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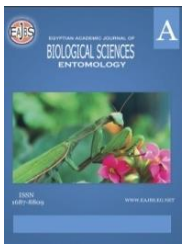

# Biochemical Composition, Toxicity and Bioactivities of the Essential Oil extracted from *Coffea arabica* L. husks against the Cotton Leafworm, *Spodoptera littoralis* (Boisduval) (Lepidoptera: Noctudiae)

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## ABSTRACT

The immense and ever-growing amounts of agricultural waste have come to be a main concern throughout the world. Therefore, approaches for their processing and value-added reusing are required to enable a sustainable utilization of feedstocks and decrease the environmental burden. In the current study, an essential oil extracted from the husk of Arabic coffee (*Coffea arabica*) was analyzed chemically using gas chromatography-mass spectrometry (GC-MS) and its constituents were screened for insecticidal activity against the fourth larval instars of the cotton leafworm *Spodoptera littoralis* (Boisdural). The GC/MS analysis revealed that the arabic coffee included 13 compounds of which Oleic acid comprises the largest proportion 65 % which is known for its insecticidal efficacy. Other compounds that have insecticidal activities were also separated; Cyclononasiloxane, octadecamethyl-, n-Hexadecanoic acid, 9,12-Octadecadienoic acid (Z, Z)- and N-Isobutyl-11-(3,4-methylenedioxyphenyl)-2E,4E,10E-undecatrienoic amide. The oil extract was tested against the 4$^{th}$ larval instars. The results indicated the efficacy of the extract as larvicide with the values of $LC_{50}$ and $LC_{90}$ equal to 1.8083 and 8.3227 respectively. The application of the oil extract against the larvae resulted in the prolongation of both larval and pupal duration and the decrease of the growth rate, pupation, adult longevity and fecundity. Also, the corrected weight of consumed leaf, consumption index (IC), relative growth rate (RGR), the efficacy of ingested and digested food conversion to body tissue (ECI) and (ECD), respectively and the approximate digestibility (AD) were decreased after the application of the oil extract on the larvae. As a result of the current study, the oil extract from the husk of *Coffea arabica* revealed its efficacy as both larvicide and deterrent against the 4$^{th}$ instar larvae of cotton leafworm *S. littoralis*.

## INTRODUCTION

The cotton leafworm *Spodoptera littoralis* (Boisdural) [Lepidoptera: Noctuidae] is a serious economic pest in Egypt (Abdel-Aziz *et al*., 2013; Ghoneim *et al*., 2020). It is a polyphagous species that feed on a wide range of plant hosts (Ben-Khalifa *et al*., 2018). It feeds on cotton and on over 40 plant families so it can attack several economically important crops all year round (Bakr., 2015). In Egypt, it causes yield losses in many economic crops in recent decades (El Sayed *et al*., 2022). It has been shown to lower cotton

yields by as much as 75 % (Nasr *et al*., 1984; Campion *et al*., 1997). Until now, in Egypt and other countries, chemicals are still the major efficient way the control *S. littoralis* however this control method is not felicitous due to the high capacity of the insect to resist most of the traditional insecticides (Hilliou *et al*., 2021; Shawer *et al*., 2022). Consequently, there is a need to use new alternatives to control this pest and protect crops (Ismail *et al*., 2022).

Botanical biocides are environmentally friendly, biodegradable and a safe economic way to get rid of agricultural wastes, particularly in developing countries. Currently, it is a strategy replacing the chemical insecticides thus avoiding their hazards to the environment and surrounding living organisms (Ashamo *et al*., 2021; Farag *et al*., 2021; Hussein *et al*., 2022).

Several studies dealt with the use of insecticides derived from agro- wastes against insect pests such as the works by Abaza (2020), Ashamo *et al*. (2021), Farag *et al*. (2021a) and Farag *et al*. (2021b) and produced encouraging results if compared with the application of chemical insecticides.

