UFSM

CIÊNCIA e NATURA

ISSN 2179-460X
OPEN ACCESS

Ci. e Nat., Santa Maria, v. 46, e73726, 2024 • https://doi.org/10.5902/2179460X73726
Submissão: 05/12/2023 • Aprovação: 06/03/2024 • Publicação: 06/09/2024

Chemistry

Chemical constituents and larvicidal activity of the microencapsulated essential oil of *Citrus aurantium* L. against *Aedes aegypti*

Constituintes químicos e atividade larvicida do óleo essencial de *Citrus aurantium* L. microencapsulado of *Citrus aurantium* L. frente *Aedes aegypti*

Breno Pereira Soares[I], Thaylanna Pinto de Lima[I], Ana Paula Muniz Serejo[I], Rodrigo Aquino de Almeida[I], Brendha Araújo de Sousa[I], Beatriz Jardim Rodrigues das Chagas[I], Marcelle Adriane Ataide Matos[I], Victor Elias Mouchrek Filho[I], Gustavo Oliveira Everton[I]

[I] Universidade Federal do Maranhão, São Luís, MA, Brasil

ABSTRACT

This study aimed to evaluate the chemical profile and larvicidal activity of the microencapsulated essential oil (EO) of *Citrus aurantium* L. against *Aedes aegypti*. The barks of *C. aurantium* were collected in São Luís-MA. The EO was extracted by hydrodistillation at 100°C for 3h, with chemical characterization through Gas Chromatography coupled with mass spectrometry (GC-MS). Encapsulation of the EO was performed by ionic gelling. To quantify the total phenolic content of the EO, the Folin-Ciocalteu spectrophotometric method was applied. Then, the lethality of the EO against *Aedes aegypti* was evaluated, and the LC_{50} for the action of the EO was calculated using the Probit method. The major constituents found in the EO of *C. aurantium* were: (-)-Terpinen-4-ol (32, 56%) and Caryophyllene oxide (23.52%). The larvicidal activity of the EO showed a LC_{50} of 3.589 mg L^{-1}. The results indicate that the evaluated EO is composed of substances that provide a good larvicidal effect, revealing its efficiency in combating and controlling *Aedes aegypti*.

Keywords: Bark; Phenolics; Larvicidal

RESUMO

Este estudo teve como objetivo avaliar o perfil químico e a atividade larvicida do óleo essencial (OE) de *Citrus aurantium* L. microencapsulado frente *Aedes aegypti*. As cascas de *C. aurantium* foram coletadas em São Luís-MA. O OE foi extraído por hidrodestilação a 100°C por 3 h, com caracterização química por cromatografia gasosa acoplada à espectrometria de massas (CG-EM). A encapsulação do OE foi

realizada por gelificação iônica. Para quantificar o teor de fenólicos totais do OE, foi aplicado o método espectrofotométrico de Folin-Ciocalteu. Em seguida, avaliou-se a letalidade do OE frente *Aedes aegypti*, calculou-se a CL_{50} para a ação do OE pelo método Probit. Os constituintes majoritários encontrados no OE de *C. aurantium* foram: (-)-Terpinen-4-ol (32,56%) e óxido de cariofileno (23,52%). A atividade larvicida do OE apresentou CL_{50} de 3,589 mg L^{-1}. Os resultados indicam que o OE avaliado é composto por substâncias que proporcionam um bom efeito larvicida, revelando sua eficiência no combate e controle do *Aedes aegypti*.

Palavras-chave: Casca; Fenólicos; Larvicida

1 INTRODUCTION

Essential oils (EOs) are volatile products that originate from the secondary metabolism of aromatic plants. They can be found in different parts of plants, generally concentrated in bark, leaves, stems, roots, flowers, rhizomes and seeds (Jain, Patel & Desai, 2022).

They have important biological and pharmacological properties such as antimicrobial, insecticide, analgesic and anti-inflammatory. Such properties are attributed to the various volatile compounds (acids, aldehydes and terpenes) present in essential oils, which are of great importance as they are considered highly bioactive compounds (Irshad et al., 2020).

Among the plants that produce essential oils, citrus fruits such as orange stand out (Khalid et al., 2021). With more than 800 thousand ha, orange is the most planted fruit in the country, orange juice is the most consumed fruit-based drink in the world, with Brazil holding 50% of world production (Fidélix et al., 2020), but its barks are commonly discarded.

Active substances of plant origin, such as monoterpenes and their complex mixtures, essential oils have a variety of properties among which they are of biological interest. However, some limitations related to the stability of these substances and their mixtures can be observed in the direct use in general (rapid volatilization, oxidation of the chemical constituents of the oil, among others). These limitations can be solved with the use of carrier systems, such as microcapsules (Ju et al., 2020).

