Journal of Stored Products Research 116 (2026) 102979

Contents lists available at ScienceDirect

Journal of Stored Products Research

journal homepage: www.elsevier.com/locate/jspr

Lethal and sub-lethal effects of two botanical nano-insecticides against *Liposcelis bostrychophila*

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ARTICLE INFO

Keywords:
Essential oil
Syzygium aromaticum
Mentha piperita
Stored product pest
Psocids

ABSTRACT

Psocids represent a serious pest species in stored products due to their small size and widespread resistance to conventional insecticides. Essential oils (EOs) are promising pest control tools, despite some drawbacks that limit their use under real operating contexts. In response to these challenges, in the last decade, the use of nanotechnology has enhanced EO applications as insecticides. In this context, this study aimed to develop *Syzygium aromaticum* L. (Clove) and *Mentha piperita* L. (Mint) EO-nano-emulsions (EO-NEs) with a low amount of co-formulants using the high-pressure microfluidization technique, assessing lethal and sub-lethal effects against *Liposcelis bostrychophila* Badonnel (Psocoptera: Liposcelididae). Gas chromatography-mass spectrometry analysis (GC-MS) showed eugenol (76.2%) and menthol (26.69%) as major compounds in clove and mint EOs, respectively. The Dynamic Light Scattering analysis (DLS) revealed excellent physical properties for both EO-NEs with particle sizes ranging in the nanoscale (<250 nm) and good PDI values (<0.25) until 6 months. The lethal effects, assed through residual contact toxicity bioassay, showed high insecticidal activity for clove EO-NE, with estimated lethal doses (LDs) considerably lower compared to mint EO-NE (i.e., LD_{50} and LD_{90} of 7.62 and 24.22 μg/cm^2 of EO and LD_{50} and LD_{90} of 96.72 and 124.79 μg/cm^2 of EO, respectively). In addition, mint EO-NE significantly affects the number of produced eggs and their hatchability compared to the negative control treatment. Overall, these findings suggest a potential use of these EO-NEs as sustainable food-stored pest management tools.

1. Introduction

Psocids, commonly known as booklice, are small insects (about 1.0 mm), usually wingless, soft-bodied, brownish or pale yellow, with large heads and protruding eyes. They were formerly considered minor pests of stored products, along with other insects, such as silverfish and cockroaches (Nayak et al., 2014). Their presence in stored products was often associated with high humidity conditions or poor storage practices, usually causing negligible or no damage (Ahmedani et al., 2010; Wei et al., 2023). Since the 1980s, severe infestations have been increasingly reported in stored products, animal feed, and food-processing facilities, including flour mills, tobacco warehouses, and bakeries. Infestations may also affect food transportation systems, resulting in substantial economic losses (Smith, 1985; Mashaya, 1999; Trematerra and Fiorilli, 1999; Arbogast et al., 2000). Moreover, large-scale infestations have been associated with significant human health concerns, such as allergic reactions and the transmission of pathogens (Ahmedani et al., 2010; Mediannikov et al., 2022). Approximately 100 psocid species can successfully infest stored products. Among them, *Liposcelis bostrychophila* Badonnel (Psocoptera: Liposcelididae) is one of the species that causes significant economic damage worldwide (Green and Turner, 2005; Nayak et al., 2014; Pollov, 2016). Traditionally, these pests were managed using synthetic insecticides, which were primarily designed to target beetles and moths. However, certain biological traits of psocids, such as parthenogenetic reproduction, high reproductive capacity, a short life cycle, and a long egg stage, greatly reduce the effectiveness of these treatments. Moreover, the emergence of insecticide resistance further limits the success of conventional control methods (Nayak et al., 2020; Tanaka et al., 2025). This has led to the need for more sustainable alternatives to synthetic insecticides, which are well known for their harmful impacts on the environment, human health, and non-target organisms (Chagnon et al.,

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https://doi.org/10.1016/j.jspr.2026.102979
Received 17 November 2025; Received in revised form 10 January 2026; Accepted 31 January 2026
Available online 4 February 2026
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2015; Serrão et al., 2022).

Given these challenges, over the past decade, the use of botanicals, particularly essential oils (EOs) as alternatives to conventional pesticides, has attracted the interest of researchers. This interest is driven not only by their natural role in plant defense, being produced in response to biotic and abiotic stresses, but also because they are considered phytocomplexes, composed of a wide mixture of bioactive compounds (Arshad et al., 2014; Kaneko et al., 2024). The different constituents of EOs act synergistically to interfere with multiple physiological and metabolic pathways in insects, particularly those linked to the nervous system, which also helps reduce the risk of resistance development. These effects include inhibition of acetylcholinesterase (AChE), modulation of octopamine and GABA receptors, and disruption of development, antioxidant defenses, and cuticular permeability (Abdelgaleil et al., 2009; Jankowska et al., 2018; Mossa, 2016). These properties make EOs promising candidates for pest control, and several studies have consequently explored their efficacy as insecticides against stored product pests, often reporting significant effects on insect mortality (Campolo et al., 2018; Chaudhari et al., 2021; Nikolaou et al., 2021; Saad et al., 2022; Leite et al., 2023). As highlighted by Rajkumar et al. (2019), *Mentha piperita* L. EO showed that the concentrations 75.0 μL/L air and 100.0 μL/L air were able to achieve 100% of mortality against *Sitophilus oryzae* L. (Coleoptera: Curculionidae) and *Tribolium castaneum* Herbst. (Coleoptera: Tenebrionidae), respectively. These findings were consistent with those obtained by other authors using the same EO (Lashgari et al., 2014; Saeidi and Mirfakhraie, 2017; Mackled et al., 2019; Atay et al., 2024). Similarly, other authors using EO extracted from *Syzygium aromaticum* L. reported high biological activities against different stored product pest including *Rhyzopertha dominica* (Fabricius) (Coleoptera: Bostrichidae), *Sitophilus zeamais* Motschulsky (Coleoptera: Curculionidae), *Oryzaephilus surinamensis* (L.) (Coleoptera: Silvanidae) (Wuttiwong et al., 2019; Wanna et al., 2025). Nevertheless, despite their considerable insecticidal potential, EOs have several limitations—including high volatility, flammability, rapid degradation, and limited or negligible water solubility—that hinder their practical application under real-world conditions (Isman, 2020; Gupta et al., 2023; Assadpour et al., 2024).