Arabica coffee (*Coffea arabica*) is among the most widely distributed types of beans that are used as raw materials for coffee drinks, and are consumed everywhere around the world. Antioxidant and anti-microbial activity of *Coffea arabica* leaves and essential oil extract from coffee husk has been previously investigated (Alamin, 2019; Kiattisin *et al*., 2019; Al-Yousef and Amina 2018). Insecticidal activity of purified protein from coffee seeds, flowers, leaves, coffee grounds and spent coffee ground extract of *Coffee arabica* were previously evaluated (Coelho *et al*., 2010; Stashenko *et al*., 2013; Nakano, 2019; Aditama *et al*., 2019; Hussein *et al*., 2022). Coffee husk is an organic waste material that's resulted at the milling stage of coffee production. Therefore, the aims of this study were to analyze biochemical constituents of essential oil extract from seed husks of *Coffea Arabica* and test its toxicity and bioactivities against the cotton leaf worm *Spodoptera littoralis* in a laboratory.

## MATERIALS AND METHODS

**Essential Oil Extraction:**

Husks of *C. arabica* seeds were dried in the air and then exposed to steam distillation for 3-4 hours to extract the essential oil. The collected oil was kept until analysis.

**Gas Chromatography-Mass Spectroscopy GC-MS:**

GC-MS analysis was done using a Shimadzu GC-MS-QP 2015 plus (Kyoto, Japan).

**Tested Species:**

A colony of the cotton leafworm *Spodoptera littoralis* was maintained in the laboratory for many generations at suitable conditions of temperature and relative humidity (27±2C and 60 ±5%) and was reared on castor leaves (El-Defrawi *et al*., 1964) for obtaining a synchronized culture.

**Treatment and Bioassay:**

Early fourth instar larvae were used to evaluate their susceptibility to oil extract by using a feeding technique.

Groups of 10 gms castor leaf discs (5 cm in diameter), previously immersed in acetone concentrations of oils and left to dry, were distributed in containers with 20 larvae. The larvae were allowed to feed on treated discs for 48 hrs and then transferred to untreated leaves until they either died or completed their development. The mortality was recorded 24 hrs later. The corrected mortalities drawn against logarithms of concentrations and LCP

lines were fitted (Finney, 1971) and the desired lethal concentrations were determined graphically to be used in the biological and physiological tests. The control tests were carried out as mentioned above without oil treatment.

**Physiological Tests:**

Nutritional indices and their contributions were measured according to Waldbauer (1968) as follows:

1. The corrected weight (wt) of leaf consumed= (I-a/ 2) [W- (L+ Bl)] where a= ratio of loss of water to the initial weight of the aliquot, b the ratio of water to the final weight of the aliquot, W= weight of food introduced, L= weight of untreated food.
2- Consumption index (CI) is a measure of food eaten per unit of time relative to the mean weight of larvae during the feeding period, CI=C/ (T x A) Where C= fresh weight of leaf consumed, T= duration of feeding period and A= mean fresh weight of larva during the feeding period.
3- Relative growth rate (RGR) measures the amount of weight gained per unit time relative to the mean weight of the larva during the feeding period, RGR= G/(TxA) where G= fresh weight gain of the larva.
4. Efficiency of the conversion of the ingested food to body tissue (ECI) is an overall measure of the larva's ability to consume ingested food for growth, ECI= (G/C) (100%)
5- Efficiency of the transformation of the digested food to body tissue (ECD) is an overall measure of the larva's ability to utilize digested food for growth, ECD= (G/C-F) (100%) Where f= faces weight during the feeding period.
6. Approximate digestibility (AD) is the degree of larva's ability to digest the introduced food AD= (C.F/C) (100%).

## RESULTS

**GC-MS Analysis:**

The biochemical analysis of the essential oil extract from *Coffea arabica* L. husks GC-MS analysis yielded 13 compounds which are listed in Table (1). The resulted compounds were specified through a comparison of the recorded mass spectra with those preserved in the Wiley library. The main constituents of the essential oil extract were fatty acids and their derivatives (76.6 %). Among the separated acids the oleic acid represented the large proportion equal to 65 %. Besides, acids the second major constituent is 1,3,5-Cycloheptatriene,3,7,7-trimethyl- a monocyclic monoterpenoid and represented by 15.57 % and the other remaining compounds (nine) constituting the ratio of 7.83 %.

