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ORIGINAL PAPER

In natura and nanoencapsulated essential oils from *Xylopia aromatica* reduce oviposition of *Bemisia tabaci* in *Phaseolus vulgaris*

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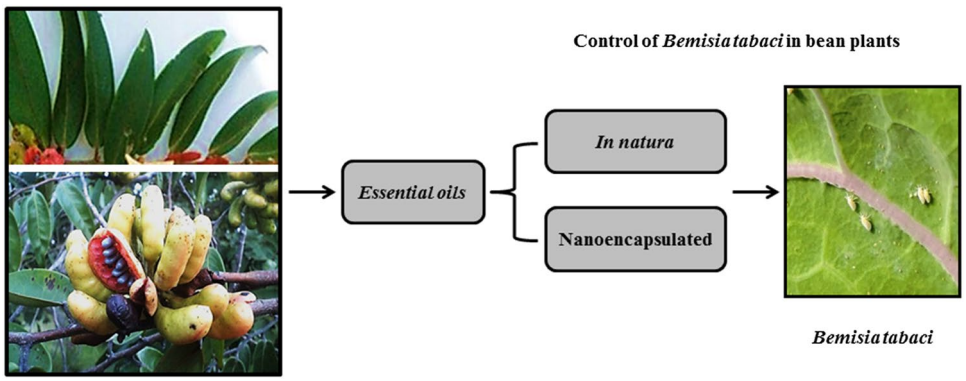
Abstract

Bemisia tabaci is an agricultural pest of worldwide distribution that causes serious damage to several crops. It is of crucial importance to control this pest, especially for large-scale production. Accordingly, formulations based on essential oils of pesticidal action are potentially promising in the agricultural sector. Additionally, the nanoencapsulation of these bioactive compounds promotes their protection from environmental degradation and prolongs their biological activity. Here, we develop PCL (poly-ε-caprolactone) nanoparticles containing essential oils of *Xylopia aromatica* leaves and fruits and evaluate their insecticidal effect in *B. tabaci* Middle East Asia Minor 1 biotype B. The average yields of essential oils from leaves and fruits of *X. aromatica* were 0.05 and 0.80%, respectively. The major compounds in the essential oil of leaves were bicyclogermacrene (44.80%), α-pinene (8.23%) and β-pinene (7.75%) while in fruits were α-pinene (35.40%), β-phellandrene (31.05%) and β-pinene (22.51%). The PCL nanoparticles containing the essential oils exhibited encapsulation efficiency of 95% and particle diameter smaller than 500 nm. Biodegradable nanospheres substantially protected the essential oils from accelerated degradation caused by UV light and also prevented possible phytotoxic activity of the in natura essential oil from leaves of *X. aromatica* in high concentrations, probably due to the gradual release. In natura and nanoencapsulated essential oils from leaves and fruits decreased (up to 98%) the oviposition of *B. tabaci* in common bean leaves. Our results indicate that both in natura and nanoencapsulated oils of *X. aromatica* may potentially be used as alternative to the chemical control of *B. tabaci*.

Graphic abstract



Keywords Nanotechnology · Natural products · Pesticide · Poly-ε-caprolactone · Whitefly

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Key message

- In natura and nanoparticles containing essential oils from both fruits and leaves of *Xylopia aromatica* were developed as alternative to the chemical control of *Bemisia tabaci*.
- Nanoprecipitation was efficient for the preparation of PCL nanoparticles containing essential oils.
- Encapsulation is seemingly an effective protector of the active ingredients of the essential oils from both fruits and leaves from the solar light-induced degradation.
- Encapsulation was efficient in preventing phytotoxic activity of essential oils on *Phaseolus vulgaris* leaves.
- In natura and nanoencapsulated essential from leaves and fruits reduce the oviposition of *B. tabaci* in common bean leaves.

Introduction

The widespread use of pesticides not only causes environmental damage (Moreira et al. 2002), but has also a social impact, posing the health of both consumers and manipulators at risk and leading to the emergence of new pests and pathogens (Oliveira-Silva et al. 2000; Rambow et al. 2014). Accordingly, the growing concern on the usage of synthetic pesticides in agriculture has led to the development of a new field of research focused on alternative forms of pest control (Fazolin et al. 2007; Andrade et al. 2013). An alternative method for the control of agricultural pests that is gaining prominence is the use of botanical products, including plant extracts and essential oils with insecticidal action, mostly due to their greater selectivity, low toxicity to mammals and low residual effect (Costa et al. 2014; Ikbal and Pavela 2019).

Botanical products hold the promise of being less harmful to the environment, and as such many studies have demonstrated insecticidal, bactericidal and fungicidal among other biological activities (Bakkali et al. 2008; Kyarimpa et al. 2014). Furthermore, botanical products contain a variety of active compounds that act synergistically and have attractive, dissuasive or repellent characteristics (Navarro-Silva et al. 2009). These natural bioactive plant compounds are produced by plants as part of their so-called secondary metabolism, with the essential oils being prominent among these bioactive compounds (Koul et al. 2008). Essential oils can therefore be potentially used for the development of effective methods of pest control. This would allow the reduction in the amount of synthetic insecticides used and the chemical contamination, simultaneously preserving the environment and the quality of food, making it a suitable practice for sustainable agriculture (Kéita et al. 2001; Roel 2001; Marangoni et al. 2012).

Bemisia tabaci Middle East Asia Minor 1 (MEAM1) (= formerly biotype B) (Hemiptera: Aleyrodidae), commonly known as whitefly, is a polyphagous insect that infests several crops, including common beans, sweet potatoes, melons, cotton, tomatoes, soybeans and ornamental plants (Oliveira et al. 2001; Palumbo et al. 2001). Damage to the host plant may be caused directly by feeding on phloem sap and indirectly by the large amounts of sticky honeydew that promotes *Capnodium* sooty mold that, in turn, causes cosmetic injury and impairs photosynthesis (Stansly and Natwick 2010). In addition, *B. tabaci* is vector of more than 300 species of virus (Navas-Castillo et al. 2011; Gilbertson et al. 2015). In some crops, including common bean *Phaseolus vulgaris* L., the resulting viral diseases are growth-limiting factors and may cause total crop loss (Horowitz and Ishaaya 2014; Faria et al. 2016). In Brazil, the propagation in the population of the *B. tabaci* MEAM1 has been favored by the agricultural system (with three growing seasons), the presence of a large diversity of differing host plants and the tropical climate (Quintela et al. 2016). The control of this insect is primarily achieved through synthetic insecticides that have already resulted in the selection of resistant populations in many regions of the world (Baldin et al. 2013; Basit et al. 2013; Horowitz and Ishaaya 2014). Thus, the implementation of alternative methods of pest control in association with other management practices that meet the requirements of agronomic efficiency, toxicological safety and low environmental impact is driving research toward the use of natural products (Yatagai 1997; Isman 2006; Ateyyat et al. 2009).