To address these challenges, nanotechnology has been increasingly employed to deliver EOs via nano-carrier systems, such as nanoparticles and nano-emulsions, which can reduce the limitations of EOs while enhancing their physical, chemical, and biological performance (Menossi et al., 2021; Kumar et al., 2025). Several authors reported the efficacy of EO-based nano-insecticides against stored product pests (Gharsan, 2024; Maurya et al., 2024; Tirunagaru et al., 2025). Sabbour (2020) reported good insecticidal activity and a reduction in fertility and emergence rate of *T. castaneum* and *Tribolium confusum* du Val (Coleoptera: Tenebrionidae) treated with coriander, Janesville, caraway, and black seed EO-based nano-formulations. Other authors reported high insecticidal activity of *Pelargonium graveolens* L'Hér. EO-based nano--emulsions against *S. oryzae* with an estimated LC_{50} of 2.298 ppm/cm^2 (Mesbah et al., 2023). Similarly, Giunti et al. (2019) developed a sweet orange EO-based nano-emulsion and tested its efficacy against *T. confusum* and *Cryptolestes ferrugineus* (Stephens) (Coleoptera: Cucujidae), highlighting good insecticidal activity with estimated LD_{90} of 18.86 and 54.39 mg*L^{-1}, respectively. Although the insecticidal efficacy of essential oil–based nano-emulsions against several stored-product pests is well documented, their potential against psocids has been largely overlooked. To the best of our knowledge, no data are available on the efficacy of essential oil–based nano-emulsions against psocids, and the only available evidence, although extremely limited, refers to the application of essential oils used as such (Liu et al., 2012; Pang et al., 2020; Wang et al., 2019).

In this context, this study aimed to develop *Syzygium aromaticum* (clove) and *Mentha piperita* (mint) essential oil-based nano-emulsions (EO-NEs) and to investigate their biological activity against *Liposcelis bostrychophila*. In particular, the chemical profiles of the EOs and the physical properties of the resulting EO-NEs were assessed, together with their lethal and sub-lethal effects on the target insect.

2. Materials and methods

2.1. Chemical composition of EOs

Commercial clove and mint EOs (label purity ≥99%) were purchased from Esperis S.p.A. (Milan, Italy). The chemical composition of the EOs was determined by gas chromatography–mass spectrometry (GC–MS). Analyses were performed using a Thermo Fisher TRACE 1300 GC system equipped with a MEGA-5 capillary column (30 m × 0.25 mm, film thickness 0.25 μm) and coupled to a Thermo Fisher ISQ LT mass spectrometer operating in electron ionization (EI) mode (scan time: 1.00 s; mass range: 30–300 m/z). Injector and transfer line temperatures were set at 250 °C and 240 °C, respectively. The oven temperature was programmed to rise from 60 °C to 240 °C at a rate of 3 °C min^{-1} (carrier gas: He 1 mL min^{-1}) (Adams, 2017). The EOs were individually diluted in hexane (95%, Sigma Aldrich, Munich, Germany) at a ratio of 1:10 v:v (EO: hexane), and 0.2 μL were injected at a split ratio of 1:30. The identification of peaks was made using computer matching against the commercial libraries (NIST 05, Wiley FFNSC, and ADAMS), comparing linear retention indices (LRI). The LRIs were calculated using the formula of Van Den Dool and Kratz (1963), by comparing the retention times of the compounds to be identified with those of a standard mixture of alkanes (C8-C20 saturated alkanes standard mixture, Supelco®, Bellefonte, PA, USA), which was analyzed in GC-MS under the identical operating conditions as the sample (Van Den Dool and Kratz, 1963; Masada, 1976; McLafferty and Stauffer, 1989; Davies, 1990; Jennings, 2012; Adams, 2017).

2.2. EO-NES: development and physical characterization

Clove and mint EO-NEs were prepared individually using high-pressure microfluidization, following the procedure described by Modafferi et al. (2024a), with slight modifications. Briefly, a coarse emulsion was first obtained by combining EO (15% w/w), Tween 80® (polyoxyethylene (20) sorbitan monooleate, Sigma-Aldrich, Munich, Germany) (5% w/w), and double-distilled water (80% w/w). The mixture was stirred at 6000 rpm for 5 min with a magnetic stirrer to ensure homogeneity. The coarse emulsion was then processed three times using a high-pressure microfluidizer (LM20 Microfluidizer™ Processor, USA) at 30,000 PSI. To prevent thermal degradation of the emulsion components (EO and Tween 80) due to the heat generated during microfluidization, the interaction chamber and heat exchanger were immersed in an ice bath to maintain temperatures below 10 °C. The resulting nano-emulsion was collected in aluminum bottles and stored at 4 °C until use.

The particle size (nm), polydispersity index (PDI), and surface charge (zeta potential) of the formulated clove and mint EO-based nano-emulsions (EO-NEs) were determined by dynamic light scattering (DLS) using a Zetasizer Nano instrument (Malvern®). Prior to analysis, samples were diluted in double-distilled water at a 1:200 (v/v) ratio. For each measurement, 1.00 mL of the diluted EO-NE was used to determine particle size and PDI, while 0.75 mL was used for zeta potential assessment.

2.3. Insect rearing

Liposcelis bostrychophila was reared for several generations on an artificial diet of wheat flour and baking yeast powder (ratio 10:1 w/w) (Opit and Throne, 2014), at the entomology laboratories of the Department of Agriculture, University "*Mediterranea*" of Reggio Calabria, Reggio Calabria, Italy. The original colony was collected from infested wheat flour. Rearing was carried out in glass bottles (500 mL) under constant climatic conditions (25 ± 2 °C, 70 ± 5% R.H., and a

photoperiod of 16:8 L: D) (Wang et al., 2000).

2.4. Residual contact toxicity

The toxicity of developed EO-NEs against *L. bostrychophila* adults (15 days old) was evaluated using contact toxicity bioassays following the method described by Liu et al. (2012), with modifications. In detail, bioassays were carried out by treating filter paper (Ø 4 cm) with 150 μL of the desired treatment. Subsequently, the treated filter paper was dried at room temperature (25 ± 2 °C for 10 min) and placed in ventilated plastic Petri dishes (Ø 4 cm). Then, twenty *L. bostrychophila* adults were gently moved on the treated filter paper, and the arena was kept at the same above-mentioned rear conditions (i.e., 25 ± 2 °C, 70 ± 5% R.H., and with a photoperiod of 16:8 L: D). The mortality was recorded 24 h after the treatments. Insects were considered dead if immobile after stimulation with a fine brush or unable to walk. In order to estimate the dose-response mortality, for each EO-NEs, a total of five dilutions (i.e., 1.5, 3, 6, 12, 24 μg/cm^2 of EO and 72, 84, 96, 108, 120 μg/cm^2 of EO for clove and mint EO-NE, respectively) were used. The test dilutions were obtained by diluting the EO-NE, which contained 15% of EO (see section 2.2), in double-distilled water (w/w). Each dilution was replicated four times, and water and deltamethrin at the label dose (12.5 mg/L) were used as negative (C-) and positive (C+) controls, respectively.

