptera: Pyralidae). *Journal of Economic Entomology*, 92(1), 56-67.

Liu, L., Song, G., & Hu, Y. (2007). GC–MS Analysis of the Essential Oils of *Piper nigrum* L. and *Piper longum* L. *Chromatographia*, 66(9), 785-790.

Paula, V. F. D., Barbosa, L. C. D. A., Demuner, A. J., Piló-Veloso, D., & Picanço, M. C. (2000). Synthesis and insecticidal activity of new amide derivatives of piperine. *Pest Management Science*, 56(2), 168-174.

Pereira, E., Farias, E., Ribeiro, A., Alvarenga, E., Aguiar, A., Ferreira, J., & Picanço, M. (2019). Toxicity of piperine amide analogs toward the tomato pinworm *Tuta absoluta* (lepidoptera: Gelechiidae) and risk assessment for two predators. *Horticulturae*, 5(4), 70.

Pouchert, C.J. (1975) Aldrich library of infrared spectra, 2nd edition, Aldrich Chemical Co. Milwaukee, Wisconisn.

Pungor, E.A (1995) A practical guide to instrumental analysis CRC,Boca Raton,1995 pp 74-80.The Merck Index,10th ed., pp 284-285.

Sangha, J.S., Astatkie,T. and Cutler,G.C. (2017). Ovicidal, larvicidal and behavioral effects of some plant essential oils on diamondback moth (Lepidoptera:Plutellidae).*The Canadian Entomologist*, 149:639-648.

SAS Institute. (2010). SAS Statistics and Graphics Guide, Release 9.1. SAS Institute, Cary, North Carolina, 27513, USA.

Scott, I. M., Jensen, H. R., Philogène, B. J., & Arnason, J. T. (2008). A review of Piper spp. (Piperaceae) phytochemistry, insecticidal activity and mode of action. *Phytochemistry Reviews*, 7(1), 65-75.

Scott, I. M., Jensen, H., Scott, J. G., Isman, M. B., Arnason, J. T., & Philogène, B. J. R. (2003). Botanical insecticides for controlling agricultural pests: piperamides and the Colorado potato beetle *Leptinotarsa decemlineata* Say (Coleoptera: Chrysomelidae). Archives of Insect Biochemistry and Physiology: Published in *Collaboration with the Entomological Society of America*, 54(4), 212-225.

Siddiqui, Z.S. (2007). Allelopathic effects of black pepper leachings on *Vigna mungo* (L.) Hepper. *Acta Physiologiae Plantarum,* 2007.29:303-308.

Singh, H.P., Kour, S., Batish,D.R. & Kohli,R.K. (2009). Essential oil of *Artemisia scoparis* inhibits plant growth by generating reactive oxygen species and causing oxidative damage. *Journal* of *Chemical Ecology,* 35:154-162.

Singh,H.P.,Batish,D.R, Kohli, R.K. (2003) Allelopathic interactions and allelochemicals: new possibilities for sustainable weed management. *Critical* Reviews in *Plant Sciences,* 22:239-311.

Su, H.C.F. (1977) Insecticidal properties of black pepper to rice weevil's and cowpea weevils. *Journal of Economic Entomology,* 70 (1) :18-21.

Tang, D., Yang, X., Li,Y., Xiao,W.,Gu,X., Jing,D., Li,R.,Fu,Y.,(2018). Investigations on the occurrence, damage and control of the invasive weed. Phalris spp. In Yunnan Province. *Plant Protection,* 44,167-169.

Tavares, W.S, Cruz, I. Petacci, F., Freitas, S.S., Serrao, J.E., Zanuncio, J.C. (2011) Insecticide activity of piperine: Toxicity to eggs of *Spodoptera frugiperda* (Lepidoptera: Noctuidae) and *Diatraea Saccharalis* (Lepidoptera: Pyralidae) and phytotexicity on several vegetables. *Journal of Medicinal Plants Research*, 5:5301-5306.

Vinturelle, R., Mattos, C., Meloni, J., Nogueira, J.,Nunes, M.J., Lione, V.et al. (2017). Invitro evaluation of essential oil derived from *Piper nigrum* (Piperaceae) and *Citurs limonum* (Rutaceae) against the Tick *Rhipicephalus* (*Boophilus) microplus* (Acari: Ixodidae). *Biochemistry Research International,* 2017, 5342947

Yasin,M., Iqbal,Z., Safdar,M., Ali,A., Asafi,M, Aziz,M., Perves,M. (2011). *Phalaris minor* control, resistance development and strategies for integrated management of resistance to fenoxaprop-ethyl. *African Journal of Biotechnology*, 10 (56):11802-11807.

Zhou, L., Li, C., Zhang, Z., Li, X., Dong, Y., Coa, H. (2021). Biological activity and safety evaluation of monoterpenes against the peach aphid (*Myzus persicae* Sulzer) (Hemiptera: Aphididae), *International Journal of Tropical Insect Science*, 41:2747-2754.