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ORIGINAL RESEARCH ARTICLE

Insecticidal, residual and sub-lethal effects of some plant essential oils on *Callosobruchus analis* (F.) infesting stored legumes

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Abstract

Stored legumes suffer both quality and quantity deterioration due to infestation by bruchids. Recently, plant essential oils (EOs) are recognized as safer substitutes to manage these pests by alleviating the concerns of residue and resistance problem of synthetic grain protectants. Insecticidal, sub-lethal and residual effects of *Pogostemon cablin* Benth, *Mentha arvensis* L., *Cymbopogon martinii* (Roxb.) Wats., *Pelargonium graveolens* L. and *Acorus calamus* L. EOs were investigated against *Callosobruchus analis* (F.) (Coleoptera: Chrysomelidae). In contact toxicity, LC_{50} values ranged from 0.040 to 0.362 µl/cm^2, being lowest for *P. cablin*. *Cymbopogon martinii*, *P. graveolens* and *A. calamus* EOs had strong repellent property (> 83%), while *M. arvensis* and *P. cablin* demonstrated moderate repellency (41–67%) at sub-lethal concentrations. Sub-lethal exposure reduced the oviposition (5.96–100%) and inhibited progeny emergence (21.22–100%) in dose-dependent manner. *Acorus calamus* EO showed potent oviposition deterrency and progeny emergence was totally abolished. EOs exhibited moderate to high residual activity, where *M. arvensis* and *A. calamus* treated seeds were completely protected (0% damage) for 70- and 84-days post-treatment. Results indicated the promising potential of five EOs to be used as bioactive ingredients for developing grain protectants to prevent post-harvest deterioration of legumes.

Keywords Bruchids · Contact toxicity · Essential oils · Residual toxicity · Sub-lethal effects · Stored legumes

Introduction

Grain legumes are important components of farming system and affordable source of dietary protein and minerals, contributing nearly 33 per cent of the dietary protein nitrogen needs in human nutrition across the globe (Vance et al. 2000). The stored legumes in the tropical and subtropical regions of world are often infested by bruchid species (Coleoptera: Chrysomelidae) (Southgate 1979; Mishra et al. 2017). Adult bruchids deposit eggs on legume seeds and larval stages are internal feeders, and finally reproductively mature adults emerge from seeds which do not require either food or water to reproduce (Credland 1987). Some bruchid species can infest the crop in field but economic loss is usually manifested at post-harvest stages. Bruchids multiply exponentially in stored legumes and cause complete loss of produce in about 6–8 months of storage (Caswell 1961; Singh et al. 1978; Dwivedi et al. 2020; Mannava et al. 2022). Losses arise from larval feeding activity that often lead to mouldiness and, loss of nutritional and commercial value of stored seeds (Caswell 1968; Ojimelukwe and Ogwumike 1999).

Over the years, synthetic insecticides and fumigants are consistently used for the disinfestation of stored grains and products during post-harvest storage. Besides, the undesirable residues in stored products (Phillips and Throne 2010), the resistance to synthetic insecticides is known to be present in at least 11 species of stored-product insects from 45 countries (Champ and Dyte 1976; Chaudhry 2000). Over reliance on key fumigant phosphine across the globe further aggravated the resistance problem (Nayak et al. 2020). This development has made the control of stored grain pests more challenging and, thus necessitated the pursuit for organic and environmentally benign alternate grain protectants against these pests. In past few years, plant essential oils (EOs) are regarded as safer and potential bioactive compounds against several stored-product pests (Regnault-Roger et al. 2012; Pavela

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and Benelli 2016). They are blends of volatile secondary metabolites, hence reported to exhibit broad spectrum activities, including insecticidal, repellent, oviposition deterrent (Shaaya et al. 1997; Kim et al. 2010), regulating growth, behaviour (Papachristos and Stamopoulos 2002a; Isman et al. 2007) and reproduction in insects (Regnault-Roger and Hamraoui 1994). EOs act at multiple and novel target sites in insects (Kostyukovsky et al. 2002; Priestley et al. 2003).

Several studies confirmed the potential bioactivity of EOs against major stored-product pests belonging to Chrysomelidae (Kim et al. 2003), Curculionidae (Tapondjou et al. 2005), Tenebrionidae (Teke and Mutlu 2021), Bostrichidae (Tripathi et al. 2003), Silvanidae (Ogendo et al. 2008), Dermestidae (Nenaah 2014a, b), Pyralidae (Tunc et al. 2000) and others. EOs offer several advantages over synthetic chemical grain protectants of being natural biocides, biodegradable, derived from renewable sources and minimal or low-risk to mammalians and environment (Rajendran and Sriranjini 2008; Regnault-Roger et al. 2012). The sub-lethal doses of insecticides are reported to elicit either detrimental effect or alterations in certain life traits (fertility, oviposition, development, longevity, etc.) of insects (Desneux et al. 2007). Biological impairments like oviposition and growth reduction are reported in female bruchids treated sub-lethally with clove (*Syzygium aromaticum* L.: Myrtaceae) and cinnamon (*Cinnamomum zeylanicum* L.: Lauraceae) EOs (Viteri Jumbo et al. 2018). Physiological or behavioural responses in sub-lethally exposed insects to botanical insecticides can also affect the efficacy of these compounds.

Among the bruchid species, *Callosobruchus analis* (F.) is predominantly oriental bruchid and true storage species capable of infesting several times to produce successive generations (Sengupta et al. 1984) in tropical Asia and Africa (Tuda et al. 2005). In India, this species demonstrated wider distribution (Revanasidda 2022) and found extremely destructive to stored food legumes (Soumia et al. 2015; Dwivedi et al. 2020) including wild *Vigna* species (Fabaceae: Fabales) (Aidbhavi et al. 2021). However, EOs were not extensively studied against this species. Hence, the present study aimed to investigate the insecticidal, sub-lethal and residual effects of essential oils of menthol-mint: *Mentha arvensis* L. (Lamiaceae), palmarosa: *Cymbopogon martinii* (Roxb.) Wats. (Poaceae), geranium: *Pelargonium graveolens* L. (Geraniaceae), patchouli: *Pogostemon cablin* Benth (Lamiaceae) and sweet flag: *Acorus calamus* L. (Acoraceae) on bruchid species, *C. analis* (Coleoptera: Bruchinae) in respect of direct contact toxicity, repellency, oviposition deterrence, inhibition to progeny emergence and persistence in order to contribute for the development of control strategies against this destructive pest.