Encapsulation technology was developed and has advantages in relation to trapping sensitive substances, in order to protect them from adverse conditions, extending their useful life, promoting the controlled release of the encapsulated material and its use in food matrices (Jiang, Liao & Charcosset, 20120).

The choice of the microencapsulation method depends on the properties of the material to be encapsulated (especially the solubility) and the type of particle desired (protection and release) , in addition to the purpose (modifications in the release) of the product (Choudhury, Meghwal & Das, 2021). Thus, this study aimed to evaluate the chemical profile and larvicidal activity of the essential oil with the scientific name (orange) against *Aedes aegypti*.

2 METHODOLOGY

2.1 Collection of plant material

Bark samples of *Citrus aurantium* L. (orange) were collected in São Luís – MA (-2.49853, – 44.27096). The plant materials were transported to the Laboratory for Research and Application of Essential Oils (LOEPAV / UFMA) of the Federal University of Maranhão (UFMA), where they were dried in a convective air oven (FANEM 520). Afterwards, being crushed and its mass measured for subsequent yield calculations.

2.2 Drying Kinetics

To carry out the drying, a digital convective air-drying oven FANEM 520 was used, standard air speed at 1 m/s. Drying was carried out on alternate days using a temperature of 45°C and the relative humidity monitored by a digital thermo-hygrometer (model INS-28 Intrusul). 5g mass was used, the samples were placed on aluminum-coated plates of dimensions 90 x 15 mm, and the mass was monitored throughout the process by discontinuous weighing on an analytical scale Shimadzu AUY220 and the weighing scheme following intervals of 5, 10, 20, 30 and 60 min, until

the end of the process.

Weighings were carried out until mass variations were negligible. Drying was completed when there was no mass variation of 0.0100 g between five successive weighings. To determine the moisture ratio (RU) during drying at different drying temperatures, Equation 1

$$RU(adm)= \frac{Ubs-Ue}{Ubs_{initiall}-Ue} \quad (1)$$

where; RU (adm)=moisture ratio, (dimensionless); $Ubs_{initial}$=initial water content (b.s.); Ue=equilibrium water content (b. s.); Ubs=water content at time t (bs)

The RU values obtained for each drying air temperature were analyzed by six different empirical and semi-empirical equations and non-linear regression, as shown in Table 1. For the adjustment of the mathematical models to the experimental data, a non-linear regression analysis was performed, by the QuasiNewton method, using the computer program Statistica 10.0.

Table 1 – Mathematical nonlinear regression models to predict drying

Model	RU	Eq.
Newton	exp (-kt)	1
Page	exp ($-kt^n$)	2
Henderson & Pabis	a.exp (-kt)	4
Logarithmic	a.exp (-kt) + c	5
Two Terms	a.exp ($-k_0t$) + b.exp ($-k_1t$)	6
Two Exponential Terms	a.exp (-kt) + (1 – a).exp (-kat)	7
Henderson & Pabis modified	a.exp (-kt) + b.exp (-gt) + c.exp (-ht)	8
Midilli-Kucuk	a.exp (-ktn) + bt	10
Wang & Singh	$1 + at + bt^2$	11

Source: Soares (2023)

The criteria used to determine the best fit of the models to the experimental data were the coefficient of determination (R^2) and the mean square deviation (DQM).

2.3 Extraction of essential oil

To extract the EOs, the hydrodistillation technique was used with a glass Clevenger extractor coupled to a round-bottomed flask placed in an electric blanket as a heat-generating source. 500 g were used, adding distilled water (1:8). The hydrodistillation was carried out at 100 °C for 3h collecting the extracted EO. Each EO was percolation dried with anhydrous sodium sulfate (Na_2SO_4) and centrifuged. These operations were carried out in triplicate and the samples were stored in amber glass under refrigeration at 4°C. Subsequently submitted to analysis.

2.4 Gas chromatography coupled with mass spectrometry

The constituents of the EOs were identified by gas chromatography coupled with mass spectrometry (GC-MS). 1.0 mg of the sample was dissolved in 1000 µL of dichloromethane (99.9%) purity.

The analysis conditions were as follows: Method: Adams (2017); Injected volume: 0.3 µL; Column: Capillary HP-5MS (5% diphenyl, 95% dimethyl polysiloxane) (Equivalent DB-5MS or CP-Sil 8CB LB/MS), in dimensions (30m x 0.25mm x 0.25 µm); Carrier gas: He (99.9995); 1.0 $mL.min^{-1}$; Injector: 280°C, Split mode (1:10); Oven: 40°C (5.0 min.) to 240°C at a rate of 4°C min^{-1}, from 240°C to 300°C (7.5 min) at a rate of 8°C (min^{-1}); tT=60.0 min; Detector: EM; EI (70 eV); Scan Mode (0.5 sec $scan^{-1}$); Mass range: 40 –500 daltons (one); Transfer line: 280°C.; Filament: off 0.0 to 4.0 min; Linear quadrupole mass spectrometer. To identify the compounds in the sample, the AMDIS program (Automated Mass spectral Deconvolution Mass & Identification System) was used.