Annonaceae plants are seemingly of importance in this field due to their insecticidal effect, mainly given to the presence of acetogenins, which are a promising class of insecticidal agents (Castillo-Sanchéz et al. 2010; Krisnki et al. 2014). Plants of the genus *Xylopia*, which belong to the Annonaceae family, represent a promising source of bioactive substances once they display several biological activities, such as antimicrobial, cytotoxic, acaricidal, antibacterial, anti-inflammatory, antifungal, fumigant, antitumor and hypolipidemic properties (Pontes et al. 2007; Ferraz et al. 2013; Oliveira et al. 2014). *Xylopia aromatica* is widely distributed in the Brazilian Savanna being almost exclusively found in tropical regions. Chemical analysis of this species, popularly known as monkey pepper, has already proved the insecticidal action of extracted substances against *Aedes aegypti* (Dip.) and *Dipetalogaster maxima* (Hem.) (Rodrigues et al. 2006; Coelho et al. 2009). Essential oils from leaves and fruits of *X. aromatica* contain higher abundance of terpenic substances such as limonene, α and β-pinene and

β-myrene (Lago et al. 2003; Stashenko et al. 2004). These compounds exhibit insecticidal activity, as well as the essential oils that contain them (Viegas Jr 2003; Moreira et al. 2013). This fact apart, the impacts of essential oils from *X. aromatica* on agricultural pests including *B. tabaci* have not been reported to date.

Due to the high volatility of the essential oils and the relatively low amount produced by the plants coupled with its biological instability under the action of oxygen, light and moderate temperature, the application of essential oils under field conditions remains a highly complicated task (Nascimento et al. 2007). This problem can be attenuated through nanotechnology that promotes the gradual release and photoprotection of the active principle, thus optimizing the pest control system. The nanoencapsulation of essential oils implies in the consequent reduction in not only the amount of insecticide applied, but also in the toxicity and undesirable effects in nontarget organisms, besides directing the effect of the active principle (Kumar et al. 2014).

The present study was conducted as part of our ongoing bioprospecting project aiming to discover potential insecticidal action produced by native plants from the Brazilian "Cerrado" (the dense forest-like savanna vegetation—Christofoli et al. 2015; Pereira et al. 2018), due to its wide distribution in the Brazilian "Cerrado," occurring from open areas of grasslands to savanna *stricto* sensu (Durigan et al. 2004; Camargo et al. 2011). As a result, the *Xylopia* genus represents a promising source of bioactive substances (Nguemtchouin et al. 2010). Essential oils from leaves and fruits of *X. aromatica* majoritarian compounds such as limonene, α and β-pinene and β-myrcene have been identified (Lago et al. 2003; Stashenko et al. 2004). These compounds have shown insecticidal activity (Viegas Jr. 2003; Moreira et al. 2013), suggesting its potential for control of *B. tabaci*. We therefore evaluated the chemical composition of the essential oils extracted from both leaves and fruits of *X. aromatica* and further investigated the effect of both in natura and nanoencapsulated essential oils on oviposition of *B. tabaci* on common bean leaves, *Phaseolus vulgaris* L., aiming at the development of new insecticides to be applied in integrated pest management systems.

Materials and methods

Materials

Poly-ε-(caprolactone) (PCL) (average molecular weight of 45,000), sorbitan 60 monostearate (Span®60) and polysorbate 80 (Tween®80) were purchased from Sigma-Aldrich (St. Louis, USA). HPLC-grade solvents were purchased from J.T. Baker (Ecatepec, Mexico). Ultra-pure water (18 MΩ-cm) was obtained by reverse osmosis using a Milli-Q system (Millipore, Bedford, MA, USA).

Sample collection

Leaves and fruits of *X. aromatic* were collected in the municipality of Iporá, state of Goiás, Brazil (16° 21′ 19.3″ S and 51° 1′ 12″ W), in a border area between riparian forest and marsh. After collection, the fresh material was moistened and placed in plastic bags until the essential oil extraction. The voucher specimen (number 472) was deposited at the Laboratory of Ecology and Vegetable Systematics of the Instituto Federal de Educação, Ciência e Tecnologia Goiano (IF Goiano)—*Campus* Rio Verde, Brazil.

Extraction of the essential oils

Essential oils from the leaves and fruits of *X. aromatica* were extracted by hydrodistillation in a Clevenger apparatus for 4 and 2 h, respectively. The organic phase was removed with dichloromethane and dried with anhydrous sodium sulfate. Next, part of the solvent was removed on a rotary evaporator. The vials containing the essential oils were stored under refrigeration at 4 °C until further analysis.

Chemical analysis of the essential oils

Chemical analysis was performed in a gas chromatograph coupled to a mass spectrometer (Shimadzu GC-17A) equipped with a DB—5 capillary column (30 m × 0.25 mm) film = 0.25 µm, IE 70 eV and a helium carrier gas. Temperature was maintained at 60° followed by an increase of 3 °C per min until reaching 280 °C. The identification of the chemical constituents of the essential oils was based on the retention index (Kovat's index) calculated with respect to the retention time of a homologous series of n-alkanes (Adams 2007), and on the fragmentation pattern observed in each of the mass spectra was also observed. Both data sets were compared with the equipment database (NIST08 mass spectral library) and to results found in the literature.

Preparation of nanoparticle

Nanospheres (NS) were prepared via nanoprecipitation of the preformed polymer (interfacial deposition/solvent displacement) (Fessi et al. 1989). First, an organic phase was prepared containing the biopolymer [PCL (poly-ε-(caprolactone)], the active principle (essential oil), Span® 60 [surfactant of low HLE (hydrophilic–lipophilic equilibrium)] and acetone. Further, this phase was poured under constant agitation over an aqueous phase containing Tween 80 (surfactant of high HLE) and was maintained for 10 min under magnetic stirring. The organic solvent and part of

the water were then removed under reduced pressure using a rotary evaporator at 40 °C, and the final volume was adjusted. Table 1 presents different formulations prepared and used here.

Characterization of nanoparticles

Quantification of encapsulated essential oil

The essential oil content in the nanoparticles was quantified using ultraviolet–visible (UV/VIS) spectroscopy at 231 and 232 nm for essential oils from the leaves and fruits of *X. aromatica*, respectively (Maji et al. 2007; Paula et al. 2011; Abreu et al. 2012). Calibration curves (Eqs. 1, 2) were obtained using linear regression, using concentrations of 0.01, 0.02, 0.04, 0.06, 0.08 e 1.0 mg mL^{-1} e 0.01, 0.02, 0.03, 0.04, 0.05 e 0.06 mg mL^{-1} for essential oils from the leaves and fruits of *X. aromatica*, respectively:

$$y = 16.681x + 0.02 \quad (1)$$

$$y = 54.414x + 0.029 \quad (2)$$

where y is the band absorption and x is the concentration (mg mL^{-1}) of essential oil in the solution. The fit was linear ($n = 3$) in the working range, and the linear correlation coefficient (r^2) was 0.9968 and 1.0 for essential oils from the leaves and fruits of *X. aromatica*, respectively. All the points from the calibration curves were obtained in triplicate.