2.5. Sub-lethal effects

The biological activity, in terms of the number of laid eggs and their hatchability, of developed EO-NEs was evaluated using two different bioassays. In detail, the first experiment (namely, direct) was carried out by treating *L. bostrychophila* specimens using the residual contact toxicity method as described above (see section 2.4). Twenty-four hours after the exposure, the treated insects were gently transferred into 1g of untreated wheat grain. The second experiment (namely, indirect) was carried out by immersing 1 g of wheat grain in the desired treatment formulation for 30 s and then drying at room temperature. Then, 24 h later, untreated insects were carefully transferred into treated wheat grain. For each experiment, the number of laid eggs was recorded 24 h after the release of the insects in the arena, using a stereoscope (Olympus SZX7, Olympus Corporation™, Japan). Instead, the hatchability of the laid eggs was monitored for 20 days, according to the method described by Nayak et al. (2003). All bioassays were carried out in ventilated Plastic Petri dishes (Ø 4 cm), kept under the same rear conditions mentioned above (see Section 2.3). For both direct and indirect bioassays, a total of three treatments were performed using water as a negative control (C-) and the lethal dose $(LD)_{25}$ (4.14 and 84.58 μg/cm^2 of EO) and LD_{50} (7.62 and 96.72 μg/cm^2 of EO) estimated in the residual contact toxicity bioassay for clove and mint EO-NEs, respectively (see result section 3.3). For all treatments, four replications of 20 adults (15 days old) each were used.

2.6. Ovideterrent and ovicidal activities

The percentage of ovideterrent and ovicidal activities of the effective EO-NE (i.e., mint EO-NE), in terms of sub-lethal effects (see results section 3.4), was calculated using the number of laid eggs and their hatchability for both direct and indirect sub-lethal bioassays described above (section 2.5). For all replicates, both parameters were calculated using the formula described by Giunti et al. (2022) with modifications:

$$POD = [(N^\circ \text{ eggs control} - N^\circ \text{ eggs treatment}) / N^\circ \text{ eggs control}] \times 100$$

$$POC = [(N^\circ \text{ eggs control} - N^\circ \text{ eggs treatment}) / N^\circ \text{ eggs control}] \times 100$$

Where POD was the percentage of ovideterrent activity, and POC was the percentage of ovicidal activity.

2.7. Data analysis

Statistical analyses were carried out using IBM® SPSS® Statistics 2v.23 (IBM Corp. Armonk, NY, USA). All data met the assumptions required for parametric testing, including normality and homoscedasticity of variance assessed by Levene and Shapiro-wilk tests respectively ($P > 0.05$).

Differences in physical characteristics between the developed mint and clove EO-NE at the different times (i.e., 1 day, 1 month, and 6 months) were analyzed using the unpaired two-tailed *t*-test.

The toxicological data obtained 24 h after the insect exposure with different doses of the developed EO-NEs were corrected for control mortality using Abbott's formula (Abbott, 1925). Differences among the different doses within the same EO-NE were assessed using ANOVA analysis, and multiple comparisons were assessed using Tukey's HSD post hoc test. In addition, the data fitted with the probit model (Person's Goodness of Fit test), and the LDs and their 95% confidence limits were estimated for both mint and clove EO-NEs.

Differences among the different treatments (i.e., LD_{25}, LD_{50}, and C-) in the total number of laid eggs and their hatchability were evaluated using ANOVA, and multiple comparisons were assessed using Tukey's HSD post hoc test.

The POD and POC (percentage of ovideterrent activity and percentage of ovicidal activity, respectively) were analyzed using an unpaired two-tailed *t*-test in order to assess differences between the two doses tested (i.e., LD_{25} and LD_{50}).

3. Results

3.1. Chemical characterization of EOs

The chemical composition of clove and mint EOs is shown in Tables 1 and 2, respectively. A total of forty-three and seventy-seven compounds corresponding to 99.03% and 98.52% of the total calculated area were detected for clove and mint EOs, respectively. In detail, clove EO was highly rich in eugenol (76.2%), followed by caryophyllene and acetyl-eugenol (corresponding to 8.17% and 7.31%, respectively). Instead, menthol (26.69%), menthone (13.57%), and isomenthone (12.74%) were the three most abundant compounds in mint EO.

3.2. Characterization of EO-Nes

The physical characterization of developed EO-NEs showed excellent properties with particle sizes ranging in the nanoscale range (<250 nm) until the end of the observations (up to six months). In detail, both EO-NEs highlighted an increasing trend over time in the particle size, with values ranging from 77.03 ± 1.09 to 149.3 ± 0.91 nm and from 151.6 ± 1.47 to 235 ± 9.92 nm for mint and clove EO-NEs, respectively. Regarding the PDI, both EO-NEs had good particle size distribution with PDI values lower than <0.25. Twenty-four hours after development, mint EO-NE showed a very low PDI value (0.085 ± 0.011) of less than half compared to clove EO-NE (0.213 ± 0.012). As shown for the particle size, PDI values also showed an increased trend over time, with values of 0.142 ± 0.012 and 0.220 ± 0.017 for mint and clove EO-NEs, respectively. Statistical differences were recorded between the developed EO-NEs at all observed times, both for size and PDI values (Table 3). Instead, zeta potential results showed negative surface charges for both EO-NEs with values ranging from 13.2 ± 0.57 to −20.7 ± 0.88 and from −15 ± 0.35 to −24.6 ± 0.91 for mint and clove EO-NE, respectively.

3.3. Residual contact toxicity bioassay

The toxicological bioassays highlighted high insecticidal activity for both developed clove and mint EO-NEs against the target insect 24 h after exposure, and statistical differences among the different tested

Table 1
Chemical characterization (GC-MS) of clove EO.

