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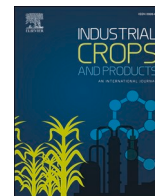
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Harnessing essential oil synergy to enhance the repellency of partially biobased polymer against sap-feeding hemipteran pests

Sylvain Benhamou^{a,b}, Chrystèle Jouve^a, Isabelle Rahioui^a, Zaïnab Belgaidi^c,
Garance Lapetoule^a, Laurence Mouton^c, Federica Calevro^a, Sébastien Livi^{b,*},
Pedro Da Silva^{a,*}

^a INSA Lyon, INRAE, BF2i, UMR 203, Villeurbanne 69621, France

^b Univ Claude Bernard Lyon 1, INSA Lyon, Univ Jean Monnet, CNRS UMR 5223, Ingénierie des Matériaux Polymères, F-69621, France

^c Université Claude Bernard Lyon 1, LBBE, UMR 5558, CNRS, VetAgro Sup, Villeurbanne 69622, France

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ABSTRACT

Sap-feeding hemipteran pests, such as aphids, cause significant yield losses and remain difficult to control sustainably, as conventional insecticides face increasing limitations due to resistance development and growing concern over human and environmental health. Plant essential oils (EOs) constitute a promising, eco-friendly alternative. However, their large-scale application is constrained by poor field stability and high production costs. These limitations may be mitigated through improved formulation and efficacy optimization, therefore reducing the amount of EO required for effective pest management. In this study, we investigated the repellent activity of binary and ternary blends of basil (*Ocimum basilicum* L.), Chinese cinnamon (*Cinnamomum cassia* (L.) J. Presl), and spearmint (*Mentha spicata* L.) EOs, incorporated into a polybutylene succinate co-adipate (PBSA) biopolymer matrix, against the pea aphid *Acyrtosiphon pisum* Harris. Applying a conceptual framework derived from mixture toxicology, we found that both the *M. spicata*–*O. basilicum* binary blend and the ternary combination exhibited strong synergistic interactions, resulting in an 8- to 24-fold increase in repellent potency compared to individual oils. Further exploration of different *M. spicata*–*O. basilicum* ratios led to additional improvements in blend efficiency. When tested on other sap-feeding hemipteran species, this optimized blend also demonstrated interspecific repellency. Our results support the rational design of EO-based bio-repellent blends as sustainable tools for pest management.

1. Introduction

Feeding a global population projected to approach 9 billion in the coming years requires fundamental rethinking of food production systems, notably emphasizing greater efficiency and a reduced environmental footprint. In this context, crop pests and diseases remain major threats to yield stability and food security. Combined, they are estimated to cause 20–40 % of global yield losses, which could rise to 40–80 % in the absence of crop protection measures (Orke and Dehne, 2004). Among the most widespread insect pests in temperate regions are aphids. Like many phloem sap-feeding hemipterans, aphids damage plants by disrupting nutrient allocation, reducing photosynthesis, promoting the growth of sooty mold through honeydew excretion, and,

most critically, acting as the main vectors for plant viruses (van Emden and Harrington, 2017). The pea aphid *Acyrtosiphon pisum* Harris (Hemiptera: Aphididae) is a major pest of leguminous crops such as peas, broad beans, and alfalfa (van Emden and Harrington, 2017; Sandhi and Reddy, 2020). These crops are valued not only for their nutritional content but also for their nitrogen-fixing capacity and their central role in crop rotation systems (Kebede, 2021; Stagnari et al., 2017; Voisin et al., 2014). Effective management of such pests is therefore essential for both yield preservation and agroecological sustainability.

Since the mid-20th century, insect management has largely relied on synthetic chemical insecticides. Although initially effective, their widespread use has raised significant concerns due to adverse effects on human health, negative impacts on non-target organisms, and the rapid

* Corresponding authors.

E-mail addresses: sylvain.benhamou@insa-lyon.fr (S. Benhamou), chrystele.jouve@inrae.fr (C. Jouve), isabelle.rahioui@inrae.fr (I. Rahioui), zainab.belgaidi@univ-lyon1.fr (Z. Belgaidi), garance.lapetoule@inrae.fr (G. Lapetoule), laurence.mouton@univ-lyon1.fr (L. Mouton), federica.calevro@insa-lyon.fr (F. Calevro), sebastien.livi@insa-lyon.fr (S. Livi), pedro.da-silva@insa-lyon.fr (P. Da Silva).

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evolution of resistance in pest populations (Aktar et al., 2009; Damalas and Eleftherohorinos, 2011; Desneux et al., 2007; Köhler and Trieb-skorn, 2013). These issues have led to increasingly stringent regulatory restrictions worldwide and have prompted the search for alternative pest management strategies that are both effective and environmentally sustainable.

Plant essential oils (EOs) have gathered growing attention as environmentally friendly biopesticides (Isman, 2020; Pavela and Benelli, 2016; Regnault-Roger et al., 2012). EOs are complex blends of volatile organic compounds (VOCs), primarily terpenoids and phenylpropanoids, synthesized by plants as secondary metabolites in response to biotic and abiotic stresses, including defense against herbivory (Pavela and Benelli, 2016). EOs exhibit a broad range of bioactivities against insects, including insecticidal, repellent, oviposition-deterrent, and anti-feeding effects, with most actions targeting the nervous system (Jankowska et al., 2018; Popescu et al., 2024). Among these, repellency is particularly appealing, as it enables effective crop protection at substantially lower doses than those required for lethal approaches, thus minimizing exposure to non-target species (Hikal et al., 2017; Ndakidemi et al., 2016). Furthermore, the natural origin and low mammalian toxicity of EOs, supported by their widespread use in food, cosmetics, and pharmaceuticals (Regnault-Roger et al., 2012), further reinforce their potential as promising candidates for biopesticide development.

Despite these advantages, the practical use of EOs remains limited by factors like high volatility, low water solubility, and instability under field conditions (Isman, 2016; Pavela and Benelli, 2016), requiring the development of improved formulations (e.g., Dunan et al., 2023). Moreover, EO production, typically achieved through steam or dry distillation (Burger et al., 2019), remains costly, and large-scale deployment often lacks an economically viable model (Isman, 2016; Lacotte et al., 2023; Pavela and Benelli, 2016). These challenges highlight the need for strategies that enhance EO performance in the field and reduce the quantity required for effective pest management.

One promising approach involves exploiting synergistic interactions. In the context of mixture toxicology, interactions are defined as deviations from the expected additive effect, where each component contributes independently (Roell et al., 2017). Synergism occurs when the observed effect exceeds this additive baseline, while antagonism reflects a sub-additive outcome (Roell et al., 2017). Such interactions have been widely studied in toxicology, ecotoxicology, pharmacology, and agriculture (Cedergreen, 2014; Duarte and Vale, 2022; Martin et al., 2021; Roell et al., 2017). As inherently complex blends, EOs are well-suited to exhibit such synergistic effects, with insecticidal activity often surpassing predictions based on the individual contributions of their components (Jiang et al., 2009; Kim et al., 2016; Tak et al., 2016). This phenomenon has been attributed to the intrinsic propensity of EO-derived compounds to act synergistically, whether they are derived from the same oil, in their natural proportions (Feng et al., 2020; Tak et al., 2016), or from different ones (Pavela, 2015, 2014). Among these compounds, some, including those that are weakly active or seemingly inactive, may indirectly enhance overall efficacy by promoting penetration (Kim et al., 2021; Tak and Isman, 2015, 2017a; Yoon and Tak, 2023), availability (Moustafa et al., 2023; Scalerandi et al., 2018; You et al., 2023) or bioactivity (Gaire et al., 2020; Kim et al., 2021) of other active constituent (Isman, 2016). Building on these internal dynamics, combining different EOs can further enhance insecticidal potency (Isman, 2016; Popescu et al., 2024). Synergistic effects in EOs-based repellency have recently begun to attract increasing attention, with a growing number of studies focusing on mosquitoes (Noosidum et al., 2014; Peach et al., 2019), storage weevils (Boukrra et al., 2022; Hategekimana and Erler, 2020; Miloudi et al., 2024), ticks (Aboelhadid et al., 2022) and household pests (Huang et al., 2020; Soonwera et al., 2024). Applications targeting agricultural crop pests, however, remain under-explored (Tak and Isman, 2017b).