Materials and methods

Test insect

The test insect, *C. analis* was reared in a controlled conditions (27 ± 1°C, 65 ± 3% RH and 12 h photoperiod) in Storage Entomology Laboratory, ICAR-IIPR, Kanpur (India) following the rearing protocol (Strong et al. 1968). The test insect culture was previously maintained (for 5 years) in the laboratory for several generations without exposure to any insecticides. A single mating pair of beetles was introduced on healthy and sterilized mungbean seeds in a sterile plastic rearing container (8 cm ht. × 11 cm dia.). The ensuing F1 adults (1–3 d) were sub-cultured to ensure continuous availability of uniformly aged population of insects for the experiments. In sub-culturing, the parent stocks were allowed to lay the eggs for 24 h and removed thereafter, and seeds bearing eggs were incubated until the emergence of adult beetles. The male and female beetles of *C. analis* were distinguished by their morphological features (Southgate et al. 1957; Southgate 1958). All the tests were conducted at above mentioned controlled laboratory conditions and employed 1–3 day old adult beetles.

Extraction of essential oils

The volatile fractions (essential oils) of plant species were obtained by hydro-distillation process using 'modified Clevenger Apparatus'. The fresh aerial portion of *M. arvensis* and *C. martinii* was used for extraction. The oil yield (v/w) of *M. arvensis* and *C. martinii* was 0.75 and 0.54%. The essential oils of *P. graveolens* and *P. cablin* were procured from CSIR-CIMAP, Pantnager (India). *A. calamus* EO was supplied by Aarnav Global Exports (India). The essential oils were preserved in amber-coloured airtight containers at 4°C for subsequent toxicity assays.

Contact toxicity assay

The contact toxicity of essential oils was determined by "Filter Paper Impregnation" method (Tapondjou et al. 2005) with slight modifications. According to the results of preliminary-assay, 5–6 concentrations of EOs were used to compute the lethal toxicity (see Table 1). Aliquots of test EOs were dissolved in acetone (100 µl) and applied uniformly to the Whatman No. 1 filter paper disc (4.60 cm diameter and 16.62 cm^2 surface area). Controls received acetone (100 µl) only. The acetone was allowed to evaporate at room temperature for 5 min and each paper was placed at the bottom of Petri dish (5 cm dia. × 1.5 cm ht.). The unsexed adult test insects (n = 20) were introduced in each Petri dishes and covered with a lid.

Table 1 Contact toxicity (LC_{50}, LC_{90} and TR values) of different essential oils to *C. analis* adults at 24 h exposure

Essential oils	Conc. range* (µl/cm^2)	LC_{50} (95% FL) µl/cm^2	LC_{90} (95% FL) µl/cm^2	Slope ± SE	*df*	*p* value	TR[a]
Pelargonium graveolens	0.241–0.481	0.362 (0.329–0.400)	0.553 (0.478–0.758)	6.96 ± 1.43	3	0.817	-
Cymbopogon martinii	0.241–0.361	0.304 (0.281–0.334)	0.437 (0.379–0.668)	8.22 ± 2.19	3	0.763	1.19
Mentha arvensis	0.132–0.223	0.168 (0.147–0.185)	0.283 (0.234–0.516)	5.63 ± 1.57	4	0.780	2.15
Acorus calamus	0.030–0.271	0.142 (0.086–0.276)	1.383 (0.519–63.48)	1.30 ± 0.40	3	0.532	2.55
Pogostemon cablin	0.024–0.072	0.040 (0.033–0.046)	0.078 (0.064–0.116)	4.46 ± 0.89	3	0.952	9.05

* Concentration range based on preliminary range finding assay

[a] TR: Toxicity Ratio (EO that exhibit the major LC_{50}/LC_{50} of other EOs)

All the treatments including controls were replicated three times. The treated insects were held under controlled conditions in the laboratory and mortality was recorded at 24, 48 and 72 h post-exposure. The adult beetles were considered dead if the appendages did not move when prodded with camel-hair brush. Lethal concentration values were computed following 24 h exposure to test EOs. Toxicity ratios (TR) were obtained by the quotient between the LC_{50} of the least toxic EO and the LC_{50} of the remaining test EOs, individually.

Repellency assay

The repellent action of test EOs was determined by "Area Preference" method following McDonald's Standard Method Number- 3 (McDonald et al. 1970) with some modifications (Fig. 1). The test arena consisted of Whatman No. 1 filter paper disc (7 cm diameter) cut into two semi-circular portions. The repellent activity of test EO's was determined at three sub-lethal concentrations, equivalent to LC_{50}, LC_{20} and LC_{10}, based on the contact toxicity test results. The test EOs were prepared in acetone and applied to semi-circular filter paper disc uniformly to obtain desired concentrations. The other half of the filter paper received acetone only and served as a control. Each treated filter paper was air dried to evaporate the solvent completely. The treated half filter paper disc was re-attached to untreated half disc lengthwise to form a full circular disc using cellulose tape with a minute gap between the filter paper halves to prevent the seepage of test EO from one halve of the circle to another. The full circle of filter paper was placed in the bottom of Petri dish with seams oriented in opposite directions to exclude the effect of external stimulus, if any on the dispersal of insects in the test arena. The unsexed test insects (n = 20) were released at the centre of the test arena and Petri dish was covered. The number of test insects settled on treated and untreated halves were counted at hourly intervals up to 5 h and average counts were expressed in terms of percent repellency (PR). Positive values indicate repellency while negative values exhibit attractant properties.

The Repellency Index (RI) was computed by adopting the formula of food preference index cited by Lin et al. (1990).

$$RI = 2G/(G + P).$$

where G = % of test insects attracted to treated arena and P = % of insects attracted to the control arena. RI values varies between 0 and 2, where RI = 1 indicates the neutral effect, RI > 1 indicates attractant effect and RI < 1 indicates the repellent effect of EO on the test insects.