2.5 Encapsulation

The encapsulation test followed the methodology described by (Dubey, 2009, p. 82). Sodium alginate, anhydrous calcium chloride was used and the EO under study, distilled water and Tween 80 surfactant were used for the synthesis of alginate microparticles. For the synthesis of particles with encapsulated EO, 60 g of sodium

alginate solution (3.5% m/v) were mixed with 15 g of Tween 80 and 6 g of essential oil. The mixture was homogenized at 10,000 rpm and then dropped onto a $CaCl_2$ solution (3.5.7%) to harden the particles by crosslinking. The microcapsules obtained were washed with distilled water and stored in a solution of sodium chloride (0.9%).

2.6 Total phenolic content

The determination of the total phenolic compounds of the EO and of the microparticles was carried out with adaptation of the Folin-Ciocalteu method (Waterhouse, 2002). 5 mg of the essential oil diluted in 1 mL of ethanol was used. To this solution, 3 mL of distilled water, 800 µL of Folin-Ciocalteu reagent and 2 mL of 20% sodium carbonate were added. After two hours, the reading was performed in a UV-VIS spectrophotometer at a length of 760 nm. The standard curve was expressed in mg L^{-1} of tannic acid.

2.7 Larvicidal activity

The eggs were collected at the Federal University of Maranhão, Campus Bacanga in São Luís/MA, using traps called ovitraps. The traps were inspected weekly to replace the straws and collect eggs. Initially, *Aedes aegypti* eggs were placed to hatch at room temperature in a circular glass aquarium containing mineral water. The species followed the methodology proposed by (Forattini, 1962). The larvae obtained were fed with cat food according to the methodology Silva et al. (1995) until they reached the third and fourth stage, the age at which the experiments were carried out.

The larvicidal activity tests were performed according to the adapted methodology proposed by (Silva, 2006). Initially, a 100 mg L^{-1} stock solution of each EO was prepared and diluted in 2% DMSO solution. Dilutions were prepared from this solution at concentrations of 10-90 mg L^{-1}.

To evaluate the larvicidal activity of the microparticles, concentrations (m/v) of them in distilled water were prepared. At each concentration of EO and microparticles,

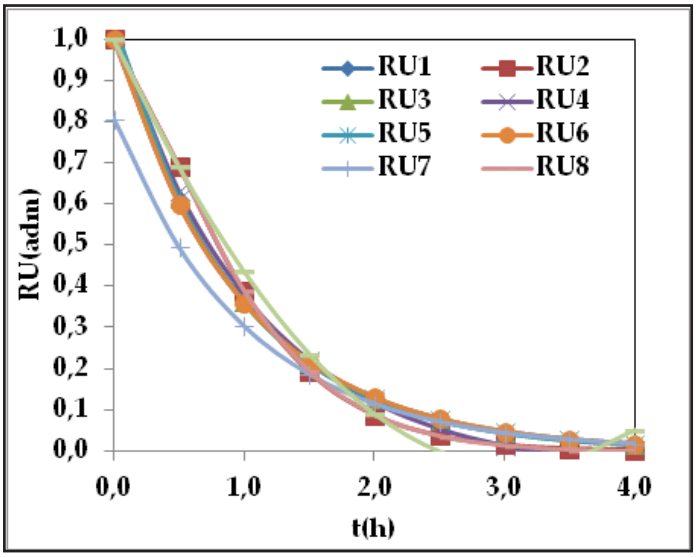
10 larvae were added at the rate of 1 mL per larva. All tests were performed in triplicate and as negative control a solution formed by DMSO 2% and as positive control a solution of temephos at 100 ppm, equivalent to the concentration used by the National Health Foundation (Funasa) for the larvicidal control of the vector. After 24 hours, the living and the dead were found, and the larvae that did not react to the touch after 24 hours from the beginning. The beginning of the experiment was considered dead. Statistical analysis of data was performed according to the method of (Finney, 1952).

3 RESULTS AND DISCUSSION

3.1 Drying Kinetics

Figure 1 shows the drying curves for the *C. aurantium* sample.

Figure 1 – Drying curves (time vs RU)



where; 1-Newton; 2-Page; 3-Henderson & Pabis; 4-logarithmic; 5-Two Terms; 6-Two Exponential Terms; 7-Henderson & Pabis modified; 8-Midilli-Kucuk; 9-Wang & Singh.
Source: Soares (2003)

It was observed in the graph that at the beginning of drying the drying rate is constant and that after approximately 2 hours this rate starts to enter a descending curve.