COMPOUND	LRI[a]	LRI[b]	RT	Area %
Furfural	831	831	3.34	0.09
α-Pinene	933	929	5.24	0.02
Camphene	948	947	5.62	c
Benzaldehyde	959	961	5.92	c
2-Furaldehyde, 5-methyl-	963	964	6.01	c
β-Pinene	977	975	6.36	c
5-Hepten-2-one, 6-methyl-	987	986	6.62	c
α-Phellandrene	1006	1005	7.14	c
o-Cymene	1024	1027	7.78	0.02
Limonene	1028	1028	7.91	0.05
Eucalyptol	1031	1031	8.01	0.07
2-Heptanol, acetate	1041	1034	8.35	c
Linalool oxide	1072	1074	9.44	c
β-Linalool	1101	1101	10.43	0.03
β-Thujone	1107	1110	10.67	c
Camphor	1144	1146	12.17	0.02
endo-Borneol	1165	1168	13	c
Benzoic acid, ethyl ester	1171	1171	13.23	c
α-Terpineol	1191	1190	14.04	c
Methyl salicylate	1194	1193	14.16	0.29
Phenol, 4-(2-propenyl)-	1256	1259	16.77	0.41
(E)-Anethole	1285	1284	17.99	c
α-Cubebene	1349	1352	20.65	c
Eugenol	1371	1373	21.58	76.2
α-Copaene	1377	1377	21.8	0.93
Vanillin	1400	1401	22.8	0.12
Methyleugenol	1408	1410	23.09	0.17
Caryophyllene	1420	1418	23.59	8.17
β-Caryophyllene	1428	1428	23.92	0.04
Humulene	1453	1451	24.93	1.4
Alloaromadendrene	1460	1462	25.21	0.02
β-Cadinene	1473	1474	25.75	0.05
γ-Muurolene	1477	1479	25.88	0.03
β-Selinene	1485	1489	26.24	c
α-Muurolene	1500	1499	26.83	0.02
δ-Cadinene	1523	1524	27.73	0.54
Acetyleugenol	1532	1525	28.06	7.31
Caryophyllenyl alcohol	1569	1568	29.52	0.07
β-Caryophyllene oxide	1583	1583	30.04	2.42
Humulene oxide	1608	1605	31.03	0.25
Alloaromadendrene oxide	1636	1631	32.05	c
Cubenol	1642	1643	32.29	0.04
Coniferyl aldehyde	1734	1741	35.63	0.04
TOTAL	**99.03**			

3 Retention time.
[a] Calculated Linear Retention Index.
[b] Litterature Linear Retention Index.
[c] ≤ 0.01 %.

doses and C+ were observed (F = 227.16; df = 5; $P < 0.001$ and F = 97.17; df = 5; $P < 0.001$ for clove and mint EO-NEs, respectively). Dose-response mortality pattern was observed for both EO-NEs, and lethal doses and their 95% confidence limits were estimated ($X^2 = 1.96$; df = 3; Sign. = 0.753 and $X^2 = 3.37$; df = 3; Sign. = 0.338 for clove and mint EO-NEs, respectively) (Fig. 1). In detail, clove EO-NE exhibited very high insecticidal activity with estimated LD_{25} and LD_{50} (4.14 and 7.62 μg/cm^2 of EO, respectively) less than ten times compared to mint EO-NE (LD_{25} of 84.58 and LD_{50} of 96.72 μg/cm^2 of EO, respectively). Notable differences were also observed in the estimated LD_{90} between the developed EO-NEs (24.22 and 124.79 μg/cm^2 of EO for clove and mint EO-NEs, respectively). Statistical differences were recorded between the developed EO-NEs in the LD_{25}, LD_{50}, and LD_{90} as their confidence intervals did not overlap (Table 4). Complete LDs and their 95% confidence intervals are shown in Supplementary Table 1.

3.4. Sub-lethal effects against the target insect

The sub-lethal effects, in terms of total number of laid eggs and their hatchability in both bioassays (i.e., direct and indirect), of developed

Table 2
Chemical characterization (GC-MS) of mint EO.

COMPOUND	LRI[a]	LRI[b]	RT	Area %
3-Hexen-1-ol, (Z)-	852	857	3.66	c
1-Hexanol	864	869	3.85	c
2,5-Diethyltetrahydrofuran	899	901	4.39	0.06
Anisole	919	917	4.9	c
α-Thujene	926	932	5.06	0.03
α-Pinene	933	939	5.24	0.94
Camphene	948	955	5.62	0.03
2,4(10)-Thujadien	953	958	5.75	c
4 (10)-Thujene	973	978	6.26	0.66
β-Pinene	977	980	6.37	1.82
3-p-Menthene	984	986	6.54	0.02
β-Myrcene	991	991	6.72	0.19
(+)-β-Pinene	996	989	6.85	0.87
3-Octanol	1005	1002	7.13	0.03
δ-3-Carene	1011	1011	7.32	c
α Terpinene	1017	1018	7.52	0.11
(2E,4E)-3,7-Dimethylocta-2,4-diene	1022	1025	7.69	c
o-Cymene	1024	1022	7.78	0.23
Limonene	1029	1031	7.94	4.13
Eucalyptol	1032	1033	8.05	5.21
(Z)-β-Ocimene	1036	1040	8.2	0.06
(E)-β-Ocimene	1047	1050	8.55	0.04
γ-Terpinene	1058	1062	8.93	0.16
cis-Sabinene hydrate	1067	1068	9.24	0.34
1-Octanol	1070	1070	9.36	0.31
Trans-Linalool oxide	1072	1074	9.44	0.05
Terpinolene	1089	1088	10	0.13
3-Nonanol	1097	1099	10.29	0.06
Linalool	1101	1098	10.44	0.52
Isopentyl pentoate	1105	1106	10.6	c
Isopentyl isovalerate	1109	1103	10.76	0.03
trans-p-Mentha-2,8-dien-1-ol	1122	1125	11.26	0.11
cis-p-Mentha-2,8-dien-1-ol	1137	1138	11.87	0.05
cis-Sabinol	1142	1143	12.08	0.08
Isopulegol	1147	1145	12.28	2.63
Menthone	1158	1154	12.71	13.57
Isomenthone	1168	1166	13.13	12.74
Menthol	1180	1178	13.58	26.69
Isomenthol	1187	1188	13.88	1.74
Neoisomenthol	1192	1188	14.07	0.48
α-Terpineol	1194	1192	14.14	0.76
1,6-Dihydrocarveol	1197	1202	14.29	0.49
trans-Dihydrocarvone	1203	1201	14.52	0.08
trans-Carveol	1220	1217	15.25	0.1
Pulegone	1241	1237	16.11	1.14
Carvone	1245	1242	16.29	0.42
Piperitone	1256	1252	16.74	1.81
1-Decanol	1275	1272	17.53	0.43
Isomenthol acetate	1276	1275	17.61	0.15
Anethole	1289	1284	18.12	6.59
Menthyl acetate	1297	1294	18.47	5.29
Isomenthol acetate	1310	1305	19	c
δ-Elemene	1339	1337	20.15	0.17
Euguenol	1358	1356	21.03	0.07
α-Ylangene	1375	1372	21.74	0.08
β-Bourbonene	1384	1384	22.12	0.67
β-Elemene	1392	1392	22.44	0.2
Longifolene	1403	1402	22.92	0.05
Isocaryophillene	1406	1404	23.03	c
Caryophyllene	1419	1418	23.54	2.47
β-Copaene	1428	1432	23.92	0.24
trans-α-Bergamotene	1435	1439	24.21	0.02
Aromadendrene	1443	1439	24.54	0.14
(E)-β-Famesene	1458	1456	25.13	0.16
Germacrene D isomer	1463	1458	25.32	0.03
Germacrene D	1480	1480	26.04	0.94
Valencene	1496	1491	26.66	0.76
α-Muurolene	1500	1499	26.82	0.08
cis-γ-Cadinene	1513	1514	27.35	0.08
δ-Cadinene	1523	1524	27.74	0.29
α-Cadinene	1537	1538	28.27	0.02
Nerolidol	1565	1566	29.36	0.08
Spathuneol	1577	1576	29.82	0.09
β-Caryophyllene oxide	1582	1581	30.02	0.16