Determination of encapsulation efficiency (EE%) of the essential oil

The amount of encapsulated essential oil was determined by UV/VIS, using the filtration–centrifugation technique, as described previously (Christofoli et al. 2015).

pH, particle diameter and zeta potential

The determinations of particle diameter (PD), pH and zeta potential (ZP) of the suspension were performed immediately after preparation of the nanoparticles. The pH of the colloidal solution was determined using a potentiometer (B474 Micronal, São Paulo, BR). Analyses of PD, PI (polydispersity index) and ZP using Zeta Sizer Nano Z-S (Malvern) were performed in a 1:10 dilution of each colloidal suspension in ultra-pure water.

Morphological evaluation of nanoparticle suspensions

The morphology of the nanospheres was evaluated by samples dilution in distilled water at 5% (v/v), allowing the colloidal suspension to be evaporated directly on the glass slide. After the complete water evaporation, samples were gold-sputtered and examined using scanning electron microscopy (SEM).

Analysis of UV light-accelerated degradation

Analyses of UV light-accelerated degradation of both in natura and nanoencapsulated essential oils were conducted in an UV chamber with a set of two special light bulbs that simulated the radiation in the UV-A and UV-B spectra with wavelengths of 400–320 and 320–280 nm, respectively. The system was maintained exactly as previously described using the same established time intervals (0.0, 0.5, 1, 2, 3, 5, 6, 7, 9 and 12 h) to assess the stability of the encapsulated substances (Christofoli et al. 2015).

Controlled release analysis of nanoparticles

The nanoparticle release profile was obtained using the inverse dialysis technique (Magenheim and Benita 1991). Briefly, a 10 mL volume of the nanoparticle suspension was added to 100 mL of aqueous solution containing 0.5% Tween 80. In this suspension, ten dialysis bags were added (MM 1200D cellulose membrane, Sigma-Aldrich Chemistry, Sintra, Portugal), each of them contained 1.0 mL of 0.5% Tween 80 aqueous. The system was maintained under magnetic agitation in a thermostatically controlled bath at room temperature (25.0 ± 2.0 °C). At the previously established time intervals (0.0, 0.5, 1, 2, 3, 6, 9, 12, 24, 48 and 72 h), the dialysis bags were removed from the release medium,

Table 1 Formulation of PCL nanospheres prepared with essential oils obtained from *X. aromatica* fruits (NSF) and leaves (NSL)

Formulations	Components					
	PCL (mg)	Span 60 (mg)	Essential oil (mg)	Acetone (mL)	Tween80 (mg)	Water (mL)
NSF1	50	100	50	10	100	20
NSF2	50	100	250	10	100	20
NSF3	150	100	50	10	100	20
NSF4	150	100	250	10	100	20
NSL	50	100	250	10	100	20

and the essential oil released was quantified by UV/VIS spectroscopy.

Biological assay

Bemisia tabaci were kindly provided by Embrapa Rice and Beans, Brazilian Agricultural Research Corporation, located in Santo Antônio de Goiás, Brazil. *B. tabaci* used in the experiments was identified as Middle East Asia Minor 1 (MEAM1) (formerly biotype B) by molecular gene sequence markers from mtDNA cytochrome oxidase I (mtCOI) (Quintela et al. 2016). Whiteflies were grown in a gauze cage (1.8 m height × 0.8 m width × 1.5 m length) with fine mesh fabric (50 mesh) and fed with cabbage (*Brassica oleraceae*) and soybean (*Glycine max* L. Merril).

All experiments were conducted in a greenhouse located on the farm of the Federal Institute of Education, Science and Technology Goiano Campus Iporá (16° 21′ 19.3″ S and 51° 1′ 12″ W), at summer, rainy season, from November to December 2013. Throughout the experiments, the average temperature was 26.2°C (minimum 22.2 °C, and maximum 34.1 °C), whereas relative humidity was 80.9% (from 69.5 to 92.5%) measured by data loggers AZ8829, AZ Instrument Corp. Ltd., Hong Kong. Common bean (*Phaseolus vulgaris* L. cv. Ouro Vermelho) was sowed in 1.5-L pots containing a mixture of soil and composted manure (2:1, v/v). Each pot received a single seed, and forty-day-old plants were used for the experiments. Plants were watered regularly and fertilized as required.

The effect of the essential oils *in natura X. aromatica* fruits and leaves at concentrations of 0.1, 0.25, 0.5, 1 and 2% was tested on the repellency of adult whiteflies in a no-choice and a free-choice test. Nanoencapsulated *X. aromatica* fruits and leaves at the same concentrations were also assessed on the repellency of adult whiteflies in both no-choice and free-choice tests. Aqueous solution of 1% (w/v) Tween® 80 was used to prepare all in natura concentrations. Three controls were additionally used in the assays: a negative control with ultra-pure water, an aqueous solution of 1% Tween 80 and a positive control with 1% pyriproxyfen (Tiger®), a juvenile hormone analog that causes a suppression of embryogenesis and a disruption of metamorphosis and adult formation (Ishaaya and Horowitz 1995; Qureshi et al. 2009). Each leaf was sprayed with 250 µL of each treatment on both the adaxial and abaxial surfaces using a microsprayer (0.3 mm needle, Paasche® H-series airbrush) coupled to a vacuum pump.

In the no-choice experiments, treated plants were kept individually inside each cage (0.3 m wide × 0.5 m long) made of a very fine mesh nylon tissue. Two hundred adults of *B. tabaci* were released into each cage. Although we did not precisely quantify the number of male and female adults, it is usually observed that whiteflies are distributed in pairs, and females are bigger than males. Thus, during the collection of the adults we took care to separate the whitefly couples, ensuring that we used similar proportions between male and female. After 24 h of oviposition, the leaves and insects were removed and the eggs were counted with a Medilux MDL-F stereoscopic microscope (40× magnification).

In the free-choice experiments, all treated plants were placed together in the same cage and females could choose the plant to oviposit. The plants were arranged equidistantly inside a cage (1.8 m high × 0.8 m wide × 1.5 m long) covered with voile cloth. The cage proportions generated one rectangle, and adults prefer to stay on cage extremity, specifically at the top and corner of the cage. Given to that, the experimental unities were equidistantly disposed from each other inside of the cage, in an oval shape. Then, 2600 adults were released in the very center of the cage. After 24 h, the eggs were counted as described for the no-choice experiment.

In both no-choice and free-choice experiments, one single plant was used and four leaves were removed to count the number of eggs, totalizing four replicates for each treatment. All experiments were repeated three times. Treatment efficiency ($E\%$) of the in natura and nanoencapsulated essential oils was determined by using the Abbott`s formula: $E\% = (1 - T/C)*100$, where T is the number of eggs in the tested treatment and C is the number of eggs in the control treatment (Abbott 1925).