A complementary strategy to enhance EO-based repellents involves

incorporating active compounds into polymer matrices, which can protect them from degradation and facilitate a more gradual, sustained release under field conditions. This strategy has been explored using various polymers combined with insecticides or repellents (Dáder et al., 2014; Finkenstadt and Tisserat, 2010; Seven et al., 2019), including EOs (Kim et al., 2020; Lacotte et al., 2025). Among these polymers, poly (butylene succinate-co-adipate) (PBSA) stands out as a biodegradable, partially bio-based material compatible with agricultural use (Akhir and Mustapha, 2022; Chien et al., 2022; Coiai et al., 2021; Lacotte et al., 2025; Nabeoka et al., 2021). Recent studies have shown that Chinese cinnamon (*Cinnamomum cassia* (L.) J. Presl), spearmint (*Mentha spicata* L.) and basil (*Ocimum basilicum* L.) EOs with repellent activity against *A. pisum* (Lacotte et al., 2023) can be effectively integrated into PBSA, yielding bioactive materials that combine efficient pest management with ecological sustainability (Lacotte et al., 2025).

In this study, we investigated whether combining *C. cassia*, *M. spicata* and *O. basilicum* EOs into binary and ternary blends, then incorporated into a PBSA-based polymer matrix, could further enhance their repellent efficacy against *A. pisum*. We first screened EO blends to identify synergistic combinations based on established toxicity additivity models, then optimized the most promising binary blend by adjusting the ratios of its components. Finally, we tested the efficacy of the optimized formulation against other agriculturally relevant sap-feeding hemipteran pests.

2. Material and methods

2.1. Insects

Five sap-feeding hemipteran crop pests were used in this study: four aphid species, *A. pisum* (pea aphid), *Myzus persicae* Sulzer (peach-potato aphid), *Rhopalosiphum padi* (L.) (bird-cherry oat aphid), and *Sipha maydis* Passerini (barley aphid) (Hemiptera: Aphididae), and the tobacco whitefly, *Bemisia tabaci* (Gennadius) Mediterranean (MED) species (Hemiptera: Aleyrodidae). All insects were reared in climate-controlled chambers on their respective host plants (all grown from organic-certified seeds) (Table 1), under conditions allowing continuous parthenogenetic reproduction in aphids. *A. pisum* (LL01 clone) and *R. padi* originated from long-established laboratory clones. *M. persicae* was sampled on peach orchards (*Prunus persica* (L.) Batsch) in Eymeux, France (2017), *S. maydis* was collected from barley (*Hordeum vulgare* L.) in Midelt, Morocco (2016) and the *B. tabaci* population was established from individuals sampled on *Mandevilla* sp. Lindl in Les Ponts-de-Cé, France (2012).

Synchronized individuals were obtained using a previously described protocol for aphids (Simonet et al., 2016), and equally applicable to whiteflies. Briefly, adult aphids (winged *A. pisum* or apterous *M. persicae*, *R. padi*, *S. maydis*) and whiteflies were transferred onto fresh, insect-free host plants and allowed to reproduce for 24 h before being removed. For aphids, the resulting synchronized first instar nymphs (<1 day old, N1) were either used immediately or aged until reaching the 2-day-old adult stage. For whiteflies, adult emergence was monitored daily and, 24 h after the first emergence, newly emerged adults were transferred to fresh plants, and 7-day-old females were used in assays.

2.2. Plant essential oils

The three EOs used in this study, Chinese cinnamon (*C. cassia*, aerial parts, Central Asia), basil (*O. basilicum*, aerial parts, India) and spearmint (*M. spicata*, aerial parts, India), were selected based on their strong and consistent repellent activity against *A. pisum* (Lacotte et al., 2023, 2025). These EOs were taken from the same stock (purchased from Voshuiles (Nevers, France)) as those used and chemically analysed in our previous study (Lacotte et al., 2025), which has been stored under constant conditions (4°C, in the dark) since. The chemical VOCs

Table 1

Host plant and climate conditions used for insect rearing.

Insect		Host plant		Climate conditions ^a		
Scientific name	Common name	Scientific name	Common name	T (°C)	RH (%)	LD (h/h)
<i>Acyrtosiphon pisum</i> Harris	Pea aphid	<i>Vicia faba</i> L.	Broad bean	20 ± 2	65 ± 5	16/8
<i>Bemisia tabaci</i> (Gennadius)	Tobacco whitefly	<i>Gossypium hirsutum</i> L.	Cotton	26 ± 1	70 ± 5	14/10
<i>Myzus persicae</i> Sulzer	Peach-potato aphid	<i>Brassica rapa</i> subsp. <i>chinensis</i> (L.) Hanelt	Pak choi	23 ± 1	55 ± 5	16/8
<i>Rhopalosiphum padi</i> (L.)	Bird-cherry oat aphid	<i>Triticum aestivum</i> L.	Bread wheat	20 ± 2	65 ± 5	16/8
<i>Sipha maydis</i> Passerini	Barley aphid	<i>Triticum aestivum</i> L.	Bread wheat	20 ± 2	65 ± 5	16/8

^a T: Temperature; RH: Relative humidity; LD: Light/dark hour cycle

composition of the EOs, as determined by gas chromatography-mass spectrometry (GC-MS) in Lacotte et al. (2025), is reported in Supplementary Table S1.

2.3. Processing of PBSA-EO films

PBSA pellets (BioPBS™ FD92PB) were purchased from MCPP Europe GmbH (Düsseldorf, Germany). PBSA-EO films were produced by melt extrusion. Before extrusion, PBSA was dried at 60 °C for 24 h. Appropriate amounts of EO or EO blend were added to 12 g of PBSA to reach the desired EO weight percentage, up to the technical limit of 10 wt%. The blends were prepared by using a 15 g-capacity DSM micro-extruder (Midi 2000 Heerlen, NL) with co-rotating screws (length-o-diameter (L/D) ratio: 18). The blends were processed at a screw speed of 100 rpm for 3 min with the barrel temperature uniformly set to 160 °C across the feeding zone, compression zone, and die. Extrusion parameters were optimized during preliminary trials to achieve a satisfactory compromise between homogeneous incorporation of the EOs into the molten polymer matrix and minimal thermal degradation (Lacotte et al., 2025). The extrudate was cast into a film in-line using a 400 µm × 45 mm flat die and a cast film line (Xplore Instruments), to obtain a final thickness of 80 ± 10 µm.

2.4. In vitro repellency bioassays

A series of *in vitro* dual-choice experiments were performed to evaluate the repellent activity of the PBSA-EO films against aphids and whiteflies. The protocol used was based on a method developed for *A. pisum* (Lacotte et al., 2023). Bioassays were carried out in air-tight T-shaped glass tubes (shaft: 3.5 cm; head: 10 cm; diameter: 0.9 cm; volume: 7.95 cm³) (Fig. 1) in climate-controlled chambers at 20 ± 2 °C with 65 % ± 5 % RH and in the dark to avoid any possible light-related behavioural bias. Tube heads were closed on both sides by caps with an opening in the center, covered with a 40 µl pouch of liquid 'Ap3'

artificial diet (Febvay et al., 1987) sealed between two layers of Parafilm®. Artificial diets were coupled to a PBSA-EO and a pure PBSA strip (30 mm × 5 mm) for the treatment and control sides, respectively (Fig. 1).