The behavior of the curves is due to the fact that the material surface is no longer saturated with water and the drying rate is controlled by the diffusion of moisture from the interior of the solid to the surface, because of this the curves tend to be more linear. The performance shown is typical of drying agricultural materials reported by many researchers.

Similar results were also observed for pineapple bark Leite et al. (2017) and for *Curcuma longa* L. (Sales et al., 2020). There are several factors that influence drying, such as the drying method used, temperature and relative humidity of the drying air, and air speed and drying time. The lack of control over these factors can compromise the quality of the final product (Goneli, 2008).

The parameters of the identified models, coefficient of determination and relative mean square deviation (DQM) for convective drying of *C. aurantium* bark are presented in Table 2.

Table 2 **–** Estimated parameters for drying

Model	DQM	P	R^2	k	a	b	n	c	k_1	g	h	K_0
1	0.0453	0.0979	0.9909	1.02	-	-	-	-	-	-	-	-
2	0.0194	0.0219	0.9983	0.95	-	-	1.37					
3	0.0434	0.1428	0.9917	1.05	1.04	-	-	-	-	-	-	-
4	0.0344	0.0000	0.9948	0.90	1.08	-	-	-0.06	-	-	-	-
5	0.0434	0.1428	0.9917	-	0.53	0.50	-	-	1.05	-	-	1.05
6	0.0453	0.0979	0.9909	1.02	1.00	-	-	-	-	-	-	-
7	0.0992	0.4224	0.9559	0.97	0.27	0.27	-	0.27	-	0.99	0.99	-
8	0.0192	0.0022	0.9984	0.94	1.00	0.00	1.37	-	-	-	-	-
9	0.0384	0.0494	0.9935	-	-0.67	0.11	-	-	-	-	-	-

where;1 – Newton; 2-Page; 3-Henderson & Pabis; 4-logarithmic; 5-Two Terms; 6-Two Exponential Terms; 7-Henderson & Pabis modified; 8-Midilli-Kucuk; 9-Wang & Singh. Source: Soares (2023)

All models offered reasonable fit to the drying kinetics data (R^2>0.90). For the drying process, the Midilli-Kucuk model was the best fit for the drying kinetics data, as it presented the highest coefficient of determination R^2 (0.9984), the lowest mean square deviation value (0.0192), and the lowest value for P (0.0022) indicating that

this model can be used successfully to predict the behavior of the drying kinetics of *C. aurantium* bark by convective drying under the conditions studied.

3.2 Chemical constituents

Through Gas Chromatography coupled with Mass Spectrometry (GC/MS) the major compound of the essential oil of *Citrus aurantium* L. barks was (-)-Terpinen-4-ol (32.56%), followed by Caryophyllene oxide (23.52%).

Martins et al. (2017) when analyzing the essential oil of bitter orange (*Citrus aurantium*) obtained as major components: d-limonene (78.53%) and γ-terpinene (12.65%). The work also analyzed the essential oil of sweet orange (*Citrus sinensis*) and as results had the following components: d-limonene (83.33%) as the major constituent, followed by linalool (8.91%). For the work carried out by Assunção et al. (2013) of the essential oil of the bark of *Citrus sinens,* it was obtained as a major component D-limonene (89.55%) and linalool (3.85%).

The chemical composition of a plant's essential oil is genetically determined, being generally specific to a particular organ and characteristic for its development (Simões et al., 2007) however, environmental conditions are capable of causing significant variations (Silvestre et al., 2010).

3.3 Total phenolic content

The encapsulation yields in calcium chloride concentrations are shown in Table 3.

Table 3 **–** Encapsulation performance based on crosslinked alginate mass

EO	**Yield *C. aurantium* 3% $CaCl_2$**	**Yield *C. aurantium* 5% $CaCl_2$**	**Yield *C. aurantium* 7% $CaCl_2$**
C. aurantium	65.60%	67.42%	41.63%

Source: Soares (2023)

Table 4 presents the result of the total phenolic content of *C. aurantium* EO. The

total phenolic content (TPC) was expressed as tannic acid equivalent (mg EAT g^{-1} of plant material) the straight equation obtained was y = 0.05857x + 0.06000 (R^2 = 0.9998), where y represents the absorbance x the equivalent concentration of tannic acid.