Oviposition deterrence assay

The oviposition deterrence property of EOs was tested at three sub-lethal concentrations, equivalent to 1/5th, 1/10th and 1/20th of the LC_{90} fraction based on contact toxicity assay. Appropriate quantity of test EOs were dissolved in

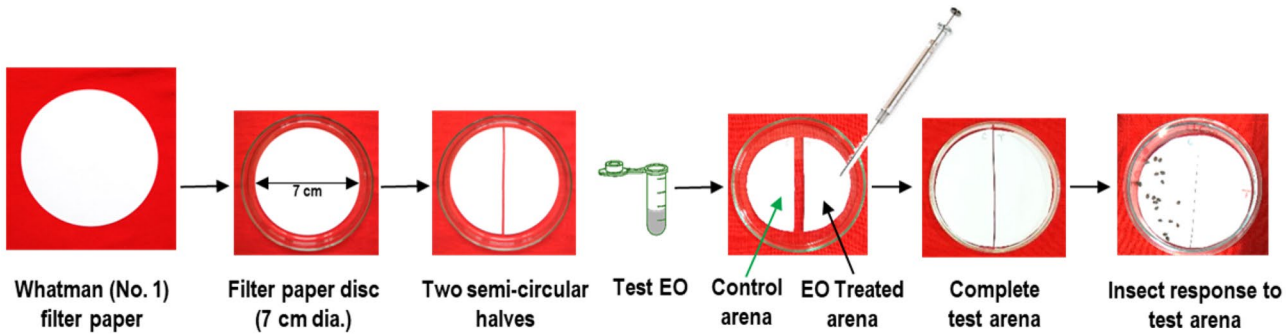

Fig. 1 Experimental set-up of repellency assay

acetone to get desired concentrations and admixed with sterilized mungbean seeds (5 g), and treated seeds were stirred manually to ensure proper mixing. Seeds in the untreated controls received acetone only. Seeds were allowed to air dry for complete evaporation of the solvent and then placed in the Petri dish. All the treatments including controls were replicated three times. Afterwards, three pairs of adult insects (1–3 d old) were released on the treated seeds and incubated. The beetles were allowed to lay the eggs on the treated seeds for 72 h, thereafter, the insects were discarded, and egg-laden seeds were incubated for adult development. The efficacy of EO was determined in terms of its ability to deter the bruchids from oviposition on the treated seeds compared to controls and expressed as per cent reduction in oviposition (PRO) as described by Elhag (2000) and reduction in adult emergence as per cent inhibition rate (PIR) as described by Tapondjou et al. (2002).

$$\mathrm{PRO} = [(N_C - N_T)/N_C] \times 100,$$

where N_C = number of eggs deposited on the control seeds and N_T = number of eggs deposited on the treated seeds.

$$\mathrm{PIR} = [(Cn - Tn)/Cn] \times 100,$$

where Cn = number of F1 adults emerged from untreated seeds and Tn = number of F1 adults emerged from treated seeds.

Persistence of biological activity

To assess the residual activity, EOs were admixed with seeds and offered to test insects after varied periods of storage as described here. Appropriate quantity of each EO at a concentration equivalent to LC_{90} fraction derived in contact toxicity assay was diluted in acetone and applied to sterilized mungbean seeds (200 g) uniformly. In control, seeds were treated with acetone only. After complete evaporation of solvent, treated seeds were stored in amber coloured glass container (0.5 L) wrapped with aluminium foil at controlled conditions for three months. The seed samples (5 g) from control and treated lots were withdrawn at every 14 days intervals and exposed to bruchid infestation at three pairs in a Petri dish. The experiment was replicated thrice. Exposure of test insects to treated and control seeds continued for 72 h, thereafter, the insects were discarded, and seeds were incubated for adult development. The insecticidal activity of EOs *vis-à-vis* time was ascertained in terms of adult mortality, oviposition, progeny emergence and seed damage.

Statistical analysis

Mortality data was corrected for natural mortality in the controls, if any, using Abbott's formula (Abbott 1925) and expressed as percentages. Bioassay data was subjected to Probit analysis (Finney 1971) to compute lethal concentration (LC) values and toxicity was expressed as µl of essential oil per cm^2 of treated area. Means (± SE) of adults (%) attracted to test EO and control as well as oviposition and F1 adult emergence from treated and untreated seeds are reported. Mean oviposition, adult emergence and number of adults attracted in each of the treatments and control were compared by *t*-test ($\alpha = 0.05$). The mean data of persistence assay was subjected to appropriate transformation methods to perform ANOVA and means were compared using Tukey's HSD *post hoc* test ($\alpha = 0.05$). All the statistical analysis were performed using SPSS Statistics 16.0 program (SPSS Inc., Chicago, Ill., USA).

Results

Contact toxicity

Concentration-mortality assay indicated substantial toxicity of tested EOs against adult beetles. Mortality responses in different test EOs varied according to concentrations or exposure times (Fig. 2). At 72 h post-exposure, *A. calamus* EO exhibited very strong insecticidal activity and caused 100 ± 0% mortality at 0.030 µl/cm^2 or higher concentrations while, at 24 and 48 h after treatment (HAT), moderate and strong toxicity was noticed, causing 25 ± 2.89–73 ± 1.67 and 70 ± 2.89–100 ± 0% mortality. Although, mortality was proportional to increased concentrations, the insecticidal activity of *A. calamus* was more pronounced at higher exposure period. However, *P. cablin, M. arvensis, P. graveolens* and *C. martinii* EOs at higher concentrations (0.072 µl/cm^2, 0.0223 µl/cm^2, 0.481 µl/cm^2, and 0.361 µl/cm^2) demonstrated effective insecticidal activity by affecting 90 ± 5.77–97.50 ± 1.44, 85 ± 5.77–100 ± 0, 85 ± 2.89–92.50 ± 1.44, 75 ± 2.89–94 ± 3.63% mortality, respectively.