Tests were initiated by introducing five insects per replicate into the center of each tube through the shaft. Adult *A. pisum*, due to their larger size, were tested individually. Individuals were free to move toward either of the tubes based on olfactory cues, pass beyond the paper strip (with or without physical contact), and settle on the artificial diet. After 24 h, their position and condition were assessed. Measurements were considered valid for healthy individuals found on or within 1 cm of the artificial diet on either side. Each treatment was tested in 20 replicates (*i.e.*, 100 insects), or 100 replicates for adult *A. pisum*. Only tests with more than 90 % total valid measurements were considered for analysis. To confirm the absence of directional bias and validate the experimental setup, an equal number of blank controls were conducted using pure PBSA strips on both sides of the tube.

2.5. Concentration-response analysis of individual EOs and EO blends

Concentration-response analysis of EO-induced repellency in *A. pisum* N1 was performed following the same procedure for all EOs and EO blends. Each dataset included an untreated control and 6–8 test concentrations (0.003–10 EO wt%, chosen to ensure even coverage of the effect range), with data pooled across all replicates. From the count data, we calculated a repellency index (%) (RI) according to Eq. (1) (Macdonald et al., 2012):

$$RI = \frac{\sum_{i=1}^N (Nb.control) - \sum_{i=1}^N (Nb.treatment)}{\sum_{i=1}^N (Nb.control + Nb.treatment)} \times 100 \quad (1)$$

where *i* is the replicate number, *N* is the total number of replicates performed and *Nb.control* and *Nb.treatment* are the number of aphids settled on or within 1 cm of the artificial diets on the control and

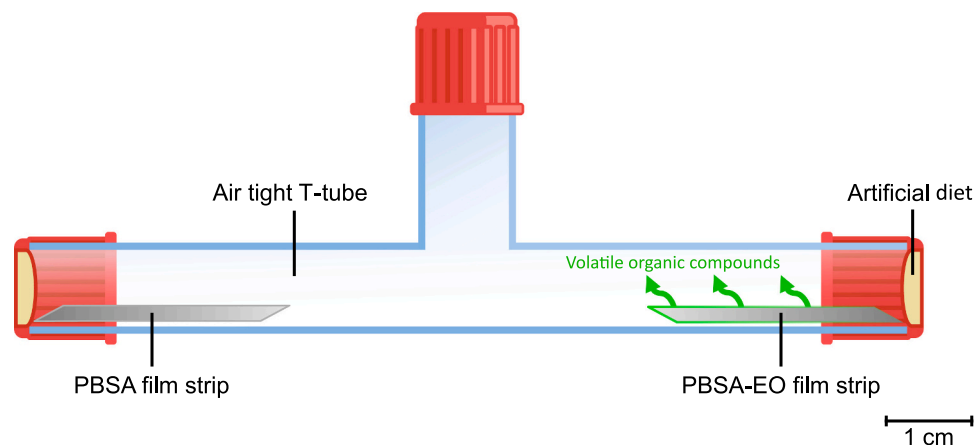


Fig. 1. Schematic representation of the *in vitro* dual-choice T-tube bioassay used to assess insect repellency. Insects are introduced through the central shaft and allowed to choose between the control arm (pure PBSA strip) and the treatment arm (PBSA-EO strip), both coupled to an artificial diet pouch. Insects were free to move and settle on either side. Choices were recorded after 24 h.

treatment sides, respectively. Positive values indicate repellency (RI \in [0,100]), while negative values denote attraction (RI \in [-100,0]).

In this section, we focused exclusively on EO concentrations eliciting repellent responses, which are biologically defined within the [0,100] interval. Accordingly, RI was modeled as a proportion response variable scaled to [0,1]. To reflect this bounded nature and ensure that ECx estimates correspond to concentrations causing $x\%$ of the maximal repellent effect, we fitted non-linear two-parameter quantal-type models with lower and upper asymptotes fixed at 0 and 1, respectively. Model fitting was performed using the *drm* function of the *drc* package (Ritz et al., 2015) in the R statistical software. Based on the Akaike Information Criterion (AIC), the asymmetric Weibull-1 distribution, given by Eq. (2) (Ritz, 2010), provided the best fit across all datasets:

$$F(x) = \exp(-\exp(b(\log(x) - \log(e)))) \quad (2)$$

where x is the EO concentration expressed as wt%, $F(x)$ corresponds to the RI, parameter b reflects the steepness of the concentration-response curve, and parameter e is the concentration where the inflection point of the curve is located.

ECx and their corresponding 95 % confidence intervals (95 % CI) were calculated from the fitted model using the *ED* function of the *drc* package (Ritz et al., 2015).

2.6. Calculation of predicted effect concentration for EO blends

We used two classical additivity models to predict the expected effect concentrations ECx_{mix} of EO blends, defined as the total concentration c_{mix} that causes a given quantitative effect $E(c_{mix}) = x\%$, with $x \in$ [0,100]. These models rely on distinct assumptions: the concentration addition (CA) model, also known as « Loewe additivity » (Cedergreen, 2014; Loewe and Muischnek, 1926), assumes that components share a similar mode of action and can substitute for one other proportionally to their potency, contributing jointly to a common effect in an additive manner. The independent action (IA) model, or « Bliss independence » (Bliss, 1939; Cedergreen, 2014), assumes that components act independently through distinct mechanisms, with their effects combining probabilistically on a common endpoint. Under both models, any ECx_{mix} value can be predicted, provided that the concentration-response relationships of the individual components are known (Faust et al., 2003).

Under the CA model, the predicted ECx_{mix} is calculated using Eq. (3):

$$ECx_{mix} = \left(\sum_{i=1}^n \frac{p_i}{ECx_i} \right)^{-1} \quad (3)$$

where p_i is the relative proportion of component i (from 1 to n) in the blend, and ECx_i is the concentration of that component that would produce the same effect level $x\%$ as the blend when applied individually. The ECx_i values were derived from the inverse of the component's concentration-response functions F_i^{-1} , such that $ECx_i = F_i^{-1}(x)$.

Under the IA model, the effect $E(c_{mix})$ of a blend is given by Eq. (4):

$$E(c_{mix}) = 1 - \prod_{i=1}^n (1 - E(c_i)) \quad (4)$$

where $E(c_i)$ represents the fractional effect caused by the individual component i at its concentration c_i in the blend. The values $E(c_i)$ were obtained from the individual concentration-response functions F_i such that $E(c_i) = F_i(c_i)$. Expressing c_i as the product of the component's relative proportion p_i and the blend total concentration c_{mix} (i.e., $c_i = p_i c_{mix}$), Eq. (4) can be rewritten as Eq. (5):

$$x\% = 1 - \prod_{i=1}^n (1 - F_i(p_i ECx_{mix})) \quad (5)$$

where ECx_{mix} corresponds to c_{mix} when $E(c_{mix})$ equals $x\%$ (Faust et al., 2003). The value of ECx_{mix} corresponding to a given effect level was determined by numerically solving Eq. (5) through a dichotomous iterative procedure.

Deviation of the experimentally observed ECx_{mix} from its predicted ECx_{mix} under CA or IA was quantified using the Model Deviation Ratio (MDR) (Belden et al., 2007), calculated according to Eq. (6):

$$\text{Model Deviation Ratio (MDR)} = \frac{ECx_{mix}(\text{predicted})}{ECx_{mix}(\text{observed})} \quad (6)$$

Blends with $0.5 \leq \text{MDR} \leq 2$ deviate less than two-fold from model predictions and were considered additive. $\text{MDR} > 2$ denoted synergistic interactions while $\text{MDR} < 0.5$ indicated antagonistic effects (Cedergreen, 2014).

