

Chemical Composition and Insecticide Activity of Essential Oil of *Mesosphaerum suaveolens* Against *Nauphoeta cinerea*

José Weverton Almeida Bezerra (Corresponding Author)

Regional University of Cariri - URCA, Crato, CE, Brazil.

E-mail: weverton.almeida@urca.com

Francisco Alves Grangeiro Neto, Jacques Tavares Pereira Filho

Federal University of Pernambuco – UFPE, Caruaru, PE, Brazil

Adrielle Rodrigues Costa, Arycelle Alves de Oliveira, Isabella Hevily Silva Torquato, Natália Barbosa Campos, Talina Guedes Ribeiro, Priscilla Augusta de Sousa Fernandes, Conceição Taís de Araújo Pereira, Gledson Ferreira Macedo, Raimunda Aureniza Feitosa, Xenia Germana Rodovalho de Alencar, João Cruz Neto, Ma Aparecida Barbosa Ferreira Gonçalo, Maria Ivaneide Rocha

Regional University of Cariri - URCA, Crato, CE, Brazil

Received: Nov. 3, 2019

Accepted: Nov. 17, 2019

Published: Jan. 20, 2019

doi:10.5296/jas.v8i2.15737

URL: <https://doi.org/10.5296/jas.v8i2.15737>

Abstract

Essential oils are compounds produced by secondary plant metabolites and are found in leaf trichomes, moreover, they are characteristic because they present strong odors and present biological activities. Thus, the objective of this work was to evaluate the insecticidal action of *Mesosphaerum suaveolens* essential oil against nymphs of *Nauphoeta cinerea* as well as to characterize the chemical constituents present in it. The EO was extracted by means of hydrodistillation and its chemical characterization was done by Gas Chromatography coupled to Mass Spectrometry (GC/MS). For the biological assay against cockroaches, nymphs of 20 days old were selected for which they were submitted to different concentrations (50 - 1000 µg/mL oil per air) and mortality was evaluated over 48 hours of EO exposure. As a positive

control, ethanol (C₂H₆O) was used. Have been identified 44 compounds in EO, with β-Caryophyllene (18.57%), sabinene (15.94%) and spatulenol (11.09%) being the major compounds of EO. This showed no activity against the nymphs at any of the concentrations, whereas the positive control caused mortality at all concentrations tested. Thus, the essential oil of *M. suaveolens* does not present insecticidal properties in low concentrations against the cockroach.

Keywords: *Hyptis suaveolens*, lamiaceae, cockroach, terpenes

1. Introduction

Essential oils are compounds produced by secondary plant metabolites and are found in leaf trichomes, moreover, they are characteristic because they present strong odors and present biological activities (Bezerra et al., 2017). Usually they are mainly composed of mono and sesquiterpenes, however it is common to find other types of terpenes such as diterpenes (Bezerra et al., 2018). Among the biological activities, the main ones are antimicrobial, larvicidal, allelopathic and insecticidal actions (Costa et al., 2017).

Regarding the insecticidal activity, some plants developed chemical constituents with repellent and/or insecticidal properties during the evolution, so that insects did not interfere in the ecological succession of the species, mainly annual species, which are more susceptible. Among the annual species with insecticidal potential is *Mesosphaerum suaveolens* (L.) Kuntze, belonging to Lamiaceae, a botanical family rich in volatile terpenes (Bezerra et al., 2017; Harley, 2016).

This species is popularly known as "bamburral" and "lavender-brava" and has been shown to have insecticidal potential against *Drosophila melanogaster* (Bezerra et al., 2017), *Sitophilus zeamais*, *Acanthscelides obtectus* (Aberé; Oguwike; Sumaila, 2016) and *Anopheles gambiae* (Ivoke; Okafor; Owoicho, 2017). In this way, the species exhibits insecticidal properties that can substitute synthetic products, since they present toxicity to non-target organisms (Zemolin et al., 2014).