(continued on next page)

Table 2 (*continued*)

COMPOUND	LRI[a]	LRI[b]	RT	Area %
Viridiflorol	1590	1590	30.35	0.06
τ-Muurolol	1642	1638	32.26	0.08
α-Cadinol	1654	1653	32.73	0.04
TOTAL	**98.52**			

3 Retention time.
[a] Calculated Linear Retention Index.
[b] Litterature Linear Retention Index.
[c] ≤ 0.01 %.

EO-NEs against *L. bostrychophila* are shown in Fig. 2 (A and B, respectively).

Regarding the total number of laid eggs, statistical differences were observed in both experiments using the LDs of mint EO-NE. In detail, in the direct bioassay ($F = 39.81$; $df = 2$; $P < 0.001$), the LD_{25} (84.58 μg/cm^2 of EO) and LD_{50} (96.72 μg/cm^2 of EO) exhibited a total number of laid eggs (i.e., 7.75 ± 0.75 and 6.5 ± 0.29, respectively) that were more than twice lower than the C- treatment (14.5 ± 0.87). A similar trend was also observed in the indirect bioassay ($F = 33.21$; $df = 2$; $P < 0.001$). The total number of eggs recorded in the C- treatment (11.25 ± 0.48) was double compared to the LD_{25} (5 ± 0.71) and the LD_{50} (3.75 ± 0.85).

In addition, mint EO-NE also affects the egg hatchability, and statistical differences among the treatments were observed. Specifically, in the indirect bioassay ($F = 262.5$; $df = 2$; $P < 0.001$), both LD_{25} and LD_{50} highlighted less hatchability (5 ± 1.25% and 6.67 ± 1.67%, respectively) compared to the C- treatment (80% ± 0.91%). Instead, no statistical differences were observed in the direct treatment ($P > 0.05$).

In contrast, the LDs (i.e., $LD_{25} = 4.14$ and $LD_{50} = 7.62$ μg/cm^2 of EO, respectively) of clove EO-NE did not determine statistical differences ($P > 0.05$) in the total number of laid eggs and their hatchability compared to the negative control (C-) in both experiments (i.e., direct and indirect).

3.5. Ovideterrent and ovicidal activities

The exposure to mint EO-NE LDs affected both the percentage of ovideterrence (POD) and ovicidal activity (POC) in both direct and indirect experiments (Fig. 3).

Specifically, POD results indicated good ovideterrent activity, with average values of 45.88 ± 6.25% for LD_{25} and 54.95 ± 1.95% for LD_{50} in the direct route of exposure, while slightly higher percentages were observed in the indirect trials (55.72 ± 5.30% and 67.58 ± 6.40% for LD_{25} and LD_{50}, respectively). However, no statistical differences were recorded either between the LDs within the same route of exposure or between the same LD across different routes of exposure ($P > 0.05$).

Regarding the POC results, both LD_{25} and LD_{50} showed intermediate values in the direct exposure (48.40 ± 8.85% and 54.45 ± 6.71%, respectively) and very high values in the indirect exposure (LD_{25} and LD_{50} of 94.37 ± 3.29% and 96.87 ± 3.12%, respectively); however, no statistical differences were recorded in both routes of exposure between the LDs ($P > 0.05$). Conversely, statistical differences were recorded between the tested LDs across different routes of exposure (LD_{25}: $F = 2.78$; $t = 5.16$; $P = 0.002$ and LD_{50}: $F = 3.87$; $t = 5.73$; $P = 0.001$).

4. Discussion

The analysis of the chemical composition revealed that clove EO was mainly composed of eugenol (76.2 % of the total area), followed by caryophyllene and acetyl-eugenol (8.17 % and 7.31 % of the total area, respectively). Instead, menthol, menthone, and isomenthone (26.69 %, 13.57 %, and 12.74 % of the total area, respectively) were the three most abundant compounds detected in mint EO. These findings were consistent with those obtained by different authors (Abbas et al., 2024; Ahamad, 2023; Moghaddam et al., 2013; Teles et al., 2021). However, the EO composition depends on several factors, including environmental conditions, the part of plants used for extraction, the methodology of extraction, and the analytical process (Barra, 2009; Das and Prakash, 2023; Mugao, 2024). Furthermore, the different EOs' composition can influence their biological activity, since the single compounds may exhibit synergistic or antagonistic effects (Jankowska et al., 2018).