2.7. Repellency spectrum analysis

For the repellency analysis of EOs against different sap-sucking hemipteran pests, insect preference was first tested against an equal distribution using exact binomial tests, under the null hypothesis that an insect would choose either side of the test tube with equal probability. Comparisons between insect species were then performed using a generalized linear model (GLM) with a binomial error structure and a log link function with the treatment and insect species set as explanatory variables. Multiple comparison analyses were performed by Tukey's test (Tukey, 1949) to investigate differences between species within treatments using the *emmeans* R package (Lenth, 2025). In both analyses, insect establishment on the control and treatment sides was coded as 'success' and 'failure', respectively. Therefore, the binary response variable was the average establishment rate on the control side ($ER_{control}$), calculated as shown in Eq. (7):

$$ER_{control} = \frac{\sum_{i=1}^N (Nb.control)}{\sum_{i=1}^N (Nb.control + Nb.treatment)} \quad (7)$$

where i is the replicate number and N is the total number of replicates. $Nb.control$ and $Nb.treatment$ refer to the number of insects established on the control and treatment sides, respectively.

While statistical analyses were performed on $ER_{control}$, results were linearly transformed into RI values using Eq. (8) to facilitate interpretation and graphical representation:

$$RI = (2ER_{control} - 1) \times 100 \quad (8)$$

3. Results

3.1. Repellency of individual essential oils against *Acyrtosiphon pisum*

To establish the baseline for EOs activity, we first modeled the concentration-response relationships of each EO against *A. pisum* N1. All three EOs exhibited concentration-dependent repellency within the

Table 2
Repellency of EOs incorporated into PBSA-based films against *A. pisum* N1.

EO	Model fit ^a		Parameters ^b		EC ₅₀ [CI] ^c	EC ₉₀ [CI] ^c
	χ^2 (df)	P	b	e		
<i>Cinnamomum cassia</i>	9.30 (5)	0.10	-0.39	0.20	0.509 [0.339 – 0.679]	64.518 [25.787 – n.r.]
<i>Mentha spicata</i>	9.29 (5)	0.10	-0.45	0.11	0.241 [0.174 – 0.308]	15.848 [7.620 – 32.960]
<i>Ocimum basilicum</i>	6.83 (5)	0.23	-0.43	0.15	0.351 [0.247 – 0.455]	28.497 [13.011 – 62.416]

^a χ^2 and P values from goodness-of-fit test (P > 0.05 indicate adequate fit).

^b Statistical estimates of the fitted Weibull-1 model parameters: b , curve steepness; e , inflection point.

^c EC₅₀, EC₉₀, effect concentrations for 50 % and 90 % repellency (wt%); CI, 95 % confidence intervals. n.r.: upper bound of the 95 % CI exceed the 100 % response, estimate not reliable.

same order of magnitude (Table 2). The Weibull-1 model asymmetric parameterization effectively captured the rapid increase in repellency from the lower limit of the curve at low concentrations, as well as a slowdown in the effect gain toward the upper limit at high concentrations (Fig. 2A–C). *M. spicata* exhibited the strongest effect, with an effective concentration inducing 50 % repellency (EC₅₀) of 0.241 wt% followed by *O. basilicum* and *C. cassia*, with EC₅₀ values of 0.351 and

0.509 wt%, respectively. Similarly, *M. spicata* had the lowest estimated concentration causing 90 % repellency (EC₉₀) of 15.848 wt%, followed by *O. basilicum* and *C. cassia*, with EC₉₀ values of 28.497 and 64.518 wt %, respectively. However, these estimates are approximate, as they were extrapolated beyond the tested concentration range and would require incorporating excessive amounts of EO into PBSA. This limitation highlights the need to improve EO efficiency in order to achieve

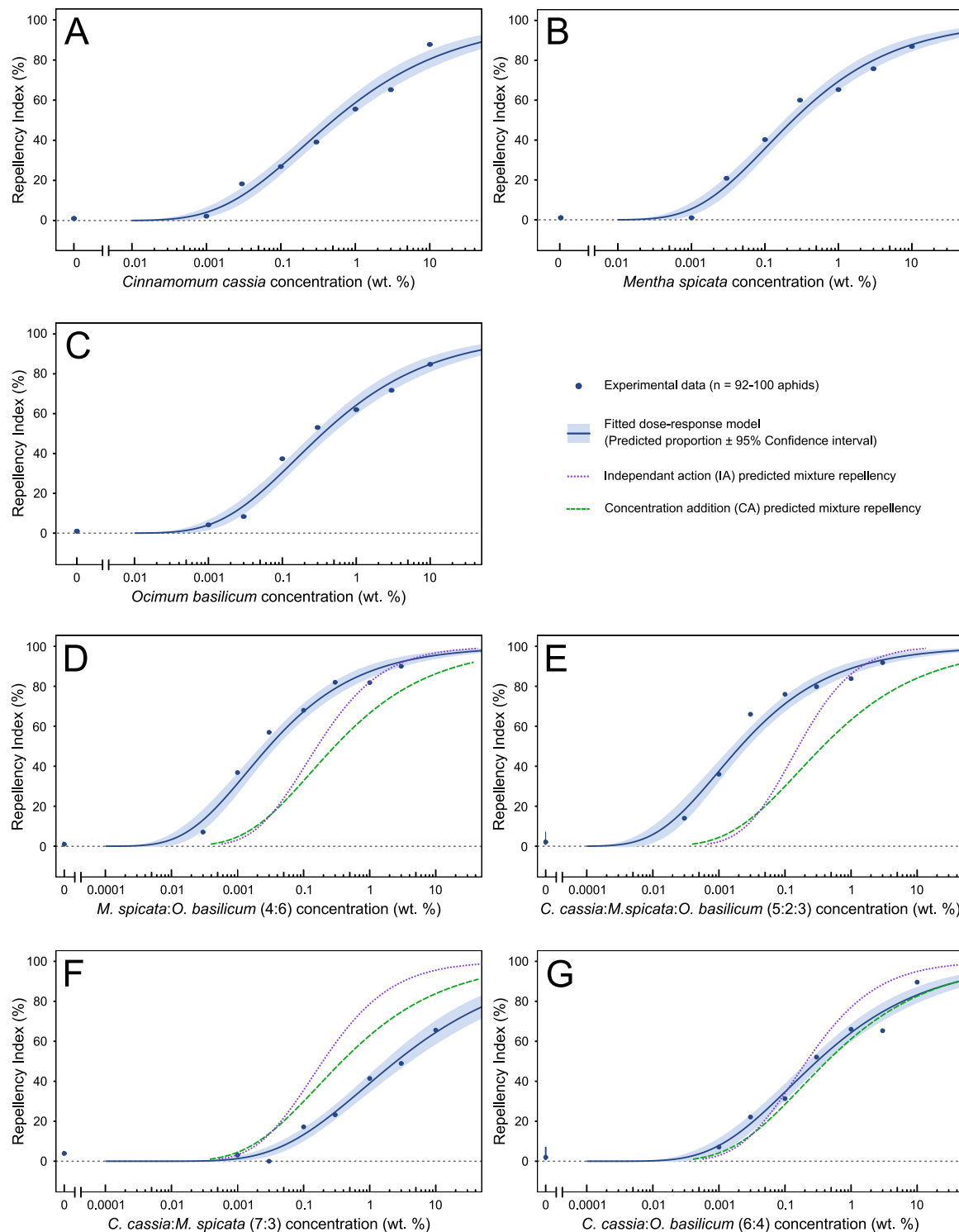


Fig. 2. Concentration-response relationship for the repellency of essential oils (EO) and EO mixtures incorporated into PBSA-based films against *A. pisum* N1. (A) *C. cassia* EO; (B) *M. spicata* EO; (C) *O. basilicum* EO; (D) *M. spicata*:*O. basilicum* (4:6) blend; (E) *C. cassia*:*M. spicata*:*O. basilicum* (5:2:3) blend; (F) *C. cassia*:*M. spicata* (7:3) blend; (G) *C. cassia*:*O. basilicum* (6:4) blend. The predicted repellency curves for the blends were generated by predicting the effective concentrations required to achieve effects ranging from 1 % to 99 % under the Concentration Addition (CA) and Independent Action (IA) models.

effective pest management.