The experimental design was completely randomized for all experiments. Curves for the number of eggs/leaf were adjusted according to nonparametric models and compared using the Wilcoxon rank sum test at $P \leq 0.05$. To estimate the lethal concentration (LC_{50}) for the treatments, generalized nonlinear models (log-logistic, log-normal or Weibull) were fitted and values were compared by the overlap of their 95% confidence intervals (95% CI).

Results

Chemical analysis of the essential oil

Through chemical analyses by GC–MS a total of 36 compounds were identified in the essential oils from *X. aromatica* leaves (Table 2), and seven were identified in fruits (Table 3). All the compounds found in the essential oils, their respective RT, relative percentages and RI are additionally presented (Tables 2, 3).

Nanospheres containing essential oil from *X. aromatica* leaves and fruits

The formulations exhibited pH around 5 (Table 4). Encapsulation efficiency (EE%) ranged between 86.0 and 99.0%. Particle diameter (DP) of prepared nanospheres showed that

Table 2 Chemical constituents of the essential oil from the *X. aromatic* leaves

Peak	RT (min)[a]	Compounds	Area (%) GC–MS	RI exp.[b]	RI lit.[c]
1	7.821	α-pinene	8.23	934	932
2	8.918	Sabinene	1.18	946	969
3	9.047	β-pinene	7.75	955	974
4	9.363	β-myrcene	1.46	978	988
5	9.804	α-felandrene	1.20	1006	1002
6	10.269	p-cymene	0.16	1025	1020
7	10.395	Limonene	0.95	1030	1024
8	10.433	β-phellandrene	14.69	1031	1029
9	10.550	β-*trans*-ocimene	0.19	1036	1044
10	11.107	γ-terpinene	0.30	1059	1054
11	14.200	α-terpineol	0.50	1197	1186
12	16.892	δ-elemeno	1.03	1337	1335
13	17.163	α-cubebene	0.24	1352	1345
14	17.614	Geranylacetate	1.07	1376	1379
15	17.716	α-copaene	2.03	1382	1374
16	17.933	β-elemene	0.37	1394	1389
17	18.257	α-gurjunene	1.65	1413	1409
18	18.527	E-caryophyllene	0.38	1428	1417
19	18.852	Aromadendrene	2.77	1448	1439
20	19.017	Allo-aromadendrene	1.07	1457	1458
21	19.225	Dehydro-aromadendrene	1.02	1470	1460
22	19.569	d-germacrene	2.17	1490	1482
23	19.726	Viridiflorene	1.43	1499	1496
24	19.815	Bicyclogermacrene	44.80	1505	1500
25	20.132	δ-cadinene	0.57	1525	1522
26	21.142	Spathulenol	1.68	1587	1577
27	21.293	Viridiflorol	1.11	1597	1592

[a]RT, retention time

[b]RI exp., retention index calculated in the DB-5 column

[c]RI lit., retention index described in the literature (Adams 2007)

Table 3 Chemical constituents of the essential oil from the *X. aromatic* fruits

Peak	RT (min)[a]	Compounds	Area (%) GC–MS	RI exp.[b]	RI lit.[c]
1	10.007	α-pinene	35.40	931	932
2	11.802	Sabinene	2.99	971	969
3	11.894	β-pinene	22.51	973	974
4	12.697	β-myrcene	2.41	991	988
5	13.186	δ-3-carene	1.17	1002	1008
6	14.201	p-cymene	3.10	1024	1022
7	14.37	β-phellandrene	31.05	1027	1025
8	22.026	Cryptone	1.37	1181	1183

[a]RT, retention time

[b]RI exp., retention index calculated in the DB-5 column

[c]RI lit., retention index described in the literature(Adams 2007)

all samples were in the nanometric range with a mean diameter < 159 nm and exhibited a narrow polydispersity index (PDI < 0.30). Similar results have been previously reported for PLC nanoparticles containing different drugs (Schaffazick et al. 2002; Cazo et al. 2012). Zeta potential (ZP) results showed that both nanosphere from fruit (NSF) and nanosphere from leaves (NSL) formulations exhibited a negative charge with values ranging from − 25.64 to − 17.70 mV, typically observed in these types of systems (Ahlin et al. 2002).

To further evaluate the morphology of the nanospheres, we used scanning electron microscopy (SEM) to obtain information about their shape, surface and diameter. Figure 1 reveals the presence of spherical nanospheres containing essential oil of *X. aromatica* fruits and leaves. Similar results were previously observed by Christofoli et al. (2015) who obtained smooth, monodisperse nanospheres with diameters between 400 and 500 nm using the PCL polymer as a matrix for encapsulating essential oils of *Z. rhoifolium* leaves. Additionally, Pinto et al. (2016) obtained

Table 4 Encapsulation efficiency (EE%), pH, particle diameter (PD), zeta potential (ZP) and polydispersity index (PDI) for various PCL nanosphere formulations of essential oil from *X. aromatica* fruits (NSF) and leaves (NSL)

Formulations	pH[a]	PD(nm)[a]	ZP(mV)[a]	PDI[a]	EE%[a]
NSF1	5.79 ± 0.1	101.62 ± 4.30	− 22.15 ± 1.1	0.20 ± 0.1	97 ± 1.1
NSF2	5.12 ± 0.3	124.57 ± 17.8	− 22.51 ± 8.8	0.27 ± 0.1	99 ± 0.0
NSF3	5.51 ± 0.1	116.61 ± 16.7	− 25.64 ± 3.7	0.21 ± 0.1	99 ± 0.0
NSF4	5,26 ± 0.2	159.32 ± 25.3	− 25.16 ± 8.8	0.30 ± 0.1	99 ± 0.0
NSL	5.46 ± 0.4	120.00 ± 2.77	− 17.70 ± 3.9	0.13 ± 0.1	86 ± 3.1

[a]Values are presented as means of replicates ± standard deviation (SD). $n = 3$ biological replicates

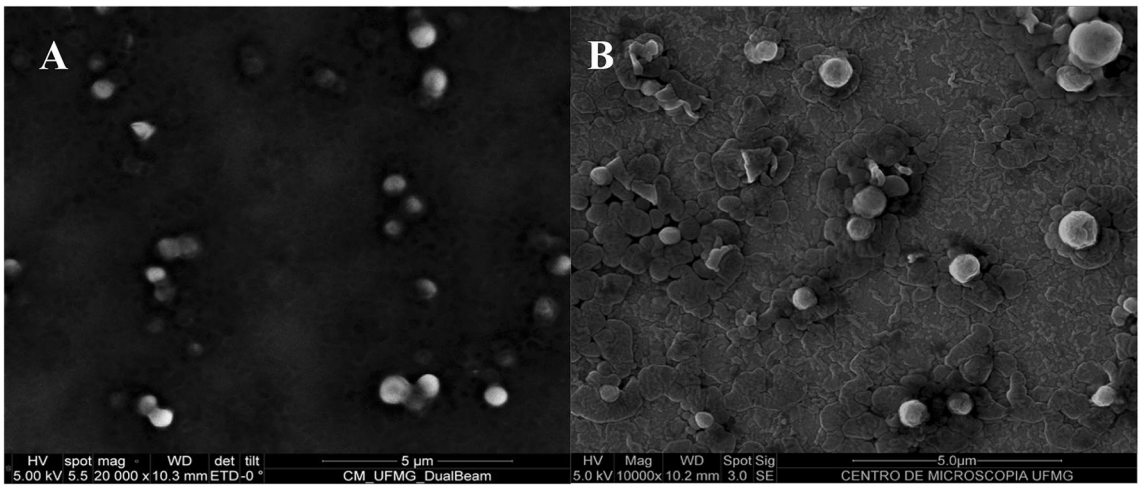

Fig. 1 SEM photomicrographs of PCL polymeric nanospheres loaded with essential oil of *X. aromatica*: **a** fruits (NSF) and **b** leaves (NSL)

PCL particles containing *Lippia sidoides* essential oils of spherical and crack-free forms, with an average diameter of 173.6 nm, which are in good agreement with our data.