LC_{50} values for test EOs ranged from 0.040 to 0.362 µl/cm^2 (Table 1). Among the EOs tested, *P. cablin* recorded lowest LC_{50} value (0.040 µl/cm^2) and highest TR, demonstrating high contact toxicity to adult beetles. *Pelargonium graveolens* registered highest LC_{50} value (0.362 µl/cm^2), being least toxic. Concentration response curve of *C. martinii* and *P. graveolens* had steepest slope which demonstrated that smaller variations in EO concentrations induced greater responses in mortality of test insects. Toxicity ratios of *P. cablin, A. calamus, M. arvensis* and *C. martinii* were 9.05, 2.55, 2.15 and 1.19 times larger when compared to *P. graveolens*, thus, toxicity of EOs was decreased in the order as follows; *P. cablin* > *A. calamus* > *M. arvensis* > *C. martinii* > *P. graveolens.*

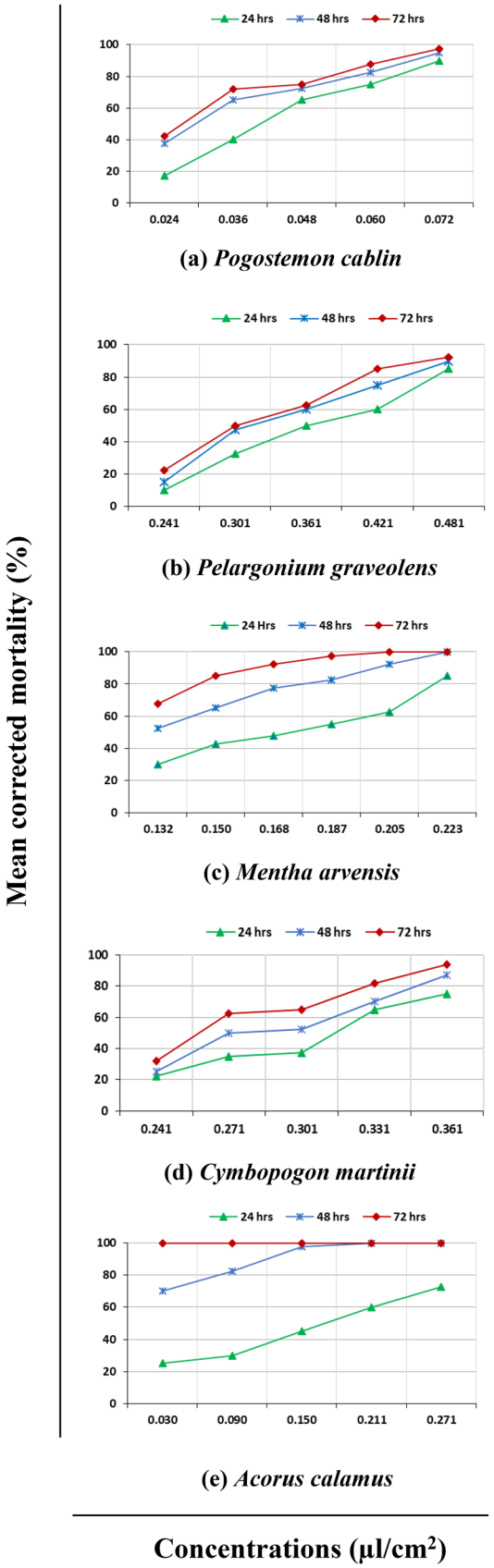

Fig. 2 Mean corrected mortality of adult beetles exposed to different ▶ concentrations of essential oils

Repellent activity

The results of repellency assays of essential oils against *C. analis* are presented in Table 2. Based on the repellency indices (RI), all the EOs had repellent activity (RI < 1.0) against *C. analis* adults at tested concentrations. The essential oils exhibited concentration-dependent repellent activity at all sub-lethal exposures. The percentage of test insects attracted to *M. arvensis* treated and control arena were variable and found significant at concentration equivalent to LC_{50}, LC_{20} and LC_{10}, and the repellency ranged from 53.54 to 67.50%. *Acorus calamus* EO exhibited strong repellent action (83.00–94.50%) and percentage of test insects on treated and control arena differed significantly at LC_{50}, LC_{20} and LC_{10}. *Cymbopogon martinii* demonstrated potent repellent action (over 95% repellency) on adult beetles and being significant for adults attracted to control and test arena at concentrations equal to LC_{50}, LC_{20} and LC_{10}. *Pogostemon cablin* exhibited moderate repellent activity (41– 63.50%) at sub-lethal concentrations and test insects attracted to control and treated arena differed significantly at LC_{50}, LC_{20} and LC_{10}. *Pelargonium graveolens* demonstrated very strong repellent activity by recording 92.67–93.67 per cent repellency in all three concentrations (LC_{50}, LC_{20} and LC_{10}) with significant variation in test insects attracted to treated and control arena (Supplementary Table 1).

Oviposition deterrent activity

All the essential oils exhibited variable deterrent activity at sub-lethal concentrations tested (Table 3 and Supplementary Table 2). Higher concentrations showed more deterrence to oviposition. Number of egg laid in *M. arvensis* treated and controls varied significantly at concentration equivalent to $1/5^{th}$ LC_{90} and registered 51.32 per cent reduction in oviposition. *Acorus calamus* exhibited over 97 per cent oviposition deterrence, being significant for number of eggs laid in treated and controls at $1/20^{th}$ LC_{90}, $1/10^{th}$ LC_{90} and $1/5^{th}$ LC_{90}. In *C. martinii*, oviposition differed significantly at $1/5^{th}$ LC_{90} and $1/10^{th}$ LC_{90} with 100 and 82.14 per cent reduction in oviposition. *Pogostemon cablin* exhibited moderate oviposition deterrence (46.22 and 37.46%) and being significant at $1/5^{th}$ LC_{90} and $1/10^{th}$ LC_{90}. In *P. graveolens*, the oviposition differed significantly at $1/5^{th}$ LC_{90} and $1/10^{th}$ LC_{90} for control and treated with 71.68 and 49.13 per cent reduction in oviposition.

All the essential oils variably inhibited the adult emergence at concentrations tested and per cent inhibition to F1 adult emergence was ranged from 21 to 100 per cent. Adult emergence differed significantly at $1/5^{th}$ LC_{90} in *M.*