3.2. Repellency of essential oil blends against *Acyrtosiphon pisum*

We next assessed the repellency of binary and ternary blends against *A. pisum* N1 by mixing the EOs in proportions relative to their EC₅₀ values, ensuring an equal expected contribution to the blend effect (Table 3, Fig. 2D-G). To evaluate interactions, we calculated the MDR, the ratio of observed to the predicted blend EC₅₀ under both CA and IA models, reflecting the fold difference in repellent potency compared to the additive expectation.

The 4:6 *M. spicata* and *O. basilicum* binary blend exhibited a synergistic effect, with an EC₅₀ of 0.030 wt% (8- and 10-fold more potent *M. spicata* and *O. basilicum* alone, respectively). MDR values > 2 (CA = 9.90; IA = 5.40), confirmed strong synergy, supported by a clear leftward horizontal shift of the fitted empirical concentration-response curve relative to CA and IA model predictions (Fig. 2D). The ternary blend outperformed all individual oils (up to a 24-fold increase in potency compared to *C. cassia* alone) showing the lowest EC₅₀ (0.021 wt%), with MDRs (CA = 17.86; IA = 7.62) corroborating a robust synergistic interaction (Fig. 2E). These two binary and ternary blends exhibited relatively low EC₉₀ values (1.700 and 1.263 wt%, respectively), emphasizing their enhanced potential for pest management. In contrast, the 7:3 *C. cassia* and *M. spicata* blend showed reduced efficacy with the highest EC₅₀ of 2.518 wt% (5- to 10-fold higher than those of its components, respectively), with MDRs < 0.5 (CA = 0.15; IA = 0.08) and a rightward shift of the fitted curve relative to predictions (Fig. 2F), consistent with antagonism. Due to this reduced efficiency, the EC₉₀ for this blend could not be estimated within the plausible concentration range. In comparison, the 6:4 *C. cassia* and *O. basilicum* combination yielded EC₅₀ and EC₉₀ values of 0.432 and 43.353 wt%, comparable to its components, with $0.5 \leq \text{MDR} \leq 2$ (CA = 1.42; IA = 0.70), indicative of additive behaviour (Fig. 2G).

Visual inspection of the additive blend (Fig. 2G) revealed that the CA model prediction closely matched the observed response across the entire concentration range. In contrast, the IA model provided a good fit up to 50 % repellency but tended to overestimate responses beyond this point. For all blends, whether synergy (Fig. 2D-E), antagonism (Fig. 2F) or additivity (Fig. 2G) was observed, fitted empirical concentration-response curves remained parallel to CA predictions, reflecting horizontal shifts in EC₅₀ without remarkable changes in slope. Together, these results reveal a spectrum of EO interactions, from strong synergy to clear antagonism, demonstrating that specific EO combinations can substantially enhance or compromise repellent efficacy compared to individual components.

3.3. Optimal *M. spicata* and *O. basilicum* ratio for repellency against *Acyrtosiphon pisum*

To refine the *M. spicata* and *O. basilicum* EO blend, we tested a series of ratios against *A. pisum* N1 (Table 4). EC₅₀ values were determined for each ratio, revealing minimal variation for blends close to pure EOs (e.g., 1:9 or 9:1 *M. spicata* and *O. basilicum* blends). A progressive decrease in EC₅₀ was observed for intermediate ratios, with the lowest value (0.021 wt%) obtained for the 7:3 ratio, corresponding to an 11- to 17-fold increase in repellent potency compared to the individual oils. A similar trend was observed for the EC₉₀, with the 7:3 ratio showing the lowest value (0.478 wt%). MDRs calculated under CA and IA models for each ratio followed an asymmetrical bell-shaped pattern (Table 4, Fig. 3). For blends close to the pure compounds, the CA model predicted near-additive effects (MDR ≈ 1), while the IA model suggested marginal antagonism ($0.5 < \text{MDR} < 1$). Synergistic interactions were detected under the CA model for ratios ranging from 2:8–5:5 (MDR > 2), and under the IA model for the 3:7 and 4:6 ratios. The 3:7 blend showed the strongest repellency and synergistic effect, with the lowest EC₅₀ and highest MDR values (CA = 14.71; IA = 8.19). As previously observed for EO blends, fitted empirical concentration-response curves for additive ratios closely matched CA predictions, providing a better fit than the IA model, while those for synergistic ratios remained parallel but shifted leftward relative to CA (Fig. S1). These findings highlight that the 3:7 *M. spicata*:*O. basilicum* blend provides a synergistic sweet spot, maximizing both efficacy and interaction strength, emphasizing the importance of ratio optimization in blend design. The estimated EC₉₀ for this ratio indicates that effective pest management can be achieved at relatively low concentrations.

3.4. Repellent efficacy of the 3:7 *M. spicata* and *O. basilicum* blend against hemipteran pests

The optimal 3:7 *M. spicata* and *O. basilicum* blend (composition provided in Supplementary Table S1) was further assessed against *A. pisum* adults and other hemipteran species to evaluate its repellent potential against key sap-feeding crop pests (Fig. 4). The tested concentrations corresponded to the EC₅₀ (0.021 wt%) and EC₉₀ (0.478 wt%) previously established for *A. pisum* N1. A significant interaction was found between treatment and insect species on repellency (binomial GLM, $\chi^2_{10} = 34.78$, $P < 0.001$). Statistical comparisons between insect species within each treatment are shown in Fig. 4. In all insect groups, blank control tests showed no significant deviation from equal distribution, indicating no directional bias and supporting the reliability of the experimental setup (exact binomial test, $P > 0.05$; Fig. 4A). When exposed to the EO blend, insects were either repelled (RI > 0) or showed no preference (RI ≈ 0); no cases of attraction (RI < 0) were observed. At the EC₅₀ concentration (Fig. 4B), the blend induced significant repellency against *A. pisum* N1 (RI = 49.49 %, $P < 0.001$), *A. pisum* adults (RI

Table 3
Observed and predicted repellency of EO blends incorporated into PBSA-based films against *A. pisum* N1.

EO blends ^a	Model fit ^b		Parameters ^c		Observed EC ₅₀ [CI] ^d	Observed EC ₉₀ [CI] ^d	Predicted EC ₅₀ ^e		MDR ^f	
	χ^2 (df)	P	b	e			CA	IA	CA	IA
Cc + Ms (7:3)	9.30 (5)	0.10	-0.33	0.831	2.518 [1.344 – 3.691]	n.e.	0.382	0.196	0.15	0.08
Cc + Ob (6:4)	9.07 (5)	0.11	-0.37	0.116	0.305 [0.206 – 0.403]	43.353 [17.534 – 82.589]	0.432	0.213	1.42	0.70
Ms + Ob (4:6)	10.37 (5)	0.07	-0.47	0.014	0.030 [0.022 – 0.038]	1.700 [0.874 – 3.303]	0.297	0.162	9.90	5.40
Cc + Ms + Ob (5:2:3)	9.98 (5)	0.76	-0.57	0.010	0.021 [0.015 – 0.027]	1.263 [0.647 – 2.463]	0.375	0.160	17.86	7.62

^a Cc, *Cinnamomum cassia*; Ms, *Mentha spicata*, Ob, *Ocimum basilicum*. Blend ratios (v/v) are indicated within brackets.