Analysis of UV light-accelerated degradation

The in natura essential oil of leaves suffered 94% photodegradation, whereas the nanospheres degradation was only 42.51% after 12 h of light exposure (Figs. 2, 3). The control was maintained in the same environment and protected against light, exhibiting 3.58% degradation. This fact notwithstanding, the in natura essential oil of fruits, nanospheres and its counterpart control suffered 91%, 66% and 3% photodegradation, respectively, after 12 h of exposure.

Analysis of in vitro release

We observed a fast initial release profile of the nanospheres of essential oils from *X. aromatica* leaves and fruits, followed by a slow release after 12 h (Figs. 4, 5).

Biological assays

The repellence effect of the essential oil from *X. aromatica* leaves and fruits against adult whiteflies was evaluated through oviposition on bean leaves for 24 h in free-choice and no-choice tests.

The number of eggs reduced with in natura essential oil concentrations from leaves and fruits for both free-choice

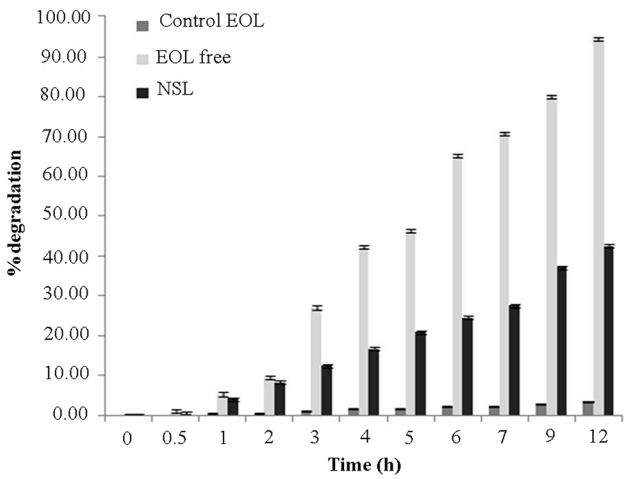

Fig. 2 Degradation assay of essential oil of leaves from *X. aromatica in natura* (EOL free), nanospheres (NSL) and control (control EOL) exposed to UV light. Each bar represents an average value ± standard deviation ($n = 3$)

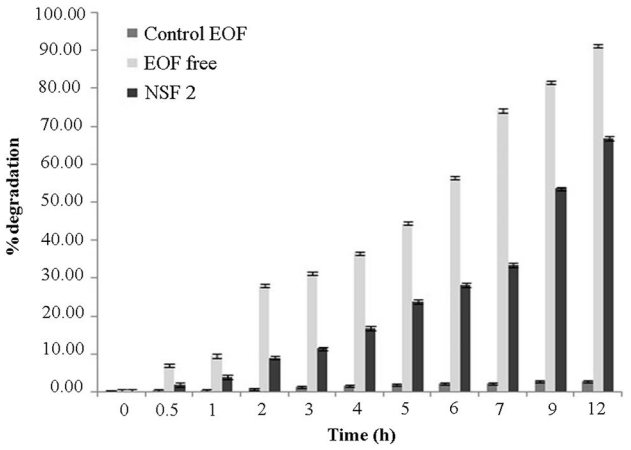

Fig. 3 Degradation assay of essential oil of fruits from *X. aromatica in natura* (EOF free), nanospheres (NSF2) and control (control EOF) exposed to UV light. Each bar represents an average value ± standard deviation ($n = 3$)

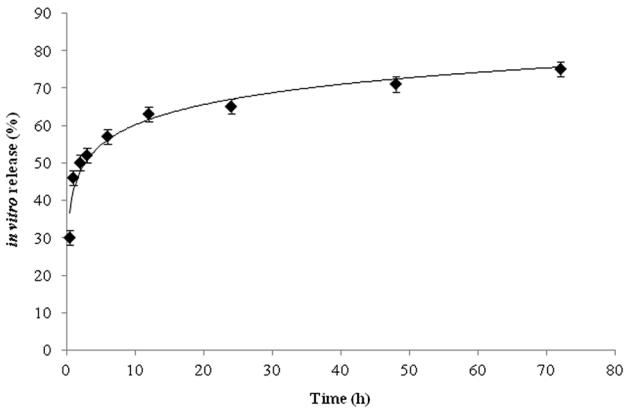

Fig. 4 In vitro release profile of the essential oil of leaves *X. aromatica*-loaded nanospheres colloidal dispersion (NSL) using the dialysis pocket diffusion technique. Each data point represents the average of three different NS batches

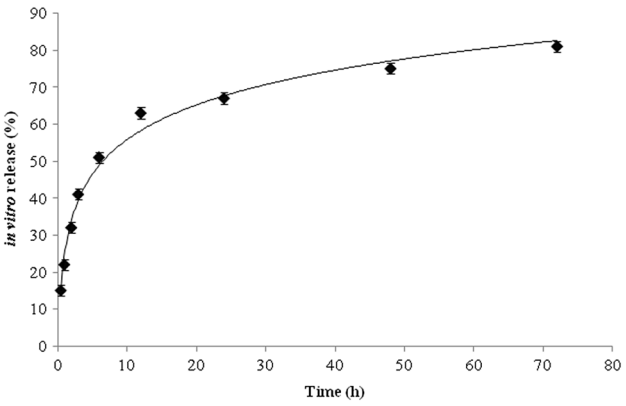

Fig. 5 In vitro release profile of the essential oil of fruits *X. aromatica*-loaded nanospheres colloidal dispersion (NSF2) using the dialysis pocket diffusion technique. Each data point represents the average of three different NS batches