Table 2 Repellent effect of essential oils on *C. analis* adults

Essential oil	Lethal Concentrations	Adults attracted (%) (Mean ± SE)		Per cent repellency	Repellency index (RI)	Classification
		Control	Treated			
Mentha arvensis	LC_{50}	83.75 ± 3.54	16.25 ± 3.54*	67.50	0.325	Repellent
	LC_{20}	79.00 ± 2.45	21.00 ± 2.45*	58.00	0.420	Repellent
	LC_{10}	77.00 ± 2.12	23.00 ± 2.12*	53.54	0.465	Repellent
Acorus calamus	LC_{50}	97.25 ± 1.18	2.75 ± 1.18*	94.50	0.055	Repellent
	LC_{20}	93.50 ± 1.17	6.50 ± 1.17*	87.00	0.130	Repellent
	LC_{10}	91.50 ± 0.87	8.50 ± 0.87*	83.00	0.170	Repellent
Cymbopogon martinii	LC_{50}	99.75 ± 0.25	0.25 ± 0.25*	99.50	0.005	Repellent
	LC_{20}	99.75 ± 0.25	0.25 ± 0.25*	99.50	0.005	Repellent
	LC_{10}	97.50 ± 1.44	2.50 ± 1.44*	95.00	0.050	Repellent
Pogostemon cablin	LC_{50}	81.75 ± 6.09	18.25 ± 6.09*	63.50	0.365	Repellent
	LC_{20}	77.75 ± 3.84	22.25 ± 3.84*	55.50	0.445	Repellent
	LC_{10}	70.50 ± 2.60	29.50 ± 2.60*	41.00	0.590	Repellent
Pelargonium graveolens	LC_{50}	96.75 ± 0.60	3.25 ± 0.60*	93.67	0.063	Repellent
	LC_{20}	96.63 ± 0.55	3.38 ± 0.55*	93.33	0.067	Repellent
	LC_{10}	96.13 ± 1.51	3.88 ± 1.51*	92.67	0.073	Repellent

* Significant by the *t*-test ($p < 0.05$)

arvensis essential oil. *Acorus calamus* completely inhibited the adult emergence (100%) and being significant for adult emergence at all the concentrations tested i.e., $1/5^{th}$, $1/10^{th}$ and $1/20^{th}$ fraction of LC_{90}. Adult emergence differed significantly in *C. martinii*, *P. cablin* and *P. graveolens* at $1/5^{th}$ LC_{90}, $1/10^{th}$ LC_{90} and $1/20^{th}$ LC_{90}. Results revealed that exposure to higher sub-lethal concentrations caused greater reduction in progeny emergence.

Persistence of biological activity

The biological activity of EOs *vis-à-vis* time in terms of adult mortality, oviposition, progeny emergence and seed damage is presented in Tables 4, 5, 6 and 7. The residual toxicity of EOs differed significantly in respect of adult mortality, number of eggs laid, F1 progeny emergence and seed damage (Supplementary Tables 3–6). *Acorus calamus*

Table 3 Reduction in oviposition and adult emergence of *C. analis* exposed to different essential oils

Essential oil	Lethal Concentrations	No. of eggs laid (Mean ± SE)		% deterrence to oviposition	No. of adults emerged (Mean ± SE)		% inhibition to F1 adults
		Control	Treated		Control	Treated	
Mentha arvensis	$1/20^{th}$ LC_{90}	100.67 ± 2.73	94.67 ± 6.57	5.96	92.67 ± 7.69	73.00 ± 12.50	21.22
	$1/10^{th}$ LC_{90}	100.67 ± 2.73	77.33 ± 17.90	23.18	92.67 ± 7.69	50.67 ± 29.63	45.32
	$1/5^{th}$ LC_{90}	100.67 ± 2.73	49.00 ± 5.69*	51.32	92.67 ± 7.69	19.67 ± 9.35*	78.78
Acorus calamus	$1/20^{th}$ LC_{90}	109.00 ± 12.66	2.67 ± 1.76*	97.55	61.67 ± 6.77	0.00 ± 0.00*	100.00
	$1/10^{th}$ LC_{90}	109.00 ± 12.66	2.33 ± 2.33*	97.86	61.67 ± 6.77	0.00 ± 0.00*	100.00
	$1/5^{th}$ LC_{90}	109.00 ± 12.66	1.00 ± 0.00*	99.08	61.67 ± 6.77	0.00 ± 0.00*	100.00
Cymbopogon martinii	$1/20^{th}$ LC_{90}	112.00 ± 2.00	57.67 ± 16.19	48.51	102.00 ± 6.93	46.00 ± 13.01*	54.90
	$1/10^{th}$ LC_{90}	112.00 ± 2.00	20.00 ± 1.15*	82.14	102.00 ± 6.93	5.67 ± 3.84*	94.44
	$1/5^{th}$ LC_{90}	112.00 ± 2.00	0.00 ± 0.00*	100.00	102.00 ± 6.93	0.00 ± 0.00*	100.00
Pogostemon cablin	$1/20^{th}$ LC_{90}	110.33 ± 4.33	79.67 ± 14.67	27.79	97.00 ± 3.61	53.67 ± 14.43*	44.67
	$1/10^{th}$ LC_{90}	110.33 ± 4.33	69.00 ± 3.79*	37.46	97.00 ± 3.61	51.33 ± 1.86*	47.08
	$1/5^{th}$ LC_{90}	110.33 ± 4.33	59.33 ± 5.78*	46.22	97.00 ± 3.61	51.67 ± 7.26*	46.74
Pelargonium graveolens	$1/20^{th}$ LC_{90}	115.33 ± 2.91	97.33 ± 9.06	15.61	96.00 ± 6.43	65.67 ± 3.84*	31.60
	$1/10^{th}$ LC_{90}	115.33 ± 2.91	58.67 ± 17.14*	49.13	96.00 ± 6.43	44.67 ± 11.86*	53.47
	$1/5^{th}$ LC_{90}	115.33 ± 2.91	32.67 ± 6.49*	71.68	96.00 ± 6.43	8.00 ± 5.03*	91.67

* Significant by the *t*-test ($p < 0.05$)

Table 4 Effect of residual toxicity of essential oils on the mortality of *C. analis* adults