In addition to diptera and insects of the order Coleoptera, organisms of the order Blattodea, which are included cockroaches, cause serious economic damages to the feeding sectors. So these insects must be controlled in order not to cause damage. As a model in insecticide studies for this order, the cockroach *Nauphoeta cinerea* has been worked. This species has several advantages for toxicological studies, since they include an easy handling, a rapid proliferation and growth and it is exempt from the ethics committee (Rodrigues et al., 2013; Rugg; Rose, 1984).

Thus, the objective of this work was to evaluate the insecticidal action of *M. suaveolens* essential oil against nymphs of *N. cinerea* as well as to characterize the chemical constituents present in it.

2. Material and Methods

2.1 Plant Material

As for the leaves of *M. suaveolens*, these were collected in the morning (09:00 hrs) in 2015 under coordinates of Lat: $-6^{\circ}14'22.40''$, Long: $-39^{\circ}16'14.29''$ and altitude of 193 m of altitude (Figure 1). Fertile branches were treated according to the collection procedures, and later deposited in the same herbarium, under voucher #12.104.

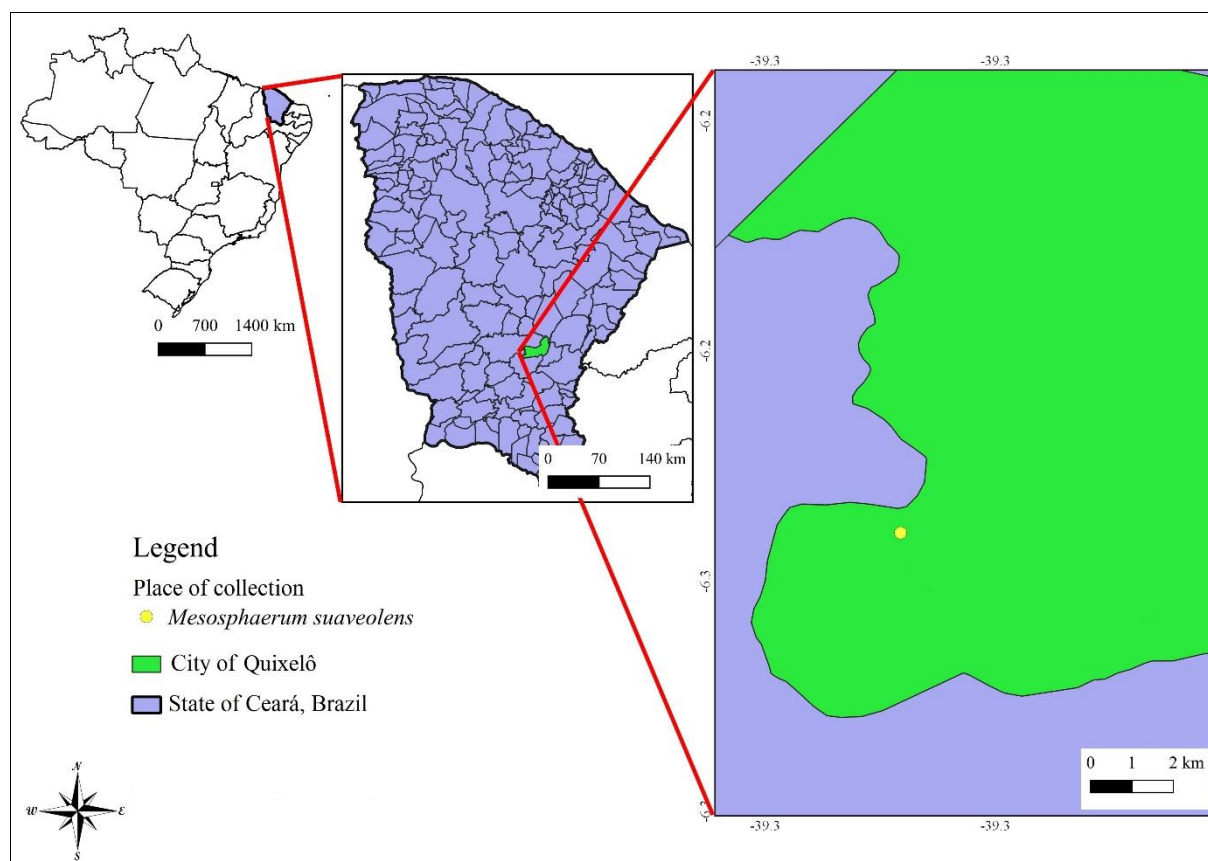


Figure 1. Map of the collection site of the species under study

2.2 Extraction of Essential Oil

The essential oil of *M. suaveolens* was extracted from dried leaves, submitted to hydrodistillation in Clevenger apparatus. After collection, the leaves were crushed into small pieces (150 g) and filled into a 1 L volumetric flask, where 2 L of distilled water was added. The flask was coupled to the Clevenger apparatus under the heating mantle and the temperature adjustment was carried out until the water boiled. After boiling, the 2 h time of the extraction cycle was started. At the end of each extractive cycle, the oil contained in the apparatus was collected with the aid of a pipette and stored in amber and refrigerated bottles. After extraction, sodium sulphate was used to remove the aqueous phase present in the essential oil (Bezerra et al., 2017; Matos, 2009).

2.3 Gas Chromatography (GC-FID)