**Toxicity Test:**

The results indicated that the coffee husk oil extract caused the mortality of 91.66 % of larvae at a concentration equal to 8 ppm as shown in Table (2). Based on the $LC_{50}$ and $LC_{90}$ values of the tested extract, the results revealed that the coffee husk oil extract has larvicidal activities against the $4^{th}$ instar larvae of *S. littoralis*.

**Bioactivities of the Oil Extract against *S. littoralis*:**

Data from Table (3) revealed that the application of the oil extract on the $4^{th}$ instar larvae of *S. littoralis* remarkably decreased the food consumption, the conversion of ingested and/or digested food into body tissue and delayed the growth rate if compared with the control experiments.

The effect of the $LC_{50}$ concentration of the oil extract on larval duration, pupal duration and adult longevity, fecundity, egg hatchability and deterrent effect of the oil on *S. littoralis* were given in Table (4). The results indicated the prolongation of the larval and pupal duration and the decreasing of the adult longevity, fecundity, hatchability and deterrent index.

**Table 1**. Chemical composition of the *Coffea Arabica* oil extract. M.F. (molecular formula); M.W. (Molecular weight); RT (retention time).

| | Phytochemical compound | M.F. | M.W. g/mol | %Area | R.T. (min) |
|---|---|---|---|---|---|
| 1 | 1,3,5-Cycloheptatriene,3,7,7-trimethyl- | $C_{10}H_{14}$ | 134.22 | 15.5765 | 8.683 |
| 2 | Benzene, 1-methyl-3-(1-methylethyl)- | $C_{10}H_{14}$ | 134.2182 | 0.2819 | 10.181 |
| 3 | Cyclononasiloxane, octadecamethyl- | $C_{18}H_{54}O_9Si_9$ | 667.3855 | 0.1002 | 12.6523 |
| 4 | 3-Oxatricyclo[4.2.0.0(2,4)]octan-7-one | $C_7H_8O_2$ | 124.14 | 0.0727 | 13.6723 |
| 5 | 1H-Indole, 5-fluoro- | $C_8H_6FN$ | 135.1383 | 1.7136 | 14.3659 |
| 6 | Benzamide,3-methoxy-N-(3-methoxybenzoyl)-N-3-methylbutyl- | $C_{21}H_{25}NO_4$ | 355.4 | 0.2758 | 14.6107 |
| 7 | n-Hexadecanoic acid | $C_{16}H_{32}O_2$ | 256.4241 | 9.5021 | 14.9488 |
| 8 | Oleic Acid | $C_{18}H_{34}O_2$ | 282.4614 | 65.4579 | 15.6191 |
| 9 | 9,12-Octadecadienoic acid (Z,Z)- | $C_{18}H_{32}O_2$ | 280.4455 | 2.1435 | 16.983 |
| 10 | Z-11-Tetradecen-1-ol monofluoroacetate | $C_{16}H_{29}FO_2$ | 272.40 | 0.494 | 18.3935 |
| 11 | N-Isobutyl-11-(3,4-methylenedioxyphenyl)-2E,4E,10E-undecatrienoic amide | $C_{22}H_{29}NO_3$ | 355.4706 | 0.5342 | 19.0055 |
| 12 | 1H-indole, 3,3'-[1,4-phenylenedi-2,1-ethenediyl]bis[1,2-dimethyl- | $C_{30}H_{28}N_2$ | 416.6 | 1.8243 | 20.2937 |
| 13 | 4-Estren-3,17-dione, di-trimethylsilyl | $C_{24}H_{40}O_2Si_2$ | 416.7 | 1.9034 | 20.3811 |