Essential oils	Per cent mortality (mean ± SE)						
	Days after treatment						
	0	14	28	42	56	70	84
Mentha arvensis	100.00 ± 0.00 $^{a\,A}$	100.00 ± 0.00 $^{a\,A}$	100.00 ± 0.00 $^{a\,A}$	94.44 ± 5.56 $^{a\,A}$	94.44 ± 5.56 $^{ab\,A}$	88.89 ± 5.56 $^{a\,A}$	55.56 ± 5.56 $^{ab\,B}$
Cymbopogon martinii	100.00 ± 0.00 $^{a\,A}$	100.00 ± 0.00 $^{a\,A}$	100.00 ± 0.00 $^{a\,A}$	33.33 ± 9.62 $^{a\,AB}$	16.67 ± 0.00 $^{c\,AB}$	5.56 ± 5.56 $^{b\,B}$	5.56 ± 5.56 $^{b\,B}$
Pogostemon cablin	88.89 ± 5.56 $^{a\,A}$	83.33 ± 9.62 $^{a\,AB}$	77.78 ± 5.56 $^{a\,AB}$	61.11 ± 5.56 $^{a\,AB}$	61.11 ± 5.56 $^{b\,AB}$	55.56 ± 5.56 $^{ab\,AB}$	16.67 ± 0.00 $^{ab\,C}$
Pelargonium graveolens	94.44 ± 5.56 $^{a\,A}$	61.11 ± 5.56 $^{a\,A}$	50.00 ± 0.00 $^{a\,A}$	50.00 ± 9.62 $^{a\,A}$	27.78 ± 5.56 $^{c\,AB}$	16.67 ± 9.62 $^{ab\,AB}$	5.56 ± 5.56 $^{b\,B}$
Acorus calamus	100.00 ± 0.00 $^{a\,A}$	100.00 ± 0.00 $^{a\,A}$	100.00 ± 0.00 $^{a\,A}$	100.00 ± 0.00 $^{a\,A}$	100.00 ± 0.00 $^{a\,A}$	94.44 ± 5.56 $^{a\,A}$	94.44 ± 5.56 $^{a\,A}$
Control	11.11 ± 5.56 $^{b\,A}$	5.56 ± 5.56 $^{b\,A}$	11.11 ± 5.56 $^{b\,A}$	5.56 ± 5.56 $^{b\,A}$	0.00 ± 0.00 $^{d\,A}$	5.56 ± 5.56 $^{b\,A}$	5.56 ± 5.56 $^{b\,A}$

Mean (± SE) followed by same lowercase letter(s) in the same column, or same uppercase letter(s) in the same row, are homogeneous subsets ($p = 0.05$, Tukey's HSD test)

demonstrated strong residual toxicity (94.44% mortality) even at 84 days after storage of treated seeds followed by *M. arvensis* (55.56%). Minimum egg deposition was observed in *A. calamus* (9 eggs), followed by *M. arvensis* (38.33 eggs) and *C. martinii* (57 eggs) treated seeds at 84 days after treatment. *Cymbopogon martinii* and *P. graveolens* EOs demonstrated high degree of seed protection (0% damage) for 42- and 28-days post-treatment. The F1 adult emergence was completely inhibited in *M. arvensis* and *A. calamus* treated seeds, hence no seed damage was observed for 70- and 84-days post-treatment.

Residual toxicity of EOs decreased with time but at varied rates for each EO. The EO's of *M. arvensis*, *C. martinii*, *P. cablin* and *P. graveolens* differed significantly for storage period in respect of adult mortality, F1 progeny emergence and seed damage (Supplementary Tables 3, 5 and 6). Oviposition varied significantly with time for all the EOs tested (Supplementary Table 4). Residual toxicity of *A. calamus* in respect of adult mortality did not reduce significantly with storage periods. *Acorus calamus* exhibited high residual activity causing 94.44–100 per cent mortality till 84 days post-treatment and, no adult emergence or seeds damage was recorded. *Mentha arvensis, C. martinii* and *P. graveolens* provided complete seed protection for 70-, 42- and 28-days post-treatment, respectively.

Discussion

Plant EOs are biodegradable and low-risk options for pest control in stored grains and products (Regnault-Roger et al. 2012) where harmful residues of synthetic insecticides are

Table 5 Effect of residual toxicity of essential oils on the oviposition by *C. analis*

Essential oils	Number of eggs laid (mean ± SE)						
	Days after treatment						
	0	14	28	42	56	70	84
Mentha arvensis	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	38.33 ± 11.17 $^{b\,B}$
Cymbopogon martinii	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	48.67 ± 6.69 $^{cd\,B}$	53.67 ± 15.21 $^{c\,B}$	57.00 ± 9.29 $^{bc\,B}$
Pogostemon cablin	3.67 ± 0.67 $^{b\,A}$	10.00 ± 1.73 $^{c\,A}$	26.33 ± 2.60 $^{c\,B}$	28.67 ± 7.67 $^{c\,B}$	32.00 ± 5.29 $^{c\,B}$	53.67 ± 6.33 $^{c\,BC}$	92.67 ± 6.49 $^{c\,C}$
Pelargonium graveolens	0.00 ± 0.00 $^{a\,A}$	1.33 ± 0.33 $^{b\,AB}$	5.00 ± 0.00 $^{b\,BC}$	12.33 ± 4.10 $^{b\,C}$	45.00 ± 16.44 $^{c\,DE}$	57.33 ± 20.80 $^{c\,E}$	71.33 ± 12.99 $^{bc\,E}$
Acorus calamus	0.00 ± 0.00 $^{a\,A}$	2.00 ± 0.58 $^{b\,B}$	7.33 ± 1.67 $^{b\,C}$	7.33 ± 0.67 $^{b\,C}$	8.00 ± 1.00 $^{b\,C}$	8.67 ± 0.33 $^{b\,C}$	9.00 ± 1.15 $^{a\,C}$
Control	78.00 ± 13.32 $^{c\,A}$	87.33 ± 6.57 $^{d\,A}$	98.33 ± 14.53 $^{d\,A}$	91.67 ± 5.49 $^{d\,A}$	114.33 ± 7.54 $^{d\,A}$	112.33 ± 11.20 $^{c\,A}$	99.00 ± 12.22 $^{c\,A}$

Mean (± SE) followed by same lowercase letter(s) in the same column, or same uppercase letter(s) in the same row, are homogeneous subsets ($p = 0.05$, Tukey's HSD test)

Table 6 Effect of residual toxicity of essential oils on the *C. analis* F1 adult emergence

Essential oils	Number of F1 adults emerged (mean ± SE)						
	Days after treatment						
	0	14	28	42	56	70	84
Mentha arvensis	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	34.00 ± 10.07 $^{b\,B}$
Cymbopogon martinii	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	17.67 ± 3.76 $^{b\,B}$	18.67 ± 1.76 $^{b\,C}$	29.00 ± 0.58 $^{b\,D}$
Pogostemon cablin	2.33 ± 0.33 $^{b\,A}$	7.33 ± 1.45 $^{b\,AB}$	14.67 ± 8.21 $^{b\,B}$	25.33 ± 9.33 $^{c\,BC}$	29.33 ± 5.46 $^{b\,BC}$	41.00 ± 2.31 $^{cd\,CD}$	72.00 ± 9.17 $^{c\,D}$
Pelargonium graveolens	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	5.67 ± 2.03 $^{b\,B}$	23.00 ± 4.04 $^{b\,C}$	35.67 ± 6.64 $^{a\,D}$	41.67 ± 2.03 $^{bc\,D}$
Acorus calamus	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{c}	0.00 ± 0.00 ^{a}
Control	49.67 ± 13.32 $^{c\,A}$	64.67 ± 8.41 $^{c\,A}$	61.33 ± 6.96 $^{c\,A}$	76.67 ± 2.60 $^{d\,A}$	73.33 ± 7.51 $^{c\,A}$	66.33 ± 6.69 $^{d\,A}$	78.67 ± 10.90 $^{c\,A}$