The gas chromatography (GC) analyses was performed with Agilent Technologies 6890N GC-FID system, equipped with DB-5 capillary column (30 m x 0.32 mm; 0.50 μ m) and connected to an FID detector. The thermal programmer was 60 °C (1min) to 180 °C at 3 °C/min; injector temperature 220 °C; detector temperature 220 °C; split ratio 1:10; carrier gas Helium; flow rate: 1.0 mL/min. The volume injected 1 μ L diluted in chloroform (1:10). Two replicates of samples were processed in the same way. Component relative concentrations were calculated based on GC peak areas without using correction factors (Boligon; Feltrin; Athayde, 2013).

2.4 Gas Chromatography-Mass Spectrometry (GC-MS)

GC-MS analyses were performed on a Agilent Technologies AutoSystem XL GC-MS system operating in the EI mode at 70 eV, equipped with a split/splitless injector (220°C). The transfer line temperature was 220 °C. Helium was used as carrier gas (1.0 mL/min) and the capillary columns used were an HP 5MS (30 m x 0.35 mm; film thickness 0.50 μ m) and an HP Innowax (30 m x 0.32 mm i.d., film thickness 0.50 μ m). The temperature programmer was the same as that used for the GC analyses. The injected volume was 1 μ L of the essential oil diluted in chloroform (1:10).

2.5 Identification of the Components

Identification of the constituents was performed on the basis of retention index (RI), determined with reference of the homologous series of n-alkanes, C₇-C₃₀, under identical experimental conditions, comparing with the mass spectra library search (NIST and Wiley), and with the mass spectra literature date Adams (Adams, 1995). The relative amounts of individual components were calculated based on the CG peak area (FID response).

2.6 Inventory and Creation of *Nauphoeta Cinerea*

The cockroaches, *N. cinerea* were obtained from the Federal University of Santa Maria - UFSM provided by Professor Dr. João Batista Teixeira da Rocha. They were created and maintained at the Laboratory of Microscopy - LABOMIC, of the Regional University of Cariri - URCA, under temperature conditions of 25 \pm 5 °C and relative humidity of 50%. The diet of adult cockroaches and nymphs consisted of dog food and water at will.