**Table 2.** Toxicity of *Coffea Arabica* husk oil extract on 4th instar larvae of *Spodoptera littoralis* after 48hrs

| Conc.(ppm) | r1 | r2 | r3 | total | % |
|---|---|---|---|---|---|
| **0.5** | 4 | 3 | 4 | 11/60 | 18.33 |
| **1** | 5 | 5 | 5 | 15/60 | 25 |
| **2** | 10 | 11 | 11 | 32/60 | 53.33 |
| **4** | 14 | 15 | 15 | 44/60 | 73.33 |
| **8** | 19 | 18 | 18 | 55/60 | 91.66 |
| **$LC_{50}$ (ppm) (co. limit)** | 1.8083 (1.5549 - 2.0962) | | | | |
| **$LC_{90}$ (ppm) (co. limit)** | 8.3227 (6.4874 - 11.591) | | | | |
| **Slope** | 1.9330 +/- 0.1626 | | | | |

**Table 3:** Nutritional indices and their related parameters for *Spodoptera littoralis* larvae after feeding on castor leaves treated with essential oils

| Items | Treated | Untreated |
|---|---|---|
| Cumulative food consumption C (mg/larva) | 1029** | 2988 |
| Consumption index (CI) | 0.51* | 0.65 |
| Relative growth rates (RGR's) | 0.0881 | 0.1000 |
| Efficiency of ingested of conversion food to body tissue (ECI %) | 17.28 | 13.92 |
| Efficiency of digested conversion food to body tissue (ECD %) | 24.39** | 15.48 |
| Approximate digestibility (AD %) | 70.85** | 89.96 |

*= significant $p<0.05$
**= highly significant $p<0.01$
***= very highly significant $p<0.001$`

**Table 4:** Effect of essential oils on some biological activities of *Spodoptera littoralis*

| Item | Treated | Untreated |
|---|---|---|
| larval duration (in days) mean±SE | 28.36±0.35** | 19.33±0.58 |
| Pupation (%) Mean ±SE | 73.50±0.38** | 95.30±233 |
| Pupal period (in days) mean±SE | 14.74±0.32* | 11.02±0.12 |
| Adult emergence (%) Mean±SE | 55.00±0.23** | 93.51±1.04 |
| Fecundity (egg/o) mean±SE | 528.70±20.70** | 1135.25±101.39 |
| Deterrent index | 36.45 | 0 |
| Hatchability (%) Mean±SE | 51.17±0.44** | 95.73±2.12 |
| Adult longevity (in days) | | |
| Male (Mean±SE) | 10.22±1.40 | 11.15±0.61 |
| Female (mean±SE) | 12.45±1.00 | 14.33±1.06 |

*= significant $p<0.05$
**= highly significant $p<0.01$
***= very highly significant $p<0.001$`

## DISCUSSION

The chemical constituents (13 compounds) of coffee husk oil extract were specified using gas chromatography (GC) and the mass spectrometer (MS). The major compound resulting from the analysis of the extract is oleic acid with a peak area equal to 65 % this may be the major component that resulted in toxicity. Oleic acid was recorded as a constituent of many plant extracts which as a whole proved to have an insecticidal activity (Sini *et al*. 2005; Farag *et al*., 2011; Farag *et al.*, 2021b). Also, it was previously reported as a successful larvicide against *Pectinophora gossypiella* (Saunders) (Lepidoptera: Gelechiidae) (Yousef and Moustafa, 2013). The second constituent is the monoterpenoid 1,3,5-Cycloheptatriene,3,7,7-trimethyl- which is one of the main components of many essential oils and plant extracts (Romanenko and Tkachev, 2006). This compound was previously isolated from the female mountain pine beetles, *Dendroctonus ponderosae* Hopkins (Coleoptera: Scolytidae) and identified as a female-produced pheromone (Gries *et al*., 1992). Of the resulted compounds, n-Hexadecanoic acid with a peak area (9.5 %) is the only one that shared with the results of the previous analysis of coffee extracts and isolated from the husk in the study by Al Yousef and Amina (2018) and exhibited both antioxidant and antimicrobial activities. It also showed toxicity against mosquito larvae (Ravindran *et al*., 2020). The next constituent; 9,12-Octadecadienoic acid (Z,Z)- a polyunsaturated fatty acid with the area (2.1 %) showed insecticidal activity (Christiana *et al*., 2019) and many other biological activities such as anti-inflammatory, anticancer and nematicide (Adeyemi *et al*., 2017) and is also a constituent of plant waste extract which had insecticidal activity (Farag *et al* . 2021b). the remaining nine constituents represented as all 8.2% of the oil extract from which the two compounds: Cyclononasiloxane, octadecamethyl- and N-Isobutyl-11-(3,4-methylenedioxyphenyl)-2E,4E,10E-undecatrienoic amide have insecticidal activities (Ojekale *et al*., 2013; Miyakado and Nakayama, 1981; Scott *et al*. 2007), the 3-Oxatricyclo[4.2.0.0(2,4)]octan-7-one has an antioxidant and anti-inflammatory effect (Oladele, *et al*. 2022) and the Benzene, 1-methyl-3-(1-methylethyl)- exhibit antimicrobial, anti-cancer, antioxidant, and anti-inflammatory activities (Marchese *et al*., 2017; Kiki and Ibrahim, 2020).