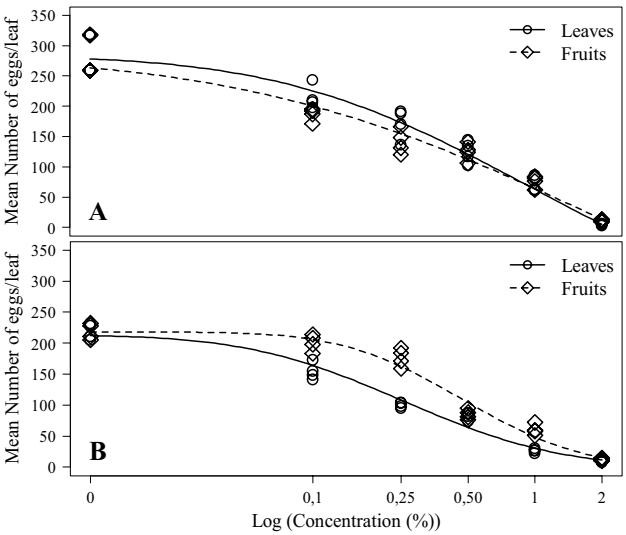

Fig. 6 Mean number of eggs/leaf 24 h after treatment of bean leaves with in natura essential oil from *X. aromatica* leaves and fruits in no-choice (**a**) and free-choice (**b**) tests

and no-choice tests and a log-normal model provided the best fit for all of them ($R^2 = 95.98\%$) (Fig. 6a, b). When the two curves were compared using Wilcoxon's test, there were no differences between the in natura essential oils from fruit and leaves in no-choice ($P = 0.8831$) and free-choice tests ($P = 0.3527$).

The number of eggs did not differ between the insecticide pyriproxyfen (mean of 288.2 ± 33.4) and water control (mean of 300.2 ± 32.1) in no-choice test ($P = 0.1361$). Similarly, in the free-choice test, no difference in the number of eggs was observed between pyriproxyfen (mean of 219.0 ± 11.3) and water control (mean of 224.7 ± 6.6) ($P = 0.8781$).

In natura oil was highly efficient in reducing the number of whitefly eggs. Treatment efficiencies ranged from 25.6 to 98.4% at concentrations of 0.1 to 2% for both tests (Table 5). Thus, the lethal concentrations to reduced 50% of the eggs number (CL_{50}) were low: ≤ 0.27% for no-choice and ≤ 0.46% for free-choice tests (Table 6). In the free-choice test, the LC_{50} of leaves was significantly lower to that of the fruit (Table 6).

Similar to in natura oil, the number of eggs reduced with the increase in the concentrations of PCL nanospheres containing essential oil from *X. aromatica* leaves and fruits (Fig. 7 a, b). A log-logistic and Weibull model provided the best fit for no-choice ($R^2 = 98.56\%$) and free-choice (97.51%) tests. According to Wilcoxon's nonparametric test, the two curves were similar for no-choice ($P = 0.6991$) and free-choice ($P = 0.9042$) tests.

Table 5 Treatment efficiency (*E*%)[a] after spray of bean leaves with essential oil in natura and nanoencapsulated from *X. aromatica* leaves and fruits in no-choice and free-choice tests

Treatments	In natura (*E*%)		Nanoencapsulated (*E*%)	
	No-choice	Free-choice	No-choice	Free-choice
Pyriproxifen 1%	6.9	8.7	6.4	5.1
Leaves				
0.1%	25.6	29.6	20.3	18.0
0.25%	40.6	54.3	43.6	49.4
0.5%	56.8	62.4	63.8	56.9
1%	76.4	88.0	76.3	74.1
2%	98.4	95.8	91.0	87.7
Fruits				
0.1%	35.4	8.3	23.3	15.4
0.25%	51.0	19.4	38.8	49.8
0.5%	56.7	61.2	60.8	57.7
1%	73.7	72.6	74.9	74.3
2%	96.1	94.3	87.90	87.5

[a]Treatment efficiency calculated by Abbott's formula. For in natura leaves and fruit treatments, the comparison control was Tween® 80 1%. For nanosphere treatments, the comparison control was empty nanospheres. For the insecticide pyriproxyfen, the comparison control was water

Table 6 Median lethal concentrations (%) (LC_{50}) for number of eggs after spray of bean leaves with in natura and nanoencapsulated essential oil from *X. aromatica* leaves and fruits in no-choice and free-choice tests

Essential oil	Model	LC_{50} (CI 95%)
In natura (no-choice test)		
Leaves	Weibull	0.24 (0.26–0.29)
Fruits		0.23 (0.22–0.26)
In natura (free-choice test)		
Leaves	Log-logistic	0.23 (0.21–0.24)
Fruits		0.46 (0.44–0.49)
Nanoencapsulated (no-choice test)		
Leaves	Log-normal	0.32 (0.29–0.34)
Fruits		0.34 (0.31–0.37)
Nanoencapsulated (free-choice test)		
Leaves	Weibull	0.31 (0.28–0.33)
Fruits		0.32 (0.29–0.34)

In the no-choice and free-choice tests, the number of eggs in the water control (means of 125.5 ± 6.1 and 128.5 ± 10.0) was similar to the empty nanosphere control (means of 132.2 ± 4.6 and 123.5 ± 6.2), respectively. These results demonstrate that the substances used in nanoencapsulation process did not interfere with the oviposition behavior of *B. tabaci.* Again, pyriproxyfen did not reduce the number of the whitefly eggs/leaf compared with the water control. In the no-choice and free-choice tests, the mean number of eggs/leaf was 123.7 ± 5.9 and 122.0 ± 7.9, respectively.

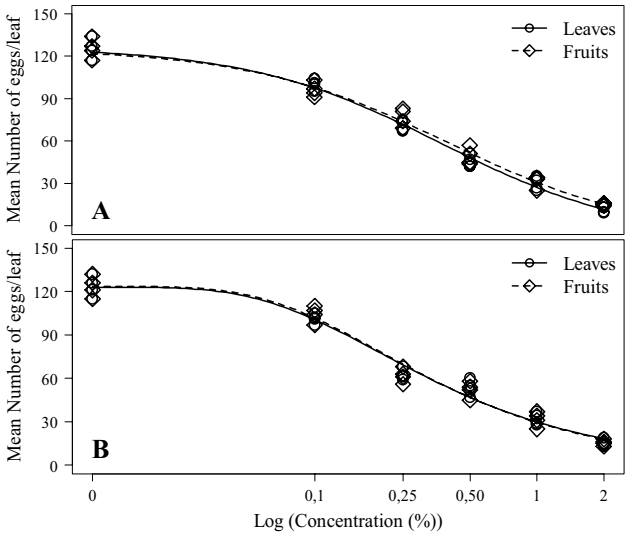

Fig. 7 Mean number of eggs/leaf 24 h after treatment of bean leaves with nanoencapsulated essential oil from *X. aromatica* leaves and fruits in no-choice (**a**) and free-choice (**b**) tests

Treatment efficiencies by Abbott formula for nanoencapsulated essential oil ranged from 15.4 to 87.90 at concentrations of 0.1 to 2% for no-choice and free-choice tests (Table 5). The LC_{50}'s was ≤ 0.31% and similar for all treatments (Table 6). These results indicate that low concentrations of this oil (formulated or not) reduce the number of eggs of *B. tabaci*.