2.7 Insecticidal Test Against *Nauphoeta Cinerea*

For the insecticidal assay the essential oil of *L. montevidensis* was contacted with filter paper and then attached to the 330 mL volume flask lid. Subsequently, 40 nymphs per group with 20 days of age were submitted to different concentrations of essential oil (50 - 1000 μ g/mL oil per air) and mortality was evaluated over 24 hours of OE exposure. The assay was accompanied by a negative control and a positive control. In this case, for the positive control ethanol (C₂H₆O) was used, since it is toxic and volatile, the ethanol was used at the same concentration. The protocol followed the methodology (Bezerra et al., 2017).

2.8 Statistical Analysis

Statistical analyzes were performed using the software GraphPad Prism 6, using One-Way Variance Analysis (ANOVA), followed by the Tukey test at 95% reliability ($p < 0.0001$).

3. Results

3.1 Chemical Composition

According to table 1, the essential oil of *M. suaveolens* presents a heterogeneous chemical composition, since it had a total of 44 components. Among these, the main constituents are β -Caryophyllene (18.57%), the sabinene (15.94%) and spathulenol (11.09%), which together account for 45.65% of the composition.

Table 1. Composition of *Mesosphaerum suaveolens* essential oil

Compounds	RI ^a	RI ^b	oil
			%
α -Thujene	989	931	1.09
α -Pinene	940	939	0.85
Sabinene	976	976	15.94
β -Pinene	980	980	2.11
Myrcene	994	991	0.26
δ -2-Carene	999	1001	0.49
α -Phellandrene	1006	1005	1.38
α -Terpinene	1019	1018	1.05
p-Cymene	1030	1029	0.76
Limonene	1031	1031	5.19
1-8-Cineole	1037	1033	3.04
(Z)- β -Ocimene	1041	1040	0.07
(E)- β -Ocimene	1055	1050	0.12
γ -Terpinene	1060	1061	2.97
cis-Sabinene hydrate	1068	1068	0.61
Linalool	1095	1098	0.43
cis-p-Menth-2-en-1-ol	1123	1121	0.28
t-Sabinol	1139	1140	0.15
4-Tepineol	1178	1177	6.82
p-Cymen-8-ol	1183	1183	0.23
α -Terpineol	1191	1189	0.94
δ -Elemene	1335	1338	1.17
α -Copaene	1377	1376	0.09
β -Elemene	1390	1391	0.78
β -Cedrene	1416	1417	0.14

β -Caryophyllene	1421	1418	18.57
β -Gurjunene	1433	1432	0.23
γ -elemene	1435	1433	1.44
aromadendrene	1439	1439	0.32
α -humelene	1453	1454	1.17
alloaromadendrene	1461	1462	0.40
γ -muurolene	1477	1477	0.28
germacreno D	1481	1480	5.21
β -selinene	1486	1485	0.89
Bicyclogermacrene	1501	1488	7.52
γ -cadinene	1512	1513	0.36
δ -cadinene	1525	1520	0.09
germacrene B	1559	1556	0.27
spathulenol	1576	1576	11.09
caryophyllene oxide	1580	1581	3.18
globulol	1582	1583	0.62
Cubenol	1641	1642	1.07
β -eudesmol	1649	1649	0.13
α -Cadinol	1656	1653	0.45
Total identified (%)			99.97

Relative proportions of the essential oil constituents were expressed as percentages.

RI^a Retention indices experimental (based on homologous series of n-alkane C₇–C₃₀).

RI^b Retention indices from literature (Adams, 2007).

3.2 Insecticide Activity Against *Nauphoeta cinerea*

As shown in Figure 2, contrary to the hypothesis of this work, the essential oil of *M. suaveolens* did not present insecticidal action in 24 hours against the nymphs of *N. cinerea*. There was only mortality in the positive control group from the lowest concentration (50 μ g/mL).