Mean (± SE) followed by same lowercase letter(s) in the same column, or same uppercase letter(s) in the same row, are homogeneous subsets ($p = 0.05$, Tukey's HSD test)

intolerable. Present study demonstrates that five EOs namely, *M. arvensis, C. martinii, P. graveolens, P. cablin* and *A. calamus* possessed insecticidal as well as repellent and oviposition deterrent properties against bruchid species, *C. analis* at sub-lethal amounts. Essential oils exhibited significant residual toxicity to ward off the bruchid infestation. In contact toxicity assay, test insect mortality varied according to test EOs, exposure duration and concentrations. Similar observations were reported in case of stored-product coleopterans [*Trogoderma granarium* (Everts) and *T. castaneum* (Herbst)] responses to EOs (Nenaah and Ibrahim 2011). Although, all EOs had insecticidal activity, *P. cablin* exhibited highest contact toxicity to adult beetles. *Acorus calamus* EO showed strong toxicity causing total mortality at higher duration of exposure (72 h). Toxicity of *A. calamus* was primarily affected by exposure time rather than dosage as previously proved by El-Nahal et al. (1989) on adults of five stored-product pest species. Mortality and exposure time relationship could be related to penetration ability of active compounds in a given time. The differences in toxicity could be largely due to the chemical composition of EOs derived from different plant species or plant families (Tapondjou et al. 2005) as well as physiological state of insects (Nenaah 2014a, b). The differential response of bruchid species to diverse EOs had been previously reported (Kim et al. 2003; Papachristos and Stamopoulos 2004; Gusmao et al. 2013; Dutra et al. 2016). Contact action of *P. cablin* to *C. maculatus* (F.) (Gusmao et al. 2013), *M. arvensis* to *C. chinensis* (L.) (Kumar et al. 2009), *P. graveolens* to *C. maculatus* (Manju et al. 2018), *Sitophilus zeamais* Motschulsky (Odeyemi et al. 2008; Kabera et al. 2011)*S. oryzae* (L.) (Abdelgaleil et al. 2016) have been demonstrated. Zimmermann et al. (2021) reported the contact toxicity of *M. arvensis* to *S. oryzae* and *S. zeamais*. *Cymbopogon martinii* exerted modest contact toxicity to *T.*

Table 7 Effect of residual toxicity of essential oils on the seed damage by *C. analis*

Essential oils	Per cent seed damage (mean ± SE)						
	Days after treatment						
	0	14	28	42	56	70	84
Mentha arvensis	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	36.56 ± 10.82 $^{bc\,B}$
Cymbopogon martinii	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	11.62 ± 2.47 $^{b\,B}$	12.28 ± 1.16 $^{b\,B}$	19.08 ± 0.38 $^{b\,C}$
Pogostemon cablin	1.90 ± 0.27 $^{b\,A}$	4.82 ± 0.96 $^{b\,A}$	9.65 ± 5.40 $^{b\,AB}$	20.60 ± 7.59 $^{c\,BC}$	19.30 ± 3.59 $^{b\,BC}$	26.97 ± 1.52 $^{c\,C}$	47.37 ± 6.03 $^{c\,C}$
Pelargonium graveolens	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	0.00 ± 0.00 $^{a\,A}$	3.73 ± 1.33 $^{b\,B}$	15.13 ± 2.66 $^{b\,C}$	23.46 ± 4.37 $^{c\,C}$	27.41 ± 1.33 $^{bc\,C}$
Acorus calamus	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{a}	0.00 ± 0.00 ^{a}
Control	32.68 ± 8.76 $^{c\,A}$	42.54 ± 5.53 $^{c\,A}$	40.35 ± 4.58 $^{c\,A}$	50.44 ± 1.71 $^{d\,A}$	48.25 ± 4.94 $^{c\,A}$	43.64 ± 4.40 $^{d\,A}$	51.75 ± 7.17 $^{c\,A}$

Mean (± SE) followed by same lowercase letter(s) in the same column, or same uppercase letter(s) in the same row, are homogeneous subsets ($p = 0.05$, Tukey's HSD test)