In this study, long-term stable mint and clove EO-NEs were developed using a high-energy system, such as high-pressure microfluidization. Generally, these systems (i.e., sonication, high-pressure homogenization or microfluidization) are well known to produce EO-NEs with optimal physical properties in terms of particle size and PDI values. Among high-energy emulsification systems, the method used in this study seems to be the best option for the preparation of EO-NEs for insecticidal applications, owing to the long-term stability and the excellent physical properties that can be achieved. This is supported by the finding of Modafferi et al. (2025), who showed that high-pressure microfluidizer system allowed the preparation of a garlic EO-NE with smaller particle sizes and excellent PDI values compared to those obtained using other high-energy methods, including sonication. Indeed, as reported in the results of the physical characterization, the developed mint and clove EO-NEs highlighted particle sizes ranging in the nanoscale (<250 nm) and good PDI values (<0.25) up to 6 months. These results were consistent with those reported by other authors. Despite using a different high-energy system (ultrasonication *vs*. microfluidization), Özakar et al., 2024 developed 1-day-old mint EO-NE with average particle size close to that observed for our 6-month-old mint EO-NE (154.6 ± 1.42 nm and 0.142 ± 0.012 nm, respectively). Other authors, using the same ultrasound technique, produced a mint EO-NE with a particle size of ~ 120 nm (Bai et al., 2025), similar to our 3-month-old mint EO-NE. High-pressure homogenization (HPH) has been used to produce clove EO-based nano-emulsions with particle sizes slightly smaller than those obtained in the present study (151.6 ± 1.47 nm) and low polydispersity index (PDI) values, achieved by increasing the surfactant concentration up to a 1:1 EO:surfactant ratio (Ghazali and Zakaria, 2025). While higher surfactant levels generally enhance the physical stability of nano-emulsions by reducing particle size and PDI, excessive surfactant content may increase the risk of adverse effects on ecosystems (Ivanković and Hrenović, 2010). For instance, Mustapha and Bawa-Allah (2020), highlighted that two non-ionic surfactants affect the survivability in *Clarias gariepinus* (Burchell) with estimated LC_{50} ranging

Table 3
Physical characteristics (i.e., size, PDI, and zeta potential) of mint and clove EO-NE, 1 day, 1 month, and 6 months after development. For each characteristic, different letters indicate statistical differences between mint and clove EO-NEs at the same time after development.

EO-NE[a]	Time	Size nm (mean ± S.D.[b])	F - t - *P*	PDI (mean ± S.D.)	F - t - P	Zeta mV (mean ± S.D.[1])	F - t - P
Mint	1 day	77.03 ± 1.09a	3.38–80.63 -< 0.001	0.085 ± 0.011a	0.01 - 13.25 -< 0.001	−13.2 ± 0.57a	1.05–4.66 - = 0.01
Clove		151.6 ± 1.47b		0.203 ± 0.012b		−15 ± 0.35b	
Mint	1 month	114.9 ± 1.05a	2.69–41.27 -< 0.001	0.119 ± 0.009a	0.79 - 4.81 - = 0.009	−20.1 ± 0.33a	6.89 - 1.42 - = 0.227
Clove		187.2 ± 2.84b		0.213 ± 0.013b		−22.9 ± 0.72a	
Mint	6 months	149.3 ± 0.91a	4.24–21.83 -< 0.001	0.142 ± 0.012a	0.77 - 7.13 - = 0.002	−20.7 ± 0.88a	0.95- 7.49 - = 0.002
Clove		235 ± 9.92b		0.220 ± 0.017b		−24.6 ± 0.91b	

[a] EO-NE.
[b] Standard deviation.

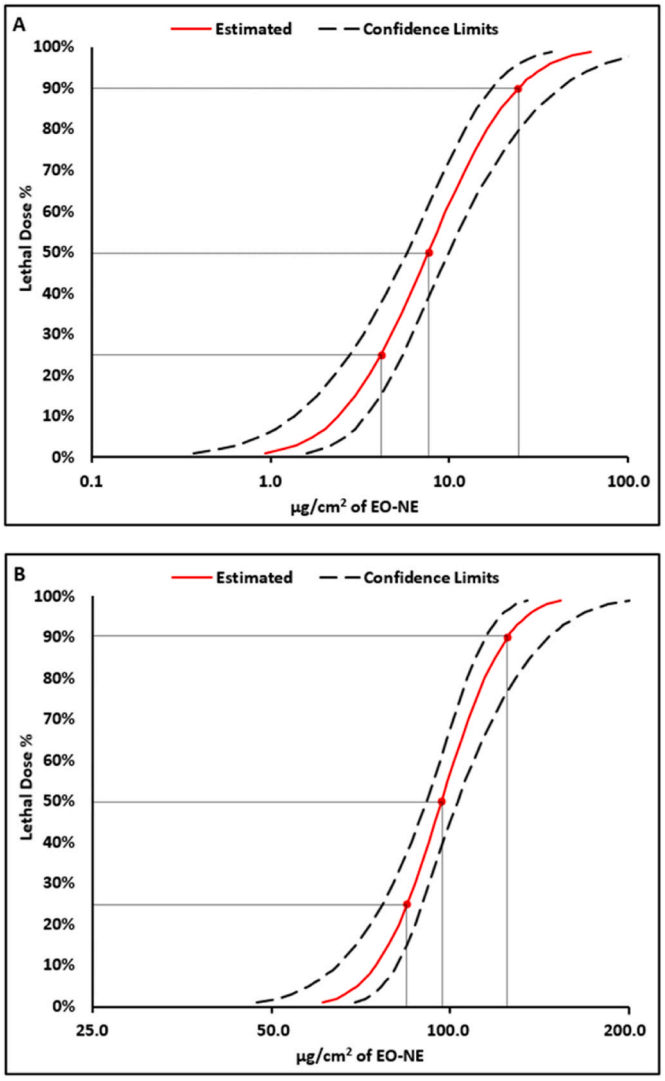


Fig. 1. Dose-response mortality (%) of the developed clove (A) and mint (B) EO-NEs against *L. bostrychophila*, 24 h after the exposure.

Table 4
Estimate LD_{25}, LD_{50}, and LD_{90} of developed *clove and mint* EO-NEs against *L. bostrychophila* adults 24 h after the exposure. Values were considered significantly different if their 95% fiducial limits did not overlap.