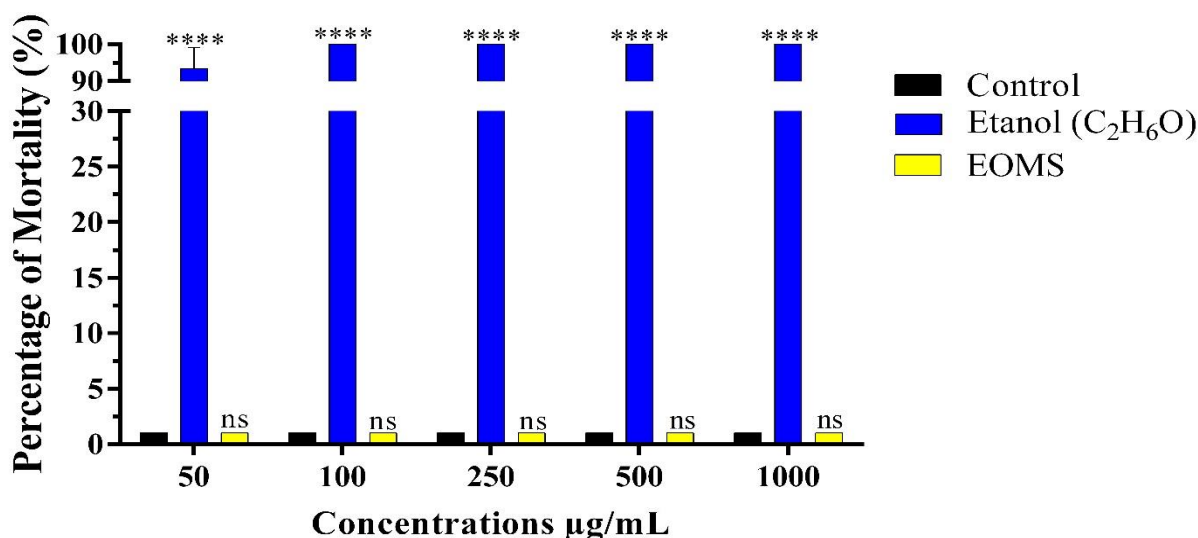


Figure 2. Percentage of mortality of nymphs of *Nauphoeta cinerea* nymphs submitted to the essential oil of *Mesosphaerum suaveolens* (EOMS) at various concentrations

4. Discussion

Phytochemically, the essential oil of *M. suaveolens* presents variations of study for study, this is common for this species since it has a great distribution throughout the terrestrial globe, consequently the environments that this species is present have their characteristics (Mishra et al., 2011). In this study the collection was carried out in the Caatinga, a seasonally dry tropical forest of Northeast Brazil, and showed as main components β -Caryophyllene (18.57%). In the study (Wangrawa et al., 2018) the major compound is 1.8 Cineole (26.4%), and the species was collected in the African continent in a Sudanian Savanna area which is a large tropical savannah belt that runs to the east and west across the African continent, from the Atlantic Ocean to the west to the western lowlands in the east (Savadogo et al., 2007).

Generally, the major compound determines the biological property of the essential oil, and in this study the main constituent was β -Caryophyllene (C₁₅H₂₄), this is a bicyclic sesquiterpene present in other essential oils such as *Syzygium aromaticum* (L.) Merr (Prasha; Locke; Evans, 2006) and *Cannabis sativa* L. (Wangrawa et al., 2018). This terpene is a sweet and dry tasting compound that can be found in a number of food items such as allspice, fig, pot marjoram, and roman camomile, which makes β -caryophyllene a potential biomarker for the consumption of these food products. Biologically, this compound presents some properties as larvicide in *Anopheles subpictus*, *Aedes albopictus* and *Culex tritaeniorhynchus* (Govindarajan et al., 2016).

The oil of the species under study has an insecticidal activity against the fly *Drosophila melanogaster* justifying that such action can be attributed to a synergistic action of the constituents, since the oil has a heterogeneous composition (Bezerra et al., 2017). While in our study there was no insecticidal action, this fact can be justified because they are different biological organisms.