^b χ^2 and P values from goodness-of-fit test (P > 0.05 indicate adequate fit).

^c Statistical estimates of the fitted Weibull-1 model parameters: b, curve steepness; e, inflection point.

^d EC₅₀, EC₉₀, effect concentrations for 50 % and 90 % repellency (wt%); CI, 95 % confidence intervals; n.e., not estimable: the predicted EC₉₀ is outside the plausible range.

^e CA, concentration addition; IA, independent action.

^f MDR, model deviation ratio comparing observed and predicted EC50 values (MDR > 2, synergy; 0.5–2, additivity; < 0.5, antagonism).

Table 4

Observed and predicted repellency of *M. spicata* and *O. basilicum* EO blends incorporated into PBSA-based films against *A. pisum* N1.

<i>M. spicata</i> : <i>O. basilicum</i> ratio	Model fit ^a		Parameters ^b		Observed EC ₅₀ [CI] ^c	Observed EC ₉₀ [CI] ^c	Predicted EC ₅₀ ^d		MDR ^e	
	χ^2 (df)	<i>P</i>	<i>b</i>	<i>e</i>			CA	IA	CA	IA
0:10	6.83 (5)	0.23	-0.43	0.149	0.351 [0.247 – 0.455]	28.497 [13.011 – 62.416]	-	-	-	-
1:9	5.87 (4)	0.21	-0.55	0.173	0.337 [0.247 – 0.427]	10.510 [5.377 – 20.542]	0.336	0.232	1.00	0.69
2:8	2.45 (4)	0.65	-0.45	0.048	0.107 [0.077 – 0.138]	7.065 [3.176 – 15.718]	0.322	0.191	3.01	1.79
3:7	4.05 (5)	0.54	-0.60	0.012	0.021 [0.016 – 0.026]	0.478 [0.289 – 0.791]	0.309	0.172	14.71	8.19
4:6	10.37 (5)	0.07	-0.47	0.014	0.030 [0.022 – 0.038]	1.700 [0.875 – 3.303]	0.297	0.162	9.90	5.40
5:5	6.29 (4)	0.18	-0.38	0.036	0.096 [0.064 – 0.128]	14.171 [5.024 – 39.970]	0.286	0.157	2.98	1.64
6:4	5.29 (4)	0.26	-0.37	0.058	0.158 [0.103 – 0.212]	25.991 [8.769 – 77.041]	0.275	0.157	1.74	0.99
7:3	5.23 (4)	0.26	-0.50	0.104	0.217 [0.158 – 0.276]	9.519 [4.581 – 19.779]	0.266	0.161	1.23	0.74
8:2	3.42 (4)	0.49	-0.36	0.084	0.235 [0.150 – 0.321]	46.258 [14.406 – n.r.]	0.257	0.172	1.09	0.73
9:1	9.50 (5)	0.09	-0.43	0.099	0.229 [0.164 – 0.295]	17.431 [8.208 – 37.018]	0.249	0.195	1.09	0.85
10:0	9.29 (5)	0.10	-0.45	0.107	0.241 [0.174 – 0.308]	15.848 [7.620 – 32.960]	-	-	-	-

^a χ^2 and *P* values from goodness-of-fit test (*P* > 0.05 indicate adequate fit).

^b Statistical estimates of the fitted Weibull-1 model parameters: *b*, curve steepness; *e*, inflection point.

^c EC₅₀, EC₉₀, effect concentrations for 50 % and 90 % repellency (wt%); CI, 95 % confidence intervals. n.r.: upper bound of the 95 % CI exceed the 100 % response, estimate not reliable.

^d CA, concentration addition; IA, independent action.

^e MDR, model deviation ratio comparing observed and predicted EC₅₀ values (MDR > 2, synergy; 0.5–2, additivity; < 0.5, antagonism)

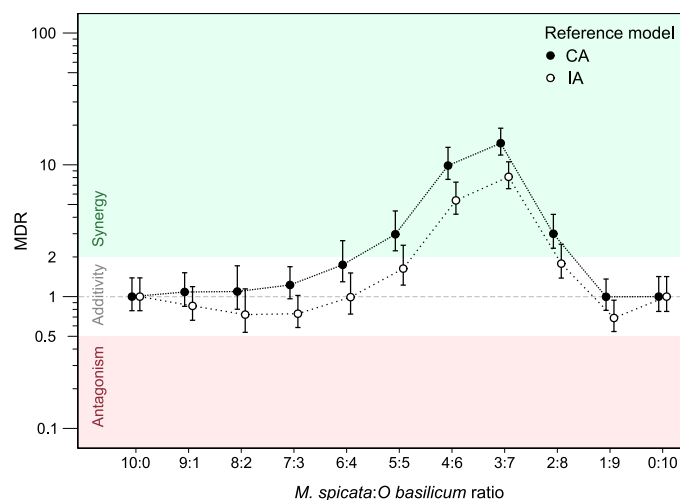


Fig. 3. Model Deviation Ratio (MDR) comparing observed and predicted EC₅₀ values for *M. spicata* and *O. basilicum* EO blends incorporated into PBSA-based films and tested for repellency against *A. pisum* N1, under Concentration Addition (CA) and Independent Action (IA) additivity models. EC₅₀: effective concentration (EO wt%) causing 50 % repellency. Blends with $0.5 \leq \text{MDR} \leq 2$ deviate less than two-fold from model predictions and are considered additive (white area). MDR > 2 denotes synergistic interactions (green area), while MDR < 0.5 indicates antagonistic effects (red area). MDR lower and upper limits are derived from the 95 % confidence intervals of the observed EC₅₀, representing the range of possible values accounting for the variability in the experimental data.

= 31.31 %, *P* = 0.0024), and *B. tabaci* (RI = 29.79 %, *P* = 0.0051), but had no detectable effect on *M. persicae*, *R. padi*, or *S. maydis* (RI ≈ 0 %; exact binomial test, *P* > 0.05). At the EC₉₀ concentration (Fig. 4C), significant repellency was observed for all species except *S. maydis* (RI = 4.08 %, *P* = 0.52). These results show the broad-spectrum potential of the optimized 3:7 blend, with the strongest effect recorded in *A. pisum* N1 (RI = 94.00 %, *P* < 0.001), followed by *A. pisum* adults (RI = 77.78 %, *P* < 0.001), and intermediate responses in the remaining species.

4. Discussion

Compounds extracted from plants have long been considered for pest management due to their relatively low toxicity to humans and non-target organisms. Among them, EOs are complex blends of VOCs with well-documented repellent activity against insect pests (Isman, 2020; Pavela and Benelli, 2016; Regnault-Roger et al., 2012). In this study, we evaluated the repellency of PBSA films containing EOs against the pea aphid *A. pisum*. To optimize bioactivity, we investigated synergistic interactions by comparing individual EOs with their binary and ternary

blends, refining blend ratios, and assessing their efficacy against other sap-feeding hemipteran pests. This systematic approach allowed us to identify specific EO combinations with enhanced potency and broad-spectrum activity, providing a foundation for the rational development of bio-based repellents.