EO-NE[a]	n[b]	Slope (±S.E.)	LD[c]	Estimate (µg/cm² of EO)	95% Confidence Limits	
					Lower Bound	Upper Bound
Clove	5	2.55 (±0.37)	LD_{25}	4.14	2.78	5.48
			LD_{50}	7.62	5.79	9.92
			LD_{90}	24.22	17.25	41.54
Mint	5	11.58 (±2.08)	LD_{25}	84.58	76.89	89.84
			LD_{50}	96.72	91.22	102.86
			LD_{90}	124.79	114.64	146.44

[a] Essential oil-based nano-emulsion.
[b] Number of cases.
[c] Lethal dose.

from 16.88 mg/L to 22.61 mg/L. Lastly, the analysis of the ζ-potential of the developed Nes reported negative values ranging from −13.2 ± 0.57 to −20.7 ± 0.88 for mint EO-NE and from −15 ± 0.35 to −24.6 ± 0.91 for clove EO-NEs. These results indicated the long-term stability (up to 6 months) achieved for both EO-NEs due to good electrostatic and steric repulsion among the particles (Malhotra and Coupland, 2004; Akbari and Nour, 2018).

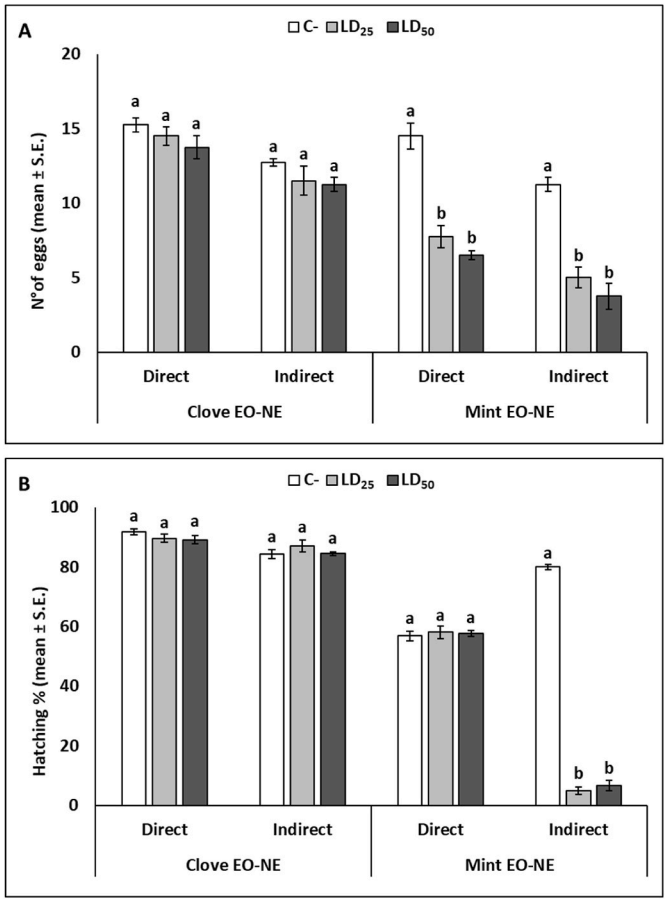


Fig. 2. Total number of laid eggs (**A**) and the percentage of hatching (**B**) (mean ± S.E.) recorded in both sub-lethal experiments (i.e., direct and indirect). Different letters indicate statistical differences among the different treatments (i.e., C-[2], LD_{25}, and LD_{50}) within the same experiment (direct or indirect) for each EO-NE (Tukey's HSD post hoc $P < 0.05$).

The study of biological effects of developed EO-NEs revealed good insecticidal activity against *L. bostrychophila*. Between the two EO-NEs tested, the mint-based one obtained the highest estimated LDs with LD_{25}, LD_{50}, and LD_{90} values of 84.58, 96.72 and 1204.79 µg/cm² of EO, respectively. Conversely, the clove EO-NE exhibited very low lethal dose values (LD_{25}, LD_{50}, and LD_{90} of 4.14, 7.62, and 24.22 µg/cm² of EO, respectively), indicating a high toxicity against the target insect. To better contextualize this strong insecticidal activity, it is noteworthy that a previous study using pyrethrum extract against the same species reported an LD_{50} value nearly twice as high (18.99 µg/cm²) (Liu et al., 2012). To the best of our knowledge, this represents the first investigation assessing the efficacy of essential oil–based nano-emulsions against this pest, while the limited data available in the literature are restricted to crude EOs. Zhao et al. (2012) investigated the contact and fumigant toxicity of different EOs, including *S. aromaticum*, against *L. bostrychophila*, showing that a concentration of 0.31 µL/cm² was able to achieve 100% mortality. Similarly, a solution containing 1% of mint EO exhibited high contact insecticidal activity against *L. bostrychophila* adults resulting in 100% mortality (Pang et al., 2020). In addition, the same authors reported good fumigant (LC_{50} and LC_{90} of 0.6 and 1.4 mg/L air, respectively) and repellent activities for mint EO. Although the activity of mint and clove EOs have been poorly studied, the effects of other EOs against *L. bostrychophila* have been better documented. Liu et al. (2012) demonstrated good contact insecticidal activity for the EO extracted from *Curcuma wenyujin* Y. H. Chen and C. Ling. Rhizomes, with an estimated LC_{50} of 208.85 µg/cm². Other authors investigated the