Usually the essential oils present antioxidant actions, however some research shows that at the cellular level in eukaryotic organisms, oils can act as pro-oxidants, affecting cell membranes and organelles like mitochondria. Furthermore, depending on the concentration, they may exhibit cytotoxic effects, thus these oils can be used in the formulation of bioinsecticide (Bakkali et al., 2008).

5. Conclusion

In our study, the oil of *M. suaveolens* did not present an insecticidal action against the cockroach, however the oil may present an insecticidal action in higher concentrations.

References

- Abere, T. A., Oguwike, E., & Sumaila, M. (2016) Pharmacognostic standardization and insecticidal activity of the leaves of *Hyptis suaveolens* (L.) Poit (Lamiaceae). *Journal of Pharmacy & Bioresources*, 13(2), 163-170. <https://doi.org/10.4314/jpb.v13i2.12>
- Adams, R. P. (1995). Identification of essential oil components by Gas Chromatography/Mass spectroscopy. *Allured Publishing Corporation*: Illinois USA, p. 456.
- Bakkali, F., Averbeck, S., Averbeck, D., & Idaomar, M. (2008). Biological effects of essential oils—a review. *Food Chemical Toxicology*, 46(2), 446-475. <https://doi.org/10.1016/j.fct.2007.09.106>
- Benelli, G., Flamini, G., Canale, A., Molfetta, I., Cioni, P. L., & Conti, B. (2012). Repellence of *Hyptis suaveolens* whole essential oil and major constituents against adults of the granary weevil *Sitophilus granarius*. *Bulletin of Insectology*, 65(2), 177-183.
- Bezerra, J. W. A., Costa, A. R., Silva, M. A. P., Rocha, M. I., Boligon, A. A., Rocha, J. B. T., ... Kamdem, J. P. (2017). Chemical composition and toxicological evaluation of *Hyptis suaveolens* (L.) Poiteau (LAMIACEAE) in *Drosophila melanogaster* and *Artemia salina*. *South African Journal of Botany*, 113(11), 437-442. <https://doi.org/10.1016/j.sajb.2017.10.003>
- Bezerra, J. W. A., Santos, M. A. F., Meiado, M. V., Linhares, K. V., Boligon, A. A., Leandro, C. S., ... Silva, M. A. P. (2018). Allelopathy of Aromatic Species on the Germination of *Cereus jamaicaru* DC. subsp. *jamaicaru* (Cactaceae). *The Journal of Agricultural Science*, 10(11), 337-348. <https://doi.org/10.5539/jas.v10n11p337>
- Boligon, A. A., Feltrin, A. C., & Athayde, M. L. (2013). Determination of chemical composition, antioxidant and antimicrobial properties of *Guzuma ulmifolia* essential oil. *American Journal of Essential Oils and Natural Products*, 1(s/n), 23-27. [10.4172/2167-0412.1000126](https://doi.org/10.4172/2167-0412.1000126)
- Costa, A. R., Lima, S. J., Lima, K. R. R., Rocha, M. I., Barros, L. M., Costa, J. G. M., & Menezes, I. R. A. (2017). *Rhaphiodon echinus* (Nees & Mart.) Schauer: Chemical, toxicological activity and increased antibiotic activity of antifungal drug activity and antibacterial. *Microbial Pathogenesis*, 107(s/n), 280-286. <https://doi.org/10.1016/j.micpath.2017.04.001>