The insecticidal toxicity of the coffee oil extract is not only due to its main constituents mentioned above, the antagonistic or the synergistic effect of a single compound in the mixture should be taken into account. Thus the activity may also be owing to certain minority components or to the synergistic effect of many constituents (Ravi *et*

*al*., 2018; Farag, *et al*. 2021b).

Concerning the toxic effect, the insecticidal activities of coffee husk oil extract against larvae of *S. littoralis* might be due to 1. The failure of the larvae to molt because of the inhibition of chitin synthesis (Abdel Rahman *et al*., 2007; Adel, 2012), 2. the molting larvae are unable to absorb large amounts of air to aid in the division of the old cuticle and therefore the expanding of the new during the molting process (Linton *et al*., 1997) and the larvae may die due to cessation of feeding and continuing starvation (Ghoneim *et al*., 2012, Ghoneim *et al*. 2020).

The pupal mortality in *S. littoralis*, might be due to the effect of the oil extract on some vital processes including suffocation, hemorrhage and dehydration due to the incomplete protrusion and the failure of vital homeostatic mechanisms (Smagghe and Degheele, 1997).

For adults, it is noteworthy that plant extracts inhibit the maturation of the imaginal discs, which are precursors of several adult integumentary structures in holometabolous insects. This can interpret the decrease in the proportion of adult emergence (Schneidermann, 1972; Suh *et al*., 2000; Farag *et al*., 2021a). Also, the adult death might be interpreted by the retention and distribution of the oil extract in the insect's body due to the fast transfer through the haemolymph to the other body tissues, and the inability of the adult to detoxify the tested extract (Osman *et al*., 1984), the distortion of adult chitin (Abo El-Mahasen *et al*., 2010), and/ or the failure of adults to exit from the pupal exuvia owing to the unsaturated fatty acids that promoted the melanization and hardening of the larvae; as a consequence, adults were failed to free themselves from the pupal exuvia, unsatisfactory pressure in the frontal suture, and the strengthening of opercular suture (Hussien, 1995, Farag, *et al*. 2021a).

The application of the oil extract on *S. littoralis* resulted in the elongation of the larval and pupal durations in contrast to the growth rate which has been decreased. These results were previously recorded after the treatment of several insect pests with insecticides of plant origin (Awad, 2001; Abdel-Rahim *et al*., 2007; Bhatnagar *et al.*, 2012; Awad *et al*., 2013; Szczepanik *et al*., 2016; Ghoneim, *et al*. 2020).

The steps of insect development are affected by three hormones: Prothoracicotropic hormone (PTTH), Ecdysone and Juvenile hormone which are secreted by neuro-secretory cells (NSC) present in the brain, Prothoracic gland (JH) and corpora allata respectively. The insect sent signals to the brain and directed it to trigger the neurosecretory cells which subsequently release PTTH that then move down to the neurohemal organ, Corpora Cardiaca (CC) to emit the stored PTTH to the hemolymph. Then, the prothoracic glands are triggered by this to release Ecdysone which triggers a group of physiological processes and finally forms the new cuticle (Kaleka *et al*. 2019). In the current study, the extension of the larval and pupal periods and delayed development of *S. littoralis* might be due to the interference of the oil extract and stopping these processes at any stage. It might incidentally interfere with organs responsible for the production and releasing of PTTH (Subrahmanyam *et al*., 1989; Ben Hamouda *et al*., 2015) or stop the pathway for chitin biosynthesis and delay molting (Djeghader *et al*., 2014). Also, the increased duration of larvae could be owing to reduced food intake and reduced biomass and lead to the extension of the period needed to complete the development (Giongo *et al*. ،2015; Ghoneim, *et al*. 2020).