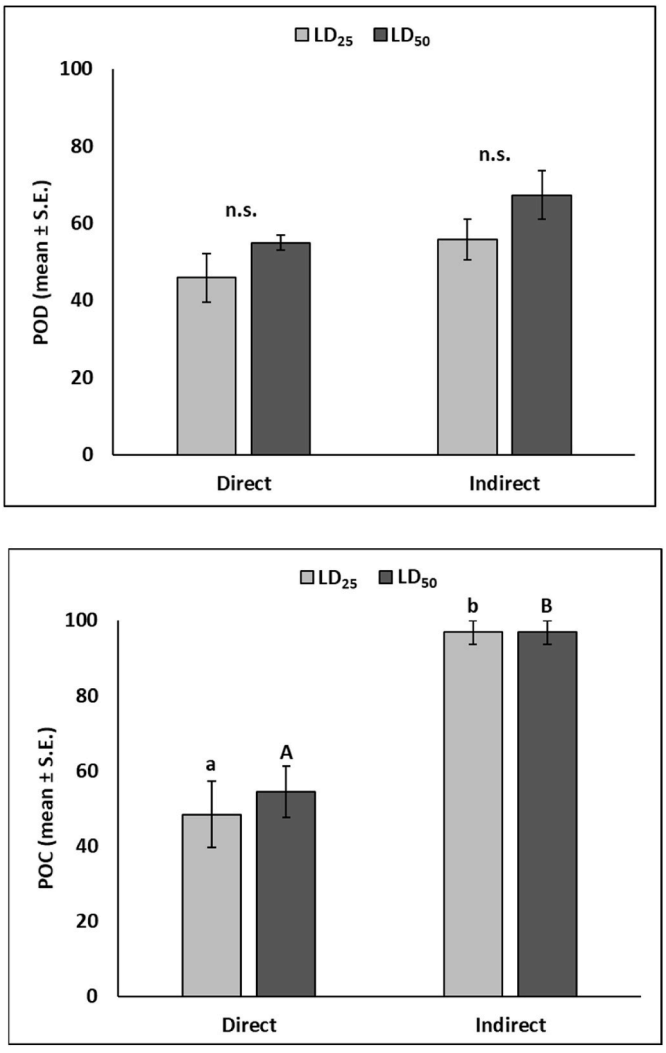


Fig. 3. POD and POC (mean ± S.E.), calculated using the total number of laid eggs and the percentage of hatching, respectively, in both experiments (i.e., direct and indirect) for mint EO-NE. Different letters indicate statistical differences between the exposure routes (direct or indirect) within the same LD (LD_{25} or LD_{50}) (*t*-test, $P < 0.05$).

biological activity of different EOs such as *Cinnamomum burmannii* (Nees & T.Nees) Blume, *Cinnamomum migao* H. W. Li, *Litsea pungens* Hemsl., and *Laurus nobilis* L. (Wang et al., 2019), highlighting good contact toxicity, with estimated LC_{50} values of 50.51, 100.15, 129.91, and 202.95 μg/cm^2, respectively.

Although the insecticidal activities of crude EOs against this pest are few documented, no data have been reported on sub-lethal effects. Nevertheless, the results of this study highlighted a reduction in the number of eggs laid and a negative effect on egg hatchability when adult insects were exposed to lethal doses of mint EO-NE. These findings are consistent with previous studies reporting sub-lethal effects, even when conventional insecticides were used. As Nayak et al. (2003) highlighted, the hatching rate of *L. bostrychophila* eggs decreases with increasing phosphine concentrations. Furthermore, a significant delay in hatching was observed in treated eggs compared to untreated eggs. Although this study is the first to document the sub-lethal effects of EO against *L. bostricophila*, the sub-lethal effects of EO against other pests have been documented. As described by Giunti et al. (2022), anise, fennel and mint EO-NEs significantly reduced the number of ovipositions in olive fruits of *Bactrocera oleae* (Rossi) (Diptera: Tephritidae) females compared to control treatment, with estimated repellence concentration $(RC)_{50}$ of 3.25%, 3.47%, and 7.95% of EO, respectively. Furthermore, *Eucalyptus globulus* Labill EO-NE could significantly affects the egg vitality on *Spodoptera litura* (Fab.) (Lepidoptera: Noctuidae) (Ankur et al., 2025). Similarly, Khater et al. (2025), observed reduced larval and pupal weight, lower adult emergence, and decreased reproductive capacity in *Earias insulana* Boisd. (Lepidoptera: Nolidae) exposed to different treatments with *Croton tiglium* L. EO and its nano-emulsion.

Although the results of the present study highlighted excellent biological activity of the developed EO-NEs against *L. bostrychophila*, further investigations are required to comprehensively assess the safety of these botanical-based nano-insecticides on non-target organisms. The potential adverse effects of nano-insecticides are influenced by several factors, including the type of EO used as active ingredient, its concentration within the nano-formulation, application rate, routes of exposure, the biological characteristics of non-target organisms, the amount and nature of co-formulants, as well as the physicochemical properties of the nano-formulations themselves (Gahukar and Das, 2020; Manna et al., 2023; Modafferi et al., 2024b). Despite the relevance of these aspects, the ecological impact of botanical-based nano-insecticides remains poorly investigated. Nevertheless, the limited available evidence suggests that the same EOs used in the present study, although formulated in different nano-systems, are associated with very low toxicity toward terrestrial and aquatic non-target organisms (Saraiva et al., 2021; Yeguerman et al., 2022). Moreover, the biological activity of clove and mint EOs used as such was widely investigated. In this regard, clove EO was reported to be safe for honeybees, inducing very low mortality rates (<3%) (Gostin and Popescu, 2023). While mint EO showed low adverse effects on pollinators and natural enemies, such as predators and parasitoids, with mortality rates generally below 10% (Attia et al., 2022; Souza et al., 2022). In addition, the application of mint EO was shown to cause negligible phytotoxic effects on sweet pepper plants and to positively modulate the expression of different genes involved in natural plant defence mechanisms (Ricupero et al., 2023).

5. Conclusion

This study underscores the efficacy of novel EO-based nano-insecticides against *L. bostrychophila*. The mint and clove EO-NEs developed using high-pressure microfluidization exhibited very good physical characteristics with particle size ranging in the nanoscale (<250 nm), low PDI values and negative ζ-potential, indicating long-term stability (up to 6 months). The study of lethal and sub-lethal effects showed very high insecticidal activity with low estimated LD_{90} for both tested EO-NEs (24.22 μg/cm^2 and 124.79 μg/cm^2 for clove and mint EO-NEs, respectively), and a reduced number of eggs laid as well as their hatchability in insects treated with mint EO-NE. Overall, the findings reveal a dual-action mechanism of these nano-formulations, supporting their potential as alternatives to conventional pesticides under controlled conditions. However, challenges such as potential effects on non-target organisms, scalability of production, and practical application need to be addressed before real-world use. Exploring these aspects would provide valuable insights for practical applications and help guide the development of sustainable stored product pest management strategies.

CRediT authorship contribution statement

Maria Pineda: Writing – original draft, Investigation. **Ilaria Latella:** Writing – review & editing, Validation, Conceptualization. **Pierluigi Forlano:** Writing – review & editing, Validation, Formal analysis. **Giulia Giunti:** Writing – review & editing, Formal analysis, Conceptualization. **Orlando Campolo:** Writing – review & editing, Formal analysis, Conceptualization. **Antonino Modafferi:** Writing – review & editing, Investigation, Formal analysis, Conceptualization. **Vincenzo Palmeri:** Writing – review & editing, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

Maria Pineda was supported by the project "Technologies for climate change adaptation and quality of life improvement" *EI TECH4YOU - SPOKE 3 (Smart technologies for sustainable agrifood chain and forestry), Goal 3.1 (*Agriculture and livestock smart farming*), PP 3.1.1 (Targeting precision farming applications to agroecological intensification) – CUP: C33C22000290006*.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jspr.2026.102979.

Data availability

Psocoptera_data_2025","edm:link":"https://data.mendeley.com/datasets/5zdf96kr72/1

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