castaneum (Caballero-Gallardo et al. 2014). Insecticidal properties of *A. calamus* were evidenced against *Callosobruchus phaseoli* (Gyllenhal) (Rahman and Schmidt 1999), *Bruchus chinensis* L. (Yadava 1971) or *C. chinensis* (El-Nahal et al. 1989; Kim et al. 2003). Insecticidal properties of EOs attributed to numerous bioactive constituent compounds (Ogendo et al. 2008). The previous studies reported high monoterpene (Bett et al. 2016; Ebadollahi et al. 2022) and sesquiterpene (Basile et al. 2022; Vaglica et al. 2022) contents in biocidal EOs. Toxicity of lemongrass to *C. maculatus* was reported to be attributed to its citral isomers (de Souza Alves et al. 2019). Toxic effect of *C. martinii* on *C. chinensis* was due to mixture of constituent compounds rather than a major compound, geraniol (Kumar et al. 2007). The major monoterpenes geraniol, linalool and citronellol exhibited similar toxicity as that of *P. graveolens* EO against *Bemisia tabaci* Genadius (Baldin et al. 2015). Huang et al. (2014) demonstrated that pogostone constituent responsible for insecticidal effects of *P. cablin* EO to lepidopteran insects. Insecticidal effects of β-asarone compound contributed to the toxicity of *A. calamus* EO to *C. chinensis* (El-Nahal et al. 1989; Schmidt et al. 1991). Lee et al. (2001) reported that menthone, linalool and α-pinene constituents possibly contributed to the toxicity of *M. arvensis* EO against *S. oryzae* weevils. Essential oils and their bioactive compounds are reported to be neurotoxic to insects (Mssillou et al. 2022) by inhibiting acetylcholinesterase (Ryan and Byrne 1988; Abdelgaleil et al. 2009) and by interfering with neuromodulator octopamine (Kostyukovsky et al. 2002; Isman et al. 2007) or gamma-aminobutyric acid (GABA) receptors (Priestley et al. 2003). The broad-spectrum insecticidal effects are principally due to the presence of multiple bioactive compounds (Park and Tak 2016). All the five EOs at sub-lethal concentrations tested, recorded RI of < 1, indicating the repellent property towards *C. analis* adults. Repellent effect was varied according to the EO concentrations. *Cymbopogon martinii, P. graveolens* and *A. calamus* EOs showed promising repellent property with over 83% repellency even at lowest sub-lethal concentrations. The presence of certain bioactive volatile compounds in these EOs possibly elicited the strong deterrent action on the visiting insects. Repellency of *M. arvensis* and *P. cablin* EOs have been reported against *C. chinensis* (Kumar et al. 2009), *T. castaneum, Lasioderma serricorne* (F.) (Feng et al. 2019), while *P. graveolens* against *C. maculatus* (Manju et al. 2018). Repellent activity of *C. martinii* was recorded against pests of stored legumes (*C. chinensis*) and cereals [*Rhyzopertha dominica* (F.), *S. oryzae, S. zeamais, T. castaneum, Oryzaephilus surinamensis* (L.)] (Kumar et al. 2007; Hernandez-Lambrano et al. 2015). *Acorus calamus* EO repelled *T. castaneum*, *R. dominica* (Jilani et al. 1988; Jilani and Saxena 1990) and *C. chinensis* adults (Shukla et al. 2016). The most toxic EO (in contact assay), *P. cablin*, did not demonstrate highest repellency at sub-lethal amounts of all EOs tested. This is in contrary to the reports of Papachristos and Stamopoulos (2002b); Kim et al. (2010). Sub-lethal exposure to EOs significantly impacted the number of eggs laid and emergence of F1 progenies. *Acorus calamus* EO found strongly oviposition deterrent, eventually complete inhibition to adult emergence was observed. *Cymbopogon martinii* at upper sub-lethal concentration, prevented the oviposition as well as progeny emergence. The egg laying (6–100% reduction in oviposition) and adult emergence (21–100% inhibition rate) was affected in a dose-dependent manner for tested EOs at sub-lethal concentrations. Adverse effect of sub-lethal exposure to clove and cinnamon EOs on developmental traits had been demonstrated in *C. maculatus* (Viteri Jumbo et al. 2018). Sub-lethal exposure to EOs substantially affected oviposition behaviour (Kiran et al. 2017), fecundity and fertility (Pavela 2012) rather than mortality. Reduced oviposition and progeny emergence by *A. calamus* vapour treatment was reported in *C. chinensis* (Schmidt et al. 1991) and *Callosobruchus phaseoli* (Gyll.) (Rahman and Schmidt 1999). Inhibitory effect of sub-lethal dosage of peppermint and eucalyptus EOs on fertility and fecundity was studied in *Acanthoscelides obtectus* (Say) (Hategekimana and Erler 2020). Inhibition to egg laying and adult emergence in *Callosobruchus spp.* (*C. maculatus* and *C. chinensis*) was previously reported from *M. arvensis, C. winterensis, Citrus* sp., *Eucalyptus* sp. and *Foeniculum vulgare* (Mill.) EO treated seeds (Raja et al. 2001; Pandey et al. 2011; Gusmao et al. 2013; Dutra et al. 2016). Progeny suppression in different stored-product pests with EOs have also been confirmed by several researchers (Tapondjou et al. 2002; Tripathi et al. 2002; Teke and Mutlu 2021; Hategekimana and Erler 2020). Changes in physiology or behaviour of insects on exposure to EOs perhaps impacted the egg laying capacity of female beetles (Raja et al. 2001; Shukla et al. 2011) and in turn reduced the number of progeny emergence. The residual toxicity of EOs markedly affected the survival and development of bruchids reducing the seed damage. The persistence of biological activity of EOs diminished with time but at varied rates for each EO. *Acorus calamus*, *M. arvensis*, *C. martinii* and *P. graveolens* treated seed were totally free from bruchid damage until 84, 70, 42 and 28 days post-treatment, respectively. Prolonged seed protection was due to the lethal effect on adults and eggs deposited. Direct seed dressing with *A. calamus* had offered a high degree of protection up to a period of 135 days against *C. chinensis* in mungbean (Chander and Ahmed 1986) and 91 days against *B. chinensis* (Yadava 1971). EOs, in the present study, could have affected according to their chemical compositions, thus, exhibiting variations in persistence. EOs undergo oxidation of mono- and sesquiterpenes (Ilboudo et al. 2010) enhancing loss in bioactivity, while EOs with high hydrogenated compounds are vulnerable to oxidation

(reviewed therein Nenaah et al. 2015). In the present study, five EOs demonstrated noticeable contact, repellent and oviposition deterrent activity against *C. analis*. The diverse bioactivity of these EOs possibly due to the presence of several bioactive ingredients and their synergistic or antagonistic interactions (Park et al. 2003; Bakkali et al. 2008; Tak and Isman 2015; Wang et al. 2019) and operating via several modes of action (Abdelgaleil et al. 2016; Campolo et al. 2018). Sub-lethal dosages of some EOs exhibited as high as 95% repellency (*C. martinii*), while reduction in viable eggs and progeny emergence was up to 100% (*A. calamus* and *C. martinii*). Insecticidal properties of plants belonging to Acoraceae, Lamiaceae and Poaceae have been previously pointed out in several studies (Jacobson 1989; Kim et al. 2003; Rajendran and Sriranjini 2008). The bioactivity of EOs at sub-lethal dosages and their residual activity revealed in the present study open new perspectives for the management of stored-product pests.

Conclusion

The results indicated a promising prospect of EOs for managing the devastating pest of stored food legumes, *C. analis*. The EOs namely *M. arvensis, C. martinii, P. graveolens, P. cablin* and *A. calamus* not only exhibited the contact toxicity and repellency but also had negative effect on egg laying and progeny emergence even at sub-lethal quantities. These EOs possessed adequate insecticidal and residual activities to be considered as active ingredients to develop eco-friendly grain-protectants for managing *C. analis* in stored food legumes, since they are organic in origin, biodegradable in environment and pose low-risk to mammals including consumers and applicators. However, further studies need to be conducted to investigate the effect of these potential EOs on treated seeds. Since the EOs are highly volatile, the improved delivery methods need to be developed.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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