- Gertsch, J., Pertwee, R. G., & Di Marzo, V. (2010). Phytocannabinoids beyond the *Cannabis* plant—do they exist?. *British Journal of Pharmacology*, 160(3), 523-529. <https://doi.org/10.1111/j.1476-5381.2010.00745.x>
- Govindarajan, M., Rajeswary, M., Hoti, S. L., Bhattacharyya, A., & Benelli, G. (2016). Eugenol, α -pinene and β -caryophyllene from *Plectranthus barbatus* essential oil as eco-friendly larvicides against malaria, dengue and Japanese encephalitis mosquito vectors. *Parasitology Research*, 115(2), 807-815. <https://doi.org/10.1007/s00436-015-4809-0>
- Harley, R. M. (2016). Flora of the cangas of the Serra dos Carajás, Pará, Brasil: Lamiaceae. *Rodriguésia*, 67(5), 1381-1398. <https://doi.org/10.1590/2175-7860201667536>
- Ivoke, N., Okafor, F. C., & Owoicho, L. O. (2009). Evaluation of ovicidal and larvicidal effects of leaf extracts of *Hyptis suaveolens* (L) poit (Lamiaceae) against *Anopheles gambiae* (Diptera: Anophelidae) complex. *Animal Research International*, 6(3), 1072-1076. <https://doi.org/10.4314/ari.v6i3.55987>
- Matos, F. J. A. *Introdução à Fitoquímica Experimental* (2009). Fortaleza: UFC.
- Mishra, S. B., Verma, A., Mukerjee, A., & Vijayakumar, M. (2011). Anti-hyperglycemic activity of leaves extract of *Hyptis suaveolens* L. Poit in streptozotocin induced diabetic rats. *Asian Pacific Journal of Tropical Medicine*, 4(9), 689-693. [https://doi.org/10.1016/S1995-7645\(11\)60175-2](https://doi.org/10.1016/S1995-7645(11)60175-2)
- Prashar, A., Locke, I. C., & Evans, C. S. (2006). Cytotoxicity of clove (*Syzygium aromaticum*) oil and its major components to human skin cells. *Cell Proliferation*, 39(4), 241-248. <https://doi.org/10.1111/j.1365-2184.2006.00384.x>
- Rodrigues, N. R., Nunes, M. E. M., Silva, D. G. C., Zemolin, A. P. P., Meinerz, D. F., Cruz, L. C., & Franco JL (2013). Is the lobster cockroach *Nauphoeta cinerea* a valuable model for evaluating mercury induced oxidative stress? *Chemosphere*, 92(9), 1177-1182. <https://doi.org/10.1016/j.chemosphere.2013.01.084>
- Rugg, D., & Rose, H. A. (1984). Reproductive biology of some Australian cockroaches (Blattodea: Blaberidae). *Australian Journal of Entomology*, 23(2), 113-117. <https://doi.org/10.1111/j.1440-6055.1984.tb01922.x>
- Savadogo, P., Tigabu, M., Sawadogo, L., & Odén, P. C. (2007). Woody species composition, structure and diversity of vegetation patches of a Sudanian savanna in Burkina Faso. *Bois et Forêts des Tropiques*, 294(4), 5-20.
- Wangrawa, D. W., Badolo, A., Ilboudo, Z., Guelbéogo, W. M., Kiendrébeogo, M., Nébié, R. C. H., & Sanon, A. (2018). Insecticidal Activity of Local Plants Essential Oils Against Laboratory and Field Strains of *Anopheles gambiae* sl (Diptera: Culicidae) From Burkina Faso. *Journal of Economic Entomology*, 111(6), 2844-2853. <https://doi.org/10.1093/jee/toy276>
- Zemolin, A. P. P., Cruz, L. C., Paula, M. T., Pereira, B. K., Albuquerque, M. P., Victoria, F. C., & Franco, J. L. (2014). Toxicity induced by *Prasiola crispa* to fruit fly *Drosophila*

melanogaster and cockroach *Nauphoeta cinerea*: evidence for Bioinsecticide action. *Journal of Toxicology and Environmental Health*, 77(3), 115-124.

<https://doi.org/10.1080/15287394.2014.866927>

Copyright Disclaimer

Copyright for this article is retained by the author(s), with first publication rights granted to the journal.

This is an open-access article distributed under the terms and conditions of the Creative Commons Attribution license (<http://creativecommons.org/licenses/by/4.0/>).