The essential oil in natura from leaves at the highest concentrations (2%) caused phytotoxicity on bean leaves. Early drying of bean leaves was observed after 48 h of oil spray at 2% (Fig. 8a). However, the oil from leaves, formulated in polymer nanoparticles, did not cause any phytotoxic symptoms when sprayed at 2% on bean leaves (Fig. 8b). This result suggests that the controlled release of the active compounds by the nanoparticles attenuated the toxicity of the oil. Others studies also observed phytotoxic on dry bean leaves treated with different oils at concentrations > 2% (Pinheiro et al. 2009; Marques et al. 2014). In addition, essential oils from different plants have been extensively tested to assess their herbicidal activities due to their phytotoxic activities (Amri et al. 2013).

Discussion

The essential oil of *X. aromatica* leaves, collected in the Brazilian "Cerrado" (central region), contained a total of 36 compounds, with bicyclogermacrene (44.80%), α-pinene (8.23%) and β-pinene (7.75%) as the major compounds. The major compounds found in our study were also present in the

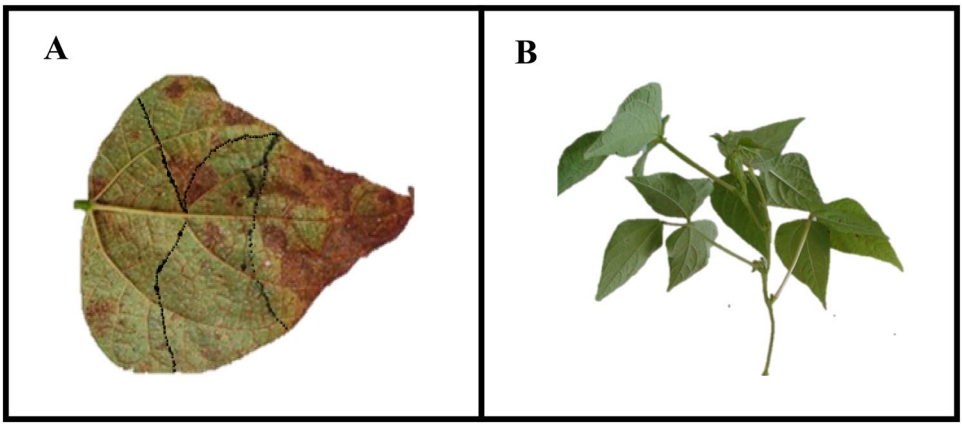

Fig. 8 **a** Phytotoxic on bean leaves caused by in natura essential oil of *X. aromatica* from leaves at 2% concentration, 48 h after application. **b** Common beans plant treated with poly-ε-caprolactone (PCL) nanospheres containing essential oil from leaves of *X. aromatica* at 2% concentration, 48 h after application

essential oil of *X. aromatica* leaves collected in the Amazon region (north of Brazil) although in different concentrations: bicyclogermacrene (36%), α-pinene (3.40%) and β-pinene (2.30%) among 27 others compounds (Andrade et al. 2004). In addition, the essential oil of *X. aromatica* leaves collected in the Atlantic Forest (Southeast of Brazil) also had α-pinene (26.10%), bicyclogermacrene (20.40%) and β-pinene (19%) as the major compounds among 12 others (Lago et al. 2003). These findings indicate a significative variation in the concentration of the major compounds and in the amount of the components of this essential oil depending upon the growth region.

Differently, only seven compounds were observed in the fruits of the essential oil of *X. aromatica*: α-pinene (35.40%) followed by β-phellandrene (31.05%), β-pinene (22.51%), p-cymene (3.10%), sabinene (2.99%), β-myrcene (2.41%), cryptone (1.37%) and δ-3-carene (1.17%). In sharp contrast, 33 compounds were observed in the essential oil of *X. aromatic* fruits collected in the Amazon region and limonene (36.40%), *α*-pinene (19.20%) and *β*-pinene (13.30%) were identified as major compounds. Notably, these compounds comprised more than 68% of all the chemical compounds of the essential oils from fruits (Andrade et al. 2004).

The wide variation in the chemical composition of these three specimens of *X. aromatica* growing in different regions (Cerrado, Amazon and Atlantic Forest) is most likely due to a large exogenous influence on the secondary metabolite production. As such, each plant, even from the same species, but from a biome that has different climatic conditions, soil, water availability and light incidence would display significant differences in the content and chemical composition of their essential oils (Pavarini et al. 2012). Accordingly, it has been found that temperature may have a significant effect on chemical composition as such on insecticidal activity of essential oils (Hansted et al. 1994; Pavela and Sedlák 2018). Further understanding of the relationship between environmental conditions and essential oil chemical composition appears to be of fundamental importance for practical recommendation of botanical insecticides based on essential oils, specifically from *X. aromatica*.

Different nanosphere formulations containing essential oil of fruits (NSF1, NSF2, NSF3, NSF4) and leaves (NSL) from *X. aromatica* were prepared in an attempt to obtain higher encapsulation efficiency (EE%) combined with greater system stability. In the prepared formulations, no free essential oil was observed, and an apparently stable and homogeneous suspension was obtained. The amount of poly-ε-caprolactone (PCL) did not affect loading capacity of the nanospheres. Colloidal suspensions with increasing amounts of essential oils were prepared to determine the maximum particle loading capacity. Colloidal suspensions with essential oils higher than 250 mg showed the presence of essential oils on the surface, indicating that the maximum loading level for the formulation had been reached.

The high instability of essential oils in the presence of light, heat and humidity leads to numerous degradation reactions. This makes difficult to preserve essential oils and also affects the feasibility of its application under field conditions (Simões and Spitzer 2000). The occurrence of such degradation reactions leads to alterations in the chemical composition of the oils and can negatively impact its biological activity as an insecticide with repellent, deterrent, nymphicidal and/or adulticidal activities. It seems clear, therefore, that for the application of essential oils under field conditions the development of formulations for protection is required. Our results demonstrated that the encapsulation of the essential oils of *X. aromatica* with by PCL nanospheres offered significant photoprotection, preventing oxidation and decomposition processes, very important features for its application under field conditions. In addition, the essential oil from leaves, formulated in polymer nanoparticles, prevented phytotoxic on bean leaves when sprayed at 2%. This protection was probably due to the controlled release of the active compounds by the nanoparticles. Others studies also observed phytotoxic on dry bean leaves treated with different oils at concentrations ≥ 2% (Pinheiro et al. 2009; Marques

et al. 2014). The phytotoxic activities of essential oils are well known and have been extensively studied to explore their herbicidal benefits (Amri et al. 2013).

In vitro analysis of controlled release offers the possibility to determine the release mechanism of the active principle. Notably, desorption of the active principle at the particles surface, diffusion of the active principle through the nanospheres matrix and physicochemical erosion of the polymer matrix are factors directly related to the release of the active principle. Although it was observed that the release profile of essential oils from *X. aromatica* leaves was around 50% after 2 h, a significant decrease in the diffusion rate was observed after 72 h (75%). The occurrence of biphasic release, as observed here, may be related to the amount of active principle adsorbed at the surface of the nanoparticles. In good agreement, results from Soppimath et al. (2001), Schaffazick et al. (2003) and Christofoli et al. (2015) also demonstrated that the release of drugs in nanospheres occurs exponentially (first order).