Consistent with previous findings, the three EOs tested in this study, *C. cassia*, *M. spicata*, and *O. basilicum*, each exhibited repellency against *A. pisum* (Lacotte et al., 2023, 2025). Their chemical composition, previously characterized, was used to train a machine-learning model designed to evaluate the contribution of key individual compounds to overall repellency (Lacotte et al., 2023). Major constituents included *trans*-cinnamaldehyde in *C. cassia*, carvone in *M. spicata*, and estragole in *O. basilicum* (Lacotte et al., 2023). Although these compounds remained largely preserved during film incorporation, partial conversion of *trans*-cinnamaldehyde into benzaldehyde was observed during the extrusion process (Lacotte et al., 2025). Nevertheless, the overall effect of an EO often exceeds the sum of its individual components (Jiang et al., 2009; Kim et al., 2016; Tak et al., 2016), a phenomenon commonly attributed to the synergistic potential of EO-derived constituents (Feng et al., 2020; Pavela, 2015, 2014; Tak et al., 2016). These insights

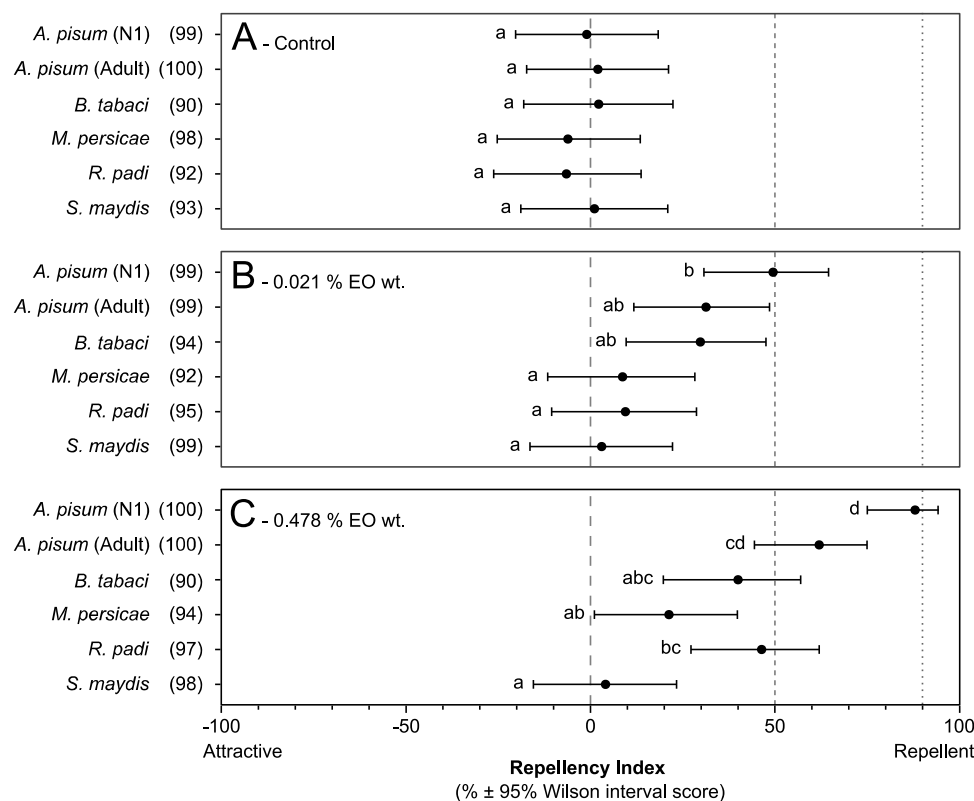


Fig. 4. Repellent activity of a 3:7 *M. spicata* and *O. basilicum* EO blend incorporated into PBSA-based films against sap-feeding pests in dual-choice experiments (% \pm 95 % Wilson interval score). (A) Control (no EO), (B) 0.021 EO wt% (*A. pisum* N1 EC₅₀), (C) 0.478 EO wt% (*A. pisum* N1 EC₉₀). Long-dashed, short-dashed and dotted vertical lines represent the 0, 50 and 90 % repellency thresholds, respectively. Brackets indicate the number of valid measures obtained per species and treatment. Letters next to data points indicate statistical groups within treatments determined by Tukey's multiple-comparison tests ($P < 0.05$).

reinforce the importance of experimentally testing EO blends rather than relying solely on the activity of major constituents, given the emergent properties of synergistic interactions.

In insects, odor detection occurs primarily through their antennae, via the sensilla that house olfactory sensory neurons (OSNs). VOCs penetrate the cuticle through small pores, dissolve in the sensillar lymph, and are transported by carrier proteins to the dendritic membrane receptors of OSNs. The resulting ligand-receptor interactions trigger a neuronal signal that drives behavioural responses, while odorant-degrading enzymes (ODEs) contribute to signal clearance (Leal, 2013; Pelosi et al., 2014). Current evidence suggests that EO compounds can interact with a family of carrier proteins known as odorant-binding proteins (OBPs) (Abendroth et al., 2023; Fuentes-Lopez et al., 2024; Gao et al., 2021; Pan et al., 2024; Qu et al., 2022). While the mode of action (MoA) underlying EO-induced repellency remains unclear, it is plausible that compounds, whether from the same or different oils, share a common MoA or act through distinct or unrelated mechanisms that nonetheless converge on the same behavioural outcome.

Despite their repellent potential, the three EOs tested in this study failed to achieve effective pest management against *A. pisum* (RI ≥ 90 %) at concentrations up to 10 wt%, with an estimated EC₉₀ exceeding this value. To enhance overall repellency, we tested a series of binary and ternary blends. The 4:6 *M. spicata*:*O. basilicum* and 5:2:3 *C. cassia*:*M. spicata*:*O. basilicum* combinations resulted in improved efficacy, with 8- to 24-fold increases in repellent potency compared to individual EOs, indicating potential synergistic interactions. Our results also suggested cases of additivity (e.g., 6:4 *C. cassia*:*O. basilicum*) and antagonism (e.g., 7:3 *C. cassia*:*M. spicata*), consistent with previous studies on EO-based repellents (Aboelhadid et al., 2022; Boukraa et al., 2022; Hategekimana and Erler, 2020; Huang et al., 2020; Miloudi et al., 2024; Noosidum et al., 2014; Peach et al., 2019; Soonwera et al., 2024; Tak and

Isman, 2017b). Interactions were quantified through the MDR, comparing the observed EC₅₀ values with those predicted under reference models of additivity. Given the uncertainty surrounding the precise repellent MoA of EOs, both the CA and IA models were used as conceptual frameworks (Bliss, 1939; Cedergreen, 2014; Loewe and Muischnek, 1926). The CA model assumes that EOs act through a similar or convergent MoA, with their effects combining in a concentration-dependent and interchangeable manner, whereas the IA model presumes independent and dissimilar MoAs. While neither model fully accounts for the intrinsic complexity of EOs, their application provides a structured approach to interpreting blend effects (Cedergreen, 2014). Both models led to similar qualitative conclusions. For additive blends, predictions from the CA model aligned more closely with empirical observations than those from the IA model, supporting the CA hypothesis of a shared or overlapping MoA among EOs. In blends exhibiting synergy or antagonism, we consistently observed horizontal shifts in empirical concentration-response curves (i.e., EC₅₀ changes), without substantial changes in slope or shape compared to CA-based predictions, contrary to IA. These shifts were reflected in MDR values that exceeded the conservative synergy threshold of 2 or fell below 0.5 for antagonism (Cedergreen, 2014). This pattern suggests that interactions modulate overall potency without fundamentally altering response dynamics, in a manner still compatible with the CA assumption.

Interestingly, the three major constituents of the EOs tested in this work, carvone, estragole, and *trans*-cinnamaldehyde, have been shown to interact both with each other and with various EO-derived compounds, producing synergistic or antagonistic insecticidal effects depending on the target species (Pavela, 2015, 2014). For instance, the carvone:estragole blend was synergistic against the mosquito *Culex pipiens* L. but antagonistic in the cotton leafworm *Spodoptera littoralis*

(Boisduval), while the opposite was observed for the carvone:trans-cinnamaldehyde blend (Pavela, 2015, 2014). Such variability, combined with the chemical complexity of EOs, further limits the predictive value of major components alone. While these findings may help interpret some of the non-additive outcomes observed in our blends, they also underscore the need for organism-specific validation in *A. pisum*.