Table 4 **–** Total phenolic content of essential oil in vitro and encapsulated

EO	TPC mg EAT g^{-1}	Equation	R^2
C. aurantium	255.00	y = 0.05857x + 0.06000	0.9998
Encapsulated 3% $CaCl_2$	TPC mg EAT g^{-1} 122.6		
5% $CaCl_2$	93.6		
7% $CaCl_2$	85.1		

Source: Soares (2023)

It was observed in Table 3 the highest yield for the encapsulation of 67.42% for the encapsulation using 5% $CaCl_2$. Table 4 shows the quantity of phenolic compounds, but this data is little disclosed in the literature for the species under study. Also, in Table 3, as the $CaCl_2$ concentration increased for the encapsulated EO, the TPC value decreased respectively. A similar quantitative was identified by Costa (2015) in gram equivalence of gallic acid for species *C. sinensis* L was reported in 0.0990 μg EAG μg^{-1}.

Anagnostopoulou et al. (2006) studied the content of total phenolic compounds for *Citrus* barks extracts, sweet orange, obtained with organic solvents and obtained a range of values from 3 mg EAG g^{-1} to 105 mg GAE g^{-1}.

Benelli (2010) analyzed the total phenolic content for *Citrus sinensis* L. Osbeck orange bagasse obtaining a value of 6 ± 2 mg EAG g^{-1} for Soxhlet extraction – H_2O, 12 ± 1 mg EAG g^{-1} for ultrasonic extraction – H_2O, but the best result according to the due study was for extraction with ultrasound-ethyl acetate presenting the TPC at 60 ±5 mg EAG g^{-1}.

The EO *C. aurantium* used in the present study, shown in Table 4, could confirm an important quantity of phenolic compounds, which becomes of great relevance since phenolics are often associated with several positive health effects, including a decrease in risk of cardiovascular disease, anticancer mechanisms, anti-inflammatory

properties and antioxidant effects (Singh et al., 2012).

These antioxidants are compounds that can delay or inhibit the oxidation of lipids or other molecules, preventing the initiation or propagation of oxidation chain reactions. Among the natural antioxidants, the most used are the phenolic compounds due to their redox properties and their chemical structure (Costa, 2015). The action of a single or the synergistic action of several phenolic compounds can exert different activities in different ways, while isolated drugs usually act in a single way (Ambriz-Pérez et al., 2016).

3.4 Larvicidal activity

It was possible to observe in Table 5 that the EO presented a LC_{50} of 3.589 mg L^{-1} with a confidence interval of 2.962 – 4.349. against *Aedes aegypti* mosquito larvae.

For 1:10 encapsulated EO (m/v), *C. aurantium* $CaCl_2$ 3% showed mortality of 100% of larvae, for *C. aurantium* $CaCl_2$ 5% showed no mortality and for *C. aurantium* $CaCl_2$ 7% showed no mortality.

Table 5 – LC_{50} of the EO against *Aedes aegypti* larvae

Concentration (mg L^{-1})	Mortality (%)	LC_{50} (mg L^{-1})	σ	X^2	R^2
100.00	100.00				
90.00	100.00				
80.00	100.00				
70.00	100.00				
60.00	100.00				
50.00	100.00				
40.00	100.00				
30.00	100.00	3,589 (2.962-4.349)	0.200	0.932	0.998
20.00	100.00				
10.00	100.00				
5.00	80.00				
4.00	50.00				
3.00	40.00				
2.00	10.00				
1.00	0.00				
0.00	0.00				

Source: Soares (2023)

High concentrations of 10 to 100 mg L^{-1} of the EO showed a high larvicidal activity, causing 100% mortality of larvae. As the concentration below 10 mg L^{-1} was decreased, the percentage of mortality decreased. For the encapsulated EO, at concentrations of 7%, 5% and 3%, mortality of 100% was observed for the encapsulated concentration of 3%, with a yield of 65.60%.

The observed larvicidal potential is classified as very active according to the criteria based on Cheng et al. (2003) for lethal concentration (LC), where EOs that obtain LC_{50} < 50 mg L^{-1} are highly active. The LC_{50} obtained qualifies the EO as a potential larvicidal agent.

The larvicidal activity for the essential oil *C. aurantium* is little known in the literature, but for the EO of *C. sinensis* (pear orange, sweet orange) in the work carried out by Assunção et al. (2013) a LC_{50} was obtained for the essential oil of 99 .01 ± 2.098 mg L^{-1}, as well as the major component d-limonene was also analyzed by the author, corresponding to 89.55% of the chemical constituents, which was also assigned larvicidal activity, the essential oil of the respective author was then classified as moderate larvicidal agent.

Results close to those obtained in this work are reported by Oliveira (2013), when analyzing the larvicidal activity against *Aedes aegypti* with essential oil of *Citrus limon* reaching a result for LC_{50} of 14.99 mg L^{-1}, which also found a LC_{50} value for action of 113.24 mg L^{-1} isolated d-limonene, in which mortality activity was also attributed, EO was qualified as a potential larvicidal agent.