Our biological assays revealed that the females of *B. tabaci* laid significantly less eggs in plants treated with the *X. aromatica* essential oil than in plants of the control groups in free-choice tests. These results suggest that the volatile components of the essential oil did not affect the control plants. Similar repellency free-choice tests conducted by Pereira et al. (2018) with *Zanthoxylum riedelianum* fruit essential oil against *B. tabaci* also showed no interference of its volatile components on the control plants. Moreover, the reduced number of *B. tabaci* eggs can be also caused by disturbance of the insect. According to Martinez (2002), the insects present chemoreceptors in their tarsus; thus, they could detect the presence of essential oils in treated plants. This may somehow irritate the whiteflies, which after detecting the presence of a foreign substance do not stop at a fixed point to lay eggs and feed (Yang et al. 2010); this observation suggests a repellence effect. Previous studies performed by Kumar et al. (2005) indicated that the reduced oviposition observed in plants treated with essential oils may occur due to excitement of the whiteflies, suggesting that the constant movement takes place because the essential oils form a protective barrier on the leaves, hampering their ability to suck the sap from the phloem.

Although several studies have already shown that the activity of essential oils against different insects is mostly explained by the major compounds (Ipek et al. 2005; Regnault-Roger et al. 2012), other minor molecules also appear to modulate their activity (Hoet et al. 2006; Singh and Pandey 2018). For example, Deletre et al. (2016) through chemical studies and biological assays demonstrated that essential oils compounds from four essential oils as lemongrass (*Cymbopogon citratus*), cinnamon (*Cinnamomum zeylanicum*), cumin (*Cuminum cyminum*) and citronella (*Cymbopogon winternarius*) possessed repellent, irritant and toxic activities, depending on their applied concentrations. It is important to mention that these characteristics were rather independent, suggesting that mechanisms associated with either repellent or the irritant/toxic activity are mostly likely distinct. Remarkably, the combined effects of different compounds from essential oils of these different species account for the bioactivity of the mixture, suggesting interactions between the compounds (Deletre et al. 2016). Altogether, this indicates that the growing interest in insecticidal efficacy of essential oils against insects, in particular *B. tabaci*, most likely will be less associated with the development of resistance due to the large number of possible active ingredients. Accordingly, the development of new technologies that guarantee abundant quantities of raw essential oil sources with homogeneous chemical compositions is clearly required.

Biological assay with nanospheres containing essential oil of the leaves and fruits of *X. aromatica* revealed a significant reduction in *B. tabaci* oviposition in leaves of common beans (*P. vulgaris*), similar to the action of the essential oil in natura. By using either in natura essential oils or nanospheres containing essential oils from *X. aromatica* leaves and fruits, similar results were observed. Both formulations significantly decreased the number of eggs of *B. tabaci* with the increase in the concentration of essential oil showing efficiency up to 98%. Others studies have also demonstrated the efficacy of essential oils in the control of *B. tabaci.* Pereira et al. (2018) observed that in natura and nanoencapsulated essential oils from *Z. riedelianum* fruit reduced the number of eggs and killed second-instar nymphs of *B. tabaci*. Oviposition, egg hatching and nymphal survival were reduced by the essencial oils from *Thymmus vulgaris*, *Pogostemon cablin* and *Corymbia citriodora* (Yang et al. 2010). Accordingly, essential oils from *Artemisia annua* reduced the ovarian development of females, suggesting a direct effect of such essential oils in egg production (Rao et al. 1999).

In contrast to commercial synthetic pesticides, bioactive natural compounds can act via multiple mechanisms, making the development of resistance less likely due to the diversity of active ingredients present in each mixture (Pavela and Benelli 2016). The toxicity of essential oils to insects is therefore influenced by their chemical composition, which depends on external factors (Lee et al. 2001), as described above. Essential oils are characterized by a diverse chemical composition, usually composed of monoterpenes and sesquiterpenes, and among the several utilities of these classes of compounds, the protection against insect pests in plants is of course appealing (Simões and Spitzer 2000).

Even though nanoencapsulation has not substantially improved the effect of essential oils, the use of nanoparticles is clearly justified due to its gradual release, photoprotection and solubility in water. Moreover, our study also

reveals the possible protection against phytotoxic action of essential oils as another advantage of nanoencapsulation application. Although terpene compounds play major roles in the metabolism of the plant of origin, the presence of these compounds may act differently in the metabolism of other organisms (Amri et al. 2012). Notably, studies on the herbicide potential of essential oils have revealed that mono- and sesquiterpenes can affect different physiological processes in plants (De Feo et al. 2002; Batish et al. 2004; Kaur et al. 2010; Grichi et al. 2016).

Although the possibility of physiological impact caused by essential oils on the plant is usually present, such impacts are highly dependent not only on the chemical components of the oil applied but also on the environmental conditions and the plant species in question. However, the occurrence of such phytotoxic effects is also dependent on how the substances are applied, as well as the dose used (Corrêa and Salgado 2011). It is worth mentioning that physiological and metabolic aspects of the interaction between plant–essential oils and plant–nanoparticles remains barely analyzed. The present work offers; therefore, the foundation for such investigation and opens up several research avenues associated with a possible impact of essential oils on ecophysiological and metabolic aspects of the application of these products in plants under field conditions for agronomic purposes.

Our results demonstrated that the nanoprecipitation method was highly efficient for the encapsulation of essential oils from leaves and fruits of *X. aromatica*. Furthermore, biological assays have clearly demonstrated that the essential oils used here can potentially inhibit or reduce the oviposition of *B. tabaci* in leaves of common beans, as well as the PCL nanospheres containing these oils. Besides, nanoencapsulated essential oils present significant advantageous since it has a photoprotective effect indicating a greater stability of the active principle. Nanospheres are also dispersed in aqueous medium and prolong the residual effect through the gradual release, decreasing the final number of applications. Notably, nanospheres were also able to prevent a possible phytotoxic activity by the in natura essential oil of the leaves from *X. aromatica* at high concentration.

The combined data set presented in our manuscript coupled with a growing body of evidence present in the literature allowed us to suggest that the nanoencapsulation is rather suitable approach for effective protection of active ingredients from the solar light-induced degradation at field conditions. In summary, our study with the essential oils from leaves and fruits of *X. aromatica* coupled with the results obtained with nanoencapsulation and biological tests demonstrates the feasibility to further continue the studies with this essential oil for the control of *B. tabaci* at field conditions in association with practices of integrated pest management.

Author contribution

MCP and CMC conceived and designed research. MCP, GCSC and LDS performed research. MFP and CCFA contributed new reagents and analytical tools. MRF contributed to the chromatographic analyzes. MCP, LELR, EDQ, WLA and CMC analyzed data and wrote the manuscript which was later approved by all authors.

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