The fecundity and hatchability were decreased after the application of the oil extracts. The reduction in the number of laid and hatched eggs might be due to the failure of sperms to transfer to the females during the copulation or owing to the partial sterilization of one of or both sexes (Ismail, 1980). Also, the treatment of *S. littoralis* with the tested oil extract might disrupt the protein synthesis of the ovarioles and consequently

inhibit ovariole maturation (Saini *et al*. 2010). Other previous studies indicated that the modification in follicular epithelium as well as deformation of vitellogenic oocytes which resulted after the application of insecticides might disrupt the oogenesis and caused chronic inhibition and reduction of fecundity (Bakr *et al*., 2010; Ahmed *et al*., 2015).

Concerning the reduction of food consumption (CI) by *S. littoralis* after the treatment with the tested oil extract this indicates that the extract act as an antifeedant or deterrent (Isman, 2002). The ECI index reflects the ability of the insect to combine the ingested food into biomass (Nathan *et al*., 2005). The lowest values of both ECI and ECD values of *S. littoralis* larvae resulted from the decreased efficacy of converting the ingested food into growth. The deficiency in ECI is related to energy-consuming physiological activities, the development and the molting process (Carne, 1966). The low ECD value might be owing to the use of energy for the renovation of the damage in the midgut epithelium (Luthy and Wolfersbrenger, 2000). The decreased RGR might be due to the reduction of the conversion efficiency of ingested and digested food (Dahlman, 1977) or the irreclaimable deterioration of the midgut cells (Jansen and Groot, 2004). Lower RGR, ECI, and ECD possibly resulted in delayed larval and pupal developments. The reduction of digestion caused due to the covalent binding of the compounds with food proteins or digestive enzymes (AbdelRahman and Al-Mozini, 2007).

The decreased AD values in the larvae indicated that the food was not being retained for a long time in the gut of the larvae and this might be the reason for the disturbance of the metabolic rate (Khedr *et al*., 2015). However, the increased deterrent index might be owing to the fast deterrence triggered by the chemical sensila on the mouth parts of larvae or contracted impulses from the stomodael nervous system after food ingestion (Sadek, 2003), furthermore, due to the presence of certain chemical compounds (Salama and Sharaby, 1988) or owing to the toxic effects after the ingestion (Khedr *et al*., 2015). These antifeedants inhibit the feeding but do not kill the larvae immediately, however partly limit their growth and development. Accordingly, the elongated duration of the larvae and pupae and the decreased pupal weight and formation might be due to the decrease in the values of CI, ECI, ECD, RGR and AD (Khedr *et al*., 2015).

The results obtained in this study agreed with those by Khosravi *et al*. (2010) and Khedr *et al*. (2015) who mentioned that deterrence delays the insect growth, prolongs its seeking for food and therefore increases the probability of its death in addition to the decreased ECD, ECI and RGR might cause a retardation in the growth of larvae resulting in the production of smaller pupa which poses a direct effect on the longevity and fecundity adults.

## Conclusion

The insecticidal and deterrent activity of the oil extract from the husk of Arabic coffee against the 4$^{th}$ instar larvae of *S. littoralis* pointed to the possibility of using it as a natural and safe source of insecticides. Its efficacy was confirmed through the values of $LC_{50}$ and $LC_{90}$ in addition to its effect on insect bioactivities. The GC-MS analysis of the extracted oil revealed the occurrence of several biologically active compounds that might be more effective to control cotton leaf worm instead of using chemical insecticides to contribute to decreasing the number of insecticides used and consequently minimize its dangers on human, non-target species and environment as well as the sustainable utilizing of agro-wastes. However, further research is needed to test the effectiveness of this compound in the field.

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