For the work carried out by Sousa (2019) with essential oil of *Citrus limon*, *Citrus limonia* and *Citrus aurantifolia* with larvicidal activity against *Aedes aegypti* larvae, the LC_{50} for each EO were calculated, respectively, 40.22 mg L^{-1}; 20.26 mg L^{-1} and 69.71 mg L^{-1}. The oils were considered biologically active against the larvae.

As in the work carried out by Martins et al. (2017), for the EO *C. sinensis*, for insecticidal activity against *Dysmicoccus brevipes*, a LC_{50} of 2.21% mL/100 mL was reached. The major component d-limonene was presented by the author, corresponding to

83.33% of the constituents of the EO, in which insecticidal activity was also attributed. In the comparison of preliminary tests, *C. sinensis* presented a mortality rate of 94.11% and for d-limonene 69.03%, however, it was found that the isolated action of this terpene presented a toxicity below 90% when compared to the sweet orange essential oil.

Still referring to the work done by Martins et al. (2017), who also used the essential oil of *Citrus limon* (sicilian lemon) for insecticidal activity, he presented d-limonene as the major constituent corresponding to 59.78%, reaching a LC_{50} of 0.72% mL/100 mL, the EO was considered more toxic on *Dysmicoccus brevipes*, in relation to sweet orange, for the due study.

Studies indicate that the terpenic components, alcohols and aldehydes of the EOs are responsible for the insecticidal or larvicidal activity against *Aedes aegypti* (Leite et al., 2009). The larvicidal activity of an EO is mainly attributed to its chemical constitution (Santos et al., 2020). According to Nascimento et al. (2016) the insecticidal effect may be due to a sum of substances present, which despite being in lower concentrations, also have insecticidal activity.

3 CONCLUSIONS

According to the results obtained for the total phenolic content and larvicidal activity of the EO extracted from the bark of *Citrus aurantium* L., it is concluded that the drying kinetics of the studied EO presents values similar to those found in the literature. An important quantity of phenolic compounds was obtained and the larvicidal activity of the oil proved to be efficient against *Aedes aegypti* larvae, being important and interesting in the control and combat of the mosquito that transmits dengue.

REFERENCES

Adams, R. P. (2017) Identification of essential oil components by gas chromatography/mass spectrometry. 5.ed. TX (USA): *Texensis Publishing*.

Ambriz-Pérez, D. L. et al. (2016). Phenolic compounds: Natural alternative in inflammation treatment. A Review. *Cogent Food & Agriculture*, 2(1), 1131412. doi: 10.1080/23311.932.2015.1131412.

Anagnostopoulou, M. A. et al. (2006). Radical scavenging activity of various extracts and fractions of sweet orange peel (Citrus sinensis). *Food chemistry*, 94(1), 19-25. doi: 10.1016/j.foodchem.2004.09.047.

Assunção, G. V. (2013). Chemical characterization and evaluation of the larvicidal activity front *Aedes aegypti* of the essential oil of the *Citrus sinensis* l. osbeck (sweet orange) species. [Dissertação de Mestrado em Química, Universidade Federal do Maranhão]. *Repositório Institucional da UFMA.*

Benelli, P. (2010). Agregação de valor ao bagaço de laranja (*Citrus sinensis* L. Osbeck) mediante obtenção de extratos bioativos através de diferentes técnicas de extração. [Dissertação de Mestrado em Engenharia de Alimentos, Universidade Federal do Maranhão] *Repositório Institucional da UFMA.*

Cheng, S. S. et al. (2003). Bioactivity of selected plant essential oils against the yellow fever mosquito Aedes aegypti larvae. *Bioresource Technology*, 89(1), 99-102. doi: 10.1016/S0960-8524(03)00008-7.

Choudhury, N., Meghwal, M., & Das, K. (2021). Microencapsulation: An overview on concepts, methods, properties and applications in foods. *Food Frontiers*, *2*(4), 426-442.

Costa, M. J. R. (2015). Determinação da atividade antioxidante e compostos fenólicos totais em óleos essenciais. [Dissertação de Graduação do Curso Tecnólogo em Processos Químicos), Universidade Tecnológica Federal do Paraná]. *Repositório Institucional da UTFPR.*

Dubey, R. et al. (2009). Microencapsulation technology and applications. *Defence science journal*, 59(1), p. 82-95. https://core.ac.uk/download/pdf/333719984.pdf.

Fidélix, M., Milenkovic, D., Sivieri, K., & Cesar, T. (2020). Microbiota modulation and effects on metabolic biomarkers by orange juice: A controlled clinical trial. *Food & function*, *11*(2), 1599-1610.

Finney, D. J. (1952). The estimation of the ED50 for a logistic response curve. *Sankhyā: The Indian Journal of Statistics*,12(1/2),121-136. https://www.jstor.org/stable/25048121.

Forattini, O, P. (1962). Entomologia médica. *Revista Saúde Pública*, 41(6), 185-302. https://www.scielosp.org/pdf/rsp/v41n6/03.pdf.