Synergy between EOs likely arises from multiple mechanisms, ranging from the physicochemical properties of the blend to biological interactions at the insect level. These may include well-documented pharmacological processes, wherein compounds modulate each other's uptake, transport, metabolism and excretion, or jointly act on multiple targets (Wagner and Ulrich-Merzenich, 2009). Physicochemical interactions between EOs or EO-derived compounds, such as increased volatility and solubility, or reduced surface tension, may enhance environmental availability or promote uptake upon contact with the insect, for instance, through greater cuticular penetration (Kim et al., 2021; Tak and Isman, 2017a, 2015; Yoon and Tak, 2023). Beyond improved uptake, synergy may also result from greater target accessibility due to interference with detoxication systems. This has been demonstrated with EO-derived compounds that either inhibit (Moustafa et al., 2023; You et al., 2023) or saturate (Scalerandi et al., 2018) key ODEs such as cytochromes P450, glutathione S-transferases, or carboxylesterases, allowing co-occurring active compounds to persist longer. Some EO compounds may also enhance neural responses either by acting synergistically on complementary neurophysiological targets. This has been observed in the bedbug *Cimex lectularius* L. with carvacrol, thymol, and eugenol (Gaire et al., 2020). Alternatively, certain compounds may potentiate the activity of others. For instance, in the tobacco cutworm *Spodoptera litura* (Fabricius), mandarin oil and its major compound limonene boosted the neuroexcitation induced by basil oil and its main constituents, estragole and linalool, despite being inactive on their own (Kim et al., 2021). These findings illustrate how synergy can emerge from multi-level interactions, from enhanced cuticle penetration to convergent neurostimulation, reinforcing the value of blend-based strategies in EO repellent design.

Critically, the outcome of EO interactions is not fixed but depends on the relative proportions of components within the blend. This was exemplified by the *M. spicata* and *O. basilicum* combination tested against *A. pisum*, which shifted from additivity to synergy depending on the ratio, with the 3:7 blend maximizing both repellency and the strength of the synergistic interaction. Similar ratio-dependent interaction patterns between EOs or EO-derived compounds have been reported in other insect models (Feng et al., 2020; Huang et al., 2020; Pavela and Novák, 2024; Scalerandi et al., 2018). In one striking example, adjusting the camphene/bornyl acetate ratio in a binary blend resulted in additive, synergistic, or even antagonistic insecticidal effects against the red flour beetle *Tribolium castaneum* Herbst (Feng et al., 2020), highlighting the crucial role of proportions in defining the interaction type. This underscores that the success of EO blends hinges not only on the identity of the components but also on their relative proportions, making blend optimization a crucial step in repellent development.

Importantly, the optimized 3:7 *M. spicata* and *O. basilicum* blend reduced the concentration required for effective repellency (RI \geq 90 %) to a relatively low level. This was experimentally confirmed in *A. pisum* N1, as application of the estimated EC₉₀ of 0.473 wt% resulted in 94 % repellency, indicating that effective pest management can be achieved at economically viable concentrations (Lacotte et al., 2023) that are compatible with practical integration into PBSA matrix by the extrusion process, commonly used in industry (Lacotte et al., 2025).

The repellency of the 3:7 *M. spicata* and *O. basilicum* blend varied across the insect species tested and, to a lesser extent, among *A. pisum* life stages, indicating both species- and age-specific sensitivity. At both concentrations tested, corresponding to the EC₅₀ and EC₉₀ values determined for *A. pisum* N1 nymphs, *A. pisum* adults were slightly less repelled than nymphs, while other hemipteran species (*B. tabaci*,

M. persicae, *R. padi*) exhibited markedly lower levels of repellency or no repellency at all (*S. maydis*). The species- and age-dependent variability observed here is consistent with a broader trend reported across insects in a recent meta-analysis (Arena et al., 2025), which showed that insect behavioural responses to EOs vary according to their taxonomic order and developmental stage. This highlights the importance of evaluating EO blends according to relevant pest targets and life stages. These results indicate that, in addition to being effective against *A. pisum*, this EO blend could also offer partial protection against species that are known to be polyphagous pests such as *M. persicae* (van Emden and Harrington, 2017) and *B. tabaci* (Stansly et al., 2010) on Fabaceae or oligophagous aphids like *R. padi* (van Emden and Harrington, 2017) on other crops.

Biopolymer-based matrices, such as PBSA films, have been proposed as carriers for active compounds in agricultural systems (Lacotte et al., 2025), and may benefit from the synergistic EO combinations explored in this study, providing a holistic and environmentally responsible solution for modern agriculture. However, translating these findings into practical solutions requires addressing key limitations. This study was conducted under controlled laboratory conditions, focused on a single behavioural endpoint that is repellency, and did not evaluate long-term efficacy, non-target effects, or field performance. Moreover, the inherent variability in EO composition across batches, origins, or storage conditions remains a limitation to regulatory approval and formulation standardization (Isman, 2016), especially when, as shown in the study, the component ratio appears critical for efficacy. These formulation challenges must be addressed to enable real-world application, particularly given the demonstrated sensitivity of repellent efficacy to component ratios and EO batch variability, notably by finding industrial-scale sources that are stable both quantitatively and qualitatively.

In our previous work, we have shown that the EOs used in this study can be incorporated into PBSA matrix due to their good thermal and chemical stability, while maintaining its thermal and physical properties and also having a positive impact on the ductility of the polymer materials produced (Lacotte et al., 2025). The EO-PBSA repellent plastics thus obtained could be adapted to various agricultural applications, such as mulching films, greenhouse covers, and insect nets in order to support agroecosystem sustainability. The feasibility of this approach is supported by previous studies that have successfully developed insect repellent films and nets using other polymers combined with insecticides or repellents (Dáder et al., 2014; Finkenstadt and Tisserat, 2010; Seven et al., 2019; Kim et al., 2020). The persistence of EO-PBSA repellent activity under field conditions, necessary to determine the long-term effectiveness of this material, is currently under investigation.

The empirical approach used in this study provides a basis for designing synergistic EO blends. These blends could be further improved through the development of predictive tools such as mechanistic interaction models or machine learning approaches based on chemical composition and biological data. Most importantly, our results open up new perspectives for the design of bioactive EO-based biodegradable and partially biobased plastics for crop protection. This approach not only offers a natural substitute for conventional pesticides in pest management, but also reduces reliance on petroleum-based plastics by promoting biodegradable and bio-based alternatives, thereby supporting a holistic and eco-friendly solution for modern agriculture.

CRediT authorship contribution statement

Sylvain Benhamou: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Pedro Da Silva:** Writing – review & editing, Writing – original draft, Supervision, Resources, Project administration, Funding acquisition. **Sébastien Livi:** Writing – review & editing, Writing – original draft, Supervision, Resources, Project administration, Funding acquisition, Conceptualization. **Federica Calevro:** Writing – review & editing, Resources, Conceptualization.

Laurence Mouton: Writing – review & editing, Resources, Conceptualization. **Garance Lapetoule:** Writing – original draft, Resources, Investigation, Conceptualization. **Zaï nab Belgaidi:** Writing – original draft, Resources, Investigation, Conceptualization. **Isabelle Rahioui:** Writing – original draft, Resources, Investigation, Conceptualization. **Chrystèle Jouve:** Writing – original draft, Resources, Investigation, Conceptualization.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Pedro Da Silva reports financial support was provided by French National Research Agency. Pedro Da Silva reports financial support was provided by Astragal SNA. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.indcrop.2025.121791](https://doi.org/10.1016/j.indcrop.2025.121791).

Data Availability

Data will be made available on request.

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