Goneli, A. (2008). Variation of physical-mechanical properties and quality of castor bean (Ricinus communis L.) during drying and storage. [Tese de Doutorado em Doutorado em Construções rurais e ambiência; Energia na agricultura; Mecanização agrícola; Processamento de produção, Universidade Federal de Viçosa]. *Repositório Institucional da UFV.*

Irshad, M., Subhani, M. A., Ali, S., & Hussain, A. (2020). Biological importance of essential oils. *Essential Oils-Oils of Nature*, *1*, 37-40.

Jain, P. L., Patel, S. R., & Desai, M. A. (2022). Patchouli oil: An overview on extraction method, composition and biological activities. *Journal of Essential Oil Research*, *34*(1), 1-11.

Jiang, T., Liao, W., & Charcosset, C. (2020). Recent advances in encapsulation of curcumin in nanoemulsions: A review of encapsulation technologies, bioaccessibility and applications. *Food Research International*, *132*, 109035.

Ju, J., Xie, Y., Guo, Y., Cheng, Y., Qian, H., & Yao, W. (2020). Application of starch microcapsules containing essential oil in food preservation. *Critical reviews in food science and nutrition*, *60*(17), 2825-2836.

Khalid, K. A., Darwesh, O. M., & Ahmed, A. M. (2021). Peel essential oils of citrus types and their antimicrobial activities in response to various growth locations. *Journal of Essential Oil Bearing Plants*, *24*(3), 480-499.

Leite, A. M. et al. (2009). Preliminary study of the molluscicidal and larvicidal properties of some essential oils and phytochemicals from medicinal plants. *Revista Brasileira de Farmacognosia*, 19(4), 842-846. doi: 10.1590/S0102-695X200.900.0600008.

Leite, D. D. F. et al. (2017). Mathematical modeling of pineapple peel drying kinetics. Green Journal of Agroecology and Sustainable Development. *Revista Verde de Agroecologia e Desenvolvimento Sustentável*, 12(4), 769-774. doi: 10.18378/rvads.v12i4.4980.

Martins, G. S. O et al. (2017). Chemical composition and toxicity of of citrus essential oils on *Dysmicoccus brevipes* (hemiptera: pseudococcidae). *Revista Caatinga*, 30(3), 811 – 817. https://www.scielo.br/j/rcaat/a/ss8NHJJCZpncgvbdryKqxHf/. doi: 10.1590/1983.212.52017v30n330rc.

Nascimento, A. A. et al. (2016) Atividade larvicida do óleo essencial de cravo-da-índia: Extração, caracterização e atividade larvicida frente ao mosquito *Aedes aegypti*. 1. ed. International Book Market Service, Riga(Letônia): *Editora Novas Edições Acadêmicas.*

Oliveira, M. B. (2013). Extraction, characterization and evaluation of larvicidal activity essential oil of linnaeus citrus limon (lemon) front mosquito Aedes aegypti. [Dissertação de Mestrado em Química, Universidade Federal do Maranhão]. *Repositório Institucional da UFMA.*

Sales, E. H. et al. (2020). Drying, toxicity and antimicrobial potential of Curcuma longa L essential oil. Research, Society and Development, 9(8), p. e511985600. doi: 10.33448/rsd-v9i8.5600.

Santos, A. B. S. et al. (2020). Óleos essenciais de *Cinnamomum zeylanicum* Blume e *Plectranthus amboinicus* (lour) Spreng como agentes larvicidas frente as larvas do Aedes aegypti. *Brazilian Journal of Development*, 6(4), p. 22355-22369. doi: 10.33448/rsd-v9i8.5600.

Silva, H. H. G. D, et al. (1995). Physiological age of aedes stegomyia aegypti eggs. Linnaeus, (1762) diptera, culicidae. *Revista de Patologia Tropical*, 24(2), 269 – 273. doi: 10.5216/rpt.v24i2.19696.

Silva, W. J. (2006). Larvicidal activity of essential oil from plants existing in the state of Sergipe against *Aedes aegypti* Linn. [Dissertação de Mestrado em Desenvolvimento e Ambiente, Universidade Federal de Sergipe. *Repositório Institucional da UFS.*

Silvestre, J. D. F. et al. (2010). Perfil da composição química e Atividade antibacteriana e antioxidante do óleo essencial do cravo-da-índia (*Eugenia caryophyllata* Thunb.). *Revista Ceres*, 57(5), 589-594. https://www.scielo.br/j/rceres/a/tgjGTK5Lj95w5HdfHMPnN9N/. doi: 10.1590/S0034-737X201.000.0500004.

Simões, C. M. et al. (2007) Pharmacognosy: from plant to medicine. Porto Alegre (Brasil): *Editora UFRGS.*

Singh, H. P. et al. (2012). Assessment of in vitro antioxidant activity of essential oil of Eucalyptus citriodora (lemon-scented Eucalypt; Myrtaceae) and its major constituents. *LWT-Food science and Technology*, 48(2), 237-241. doi: 10.1016/j.lwt.2012.03.019.

Sousa, D. A. (2019). Avaliação da atividade larvicida de óleos essenciais de espécies de citrus frente às larvas de Aedes aegypti. 2019. [Dissertação de Mestrado em Saúde e Ambiente, Universidade Federal do Maranhão]. *Repositório Institucional da UFMA.*

Waterhouse, A. L. (2002). Determination of total phenolics. *Current protocols in food analytical chemistry*, 6(1), 1-8.

Authorship contributions

1- Breno Pereira Soares

Graduação em Química Industrial (Universidade Federal do Maranhão); Laboratório de Pesquisa e Aplicação de Óleos Essenciais (LOEPAV/UFMA)
https://orcid.org/0000-0002-7836-1629 • breno_soares16@hotmail.com
Contribution: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Resources, Software, Visualization, Writing – original draft, Writing – review editing.

2 – Thaylanna Pinto de Lima

Graduação em Química Industrial (Universidade Federal do Maranhão); Laboratório de Pesquisa e Aplicação de Óleos Essenciais (LOEPAV/UFMA)
https://orcid.org/0000-0003-4504-4341• thaylana190@gmail.com
Contribution: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Resources, Software, Visualization, Writing– original draft, Writing– review editing.

3- Ana Paula Muniz Serejo

Doutoranda em Biotecnologia (RENORBIO); Professora (Faculdade Maurício de Nassau)
https://orcid.org/0000-0002-4376-4364 • apsmuniz1@gmail.com
Contribution: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Resources, Software, Visualization, Writing– original draft, Writing– review editing.

4 – Rodrigo Aquino de Almeida

Graduação em Química (Universidade Federal do Maranhão); Laboratório de Pesquisa e Aplicação de Óleos Essenciais (LOEPAV/UFMA)
https://orcid.org/0000-0001-6109-1282 • rodrigoaquino201494@gmail.com
Contribution: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Resources, Software, Visualization, Writing– original draft, Writing– review editing.

5 – Brendha Araújo de Sousa

Graduação em Química (Universidade Federal do Maranhão); Laboratório de Pesquisa e Aplicação de Óleos Essenciais (LOEPAV/UFMA)
https://orcid.org/0000-0003-4504-4341• asbrendha@gmail.com
Contribution: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Resources, Software, Visualization, Writing– original draft, Writing– review editing.

6 – Beatriz Jardim Rodrigues das Chagas

Graduação em Química Industrial (Universidade Federal do Maranhão); Laboratório de Pesquisa e Aplicação de Óleos Essenciais (LOEPAV/UFMA)
https://orcid.org/0000-0002-8940-0064 • jardimbeattriz@gmail.com
Contribution: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Resources, Software, Visualization, Writing– original draft, Writing– review editing.

7 – Marcelle Adriane Ataide Matos

Graduação em Química (Universidade Federal do Maranhão); Laboratório de Pesquisa e Aplicação de Óleos Essenciais (LOEPAV/UFMA)
https://orcid.org/0000-0001-5338-8123 • marcelle.mattooss@gmail.com
Contribution: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Resources, Software, Visualization, Writing– original draft, Writing– review editing.

8 – Victor Elias Mouchrek Filho

Professor Titular UFMA (Universidade Federal do Maranhão); Laboratório de Pesquisa e Aplicação de Óleos Essenciais (LOEPAV/UFMA)
https://orcid.org/0000-0003-2855-7292 • victor.mouchrek@ufma.br
Contribution: Funding acquisition, Project administration.

9 – Gustavo Oliveira Everton

Doutorando em Química (Universidade Federal do Maranhão); Laboratório de Pesquisa e

Aplicação de Óleos Essenciais (LOEPAV/UFMA)
https://orcid.org/0000-0002-0457-914X • gustavooliveiraeverton@gmail.com
Contribution: Conceptualization, Data, Curation, Formal Analysis, Funding, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – original draft, Writing – review editing.

How to quote this article

Pereira Soares, B., Lima, T. P. de, Serejo, A. P. M., Almeida, R. A. de, Sousa, B. A. de, Chagas, B. J. R. das, Matos, M. A. A., Mouchrek Filho, V. E., & Everton, G. O. (2024). Chemical constituents and larvicidal activity of the microencapsulated essential oil of *Citrus aurantium* L. against *Aedes aegypti*. *Ciência e Natura*, Santa Maria, 46, e73726 https://doi.org/10.5902/2179460X73726