

International Journal of Advanced Engineering Research and Science (IJAERS)

Peer-Reviewed Journal

ISSN: 2349-6495(P) | 2456-1908(O)

Vol-9, Issue-5; May, 2022

Journal Home Page Available: https://ijaers.com/ Article DOI: https://dx.doi.org/10.22161/ijaers.95.31



Chemical Composition and Bioactivity of Essential Oils of Croton blanchetianus Bailland and Sida cordifolia L. on Callosobruchus maculatus (Fabr., 1775)

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Received: 11 Apr 2022,

Received in revised form: 01 May 2022,

Accepted: 06 May 2022,

Available online: 29 May 2022

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Keywords— aromatic plants, hydrodistillation, Yield, pest insects, stored grains.

Abstract— This study aimed to evaluate the yield and chemical composition of essential oils from Croton blanchetianus and Sida cordifolia, in addition to investigating the insecticidal potential of these oils on adults of the bean weevil, Callosobruchus maculatus. To obtain the oils, the hydrodistillation method was adopted, using a Clevenger apparatus. The identification and quantification of chemical constituents was performed by means of gas chromatography coupled to mass spectrometry (GC-MS). For each oil, fumigation bioassays were performed on adults of C. Macullatus in six concentrations, 0; 0,30; 0,48; 0,60 e 0,70 (µL/750 mL) for C. Blanchetianus. As for the essential oil of S. cordifolia, the concentrations applied were 0, 3, 6, 9, 12 and 15 (μ g/750 mL). The bioassays were carried out under constant conditions of temperature (25 \pm 2°C) and relative humidity (60% \pm 10%). In the fumigation tests, different concentrations of the oils were applied to strips of filter paper (2x3 cm) attached to the bottom of the lid of a glass container (750mL), which contained 10 grams of cowpea and 15 unsexed adult insects. Insect mortality was evaluated 48 hours after the treatments were applied. Data were submitted to analysis of variance and regression analysis. When possible, lethal doses were calculated for 50 and 95% of the test population (LD50 and LD95). The yield of essential oil from C. blanchtianus was 0.50%, higher than that obtained for S.cordifolia, which was 0.03%. For the species C. blanchetianus, 20 components were identified, with the monoterpene eucalyptol being the major compound (32.89%). For S.cordifolia, 12 components were identified, with sesquiterpene spathulenol being the major compound (31.76%). In fumigation bioassays, considerable insecticidal activity of C. blanchetianus essential oil was observed on C. maculatus adults, with relatively low lethal doses. As for the essential oil of S.cordifolia, the

doses used in the study caused a maximum of 5.3% mortality, and it was not possible to determine the LD50 and 90 for the essential oil of this species.

I. INTRODUCTION

Grain loss due to insect pest infestation during storage can reach 50 to 60% in extreme situations (KUMAR et al., 2017; LUO et al., 2020) which is one of the major obstacles to improve food quality and achieve food security (BOXALL, 2001). Among the insects that damage cowpea under storage conditions, the woodworm *Callosobruchus maculatus* stands out. It causes a quantitative and qualitative damage to grains and seeds (SHAAYA et al., 1997; RAJKUMAR et al., 2019).

The management of stored grain insect pests is usually carried out with chemical pesticides (WAQAS *et al.*, 2018). However, its abuse leads to ecological, environmental and health risks (WERRIE *et al.*, 2021). In addition, it promotes the development of tough insect populations resistant to active ingredients (SISAY *et al.*, 2019; RUSIN e GOSPODAREK, 2018). Therefore, the discovery of a relatively safe new natural products effective against new insect, that contributes to a sustainable management of the agroecosystem, has become increasingly frequent.

Herbal products are potentially important sources of botanical pesticides. In such context, essential oils (EO) are presented as a promising alternative. They have insecticide activity, great bioavailability, and a great cost-effective relation (CAMPOLO e ORLANDO, 2018). Besides, they are efficient for insect control and the application does not require special care. Generalym they are safe and do not cause damage to human health and environment. (ISMAN, 2020; Zimmermann, 2021).

The methods for obtaining essential oils vary according to the part of the plant that is used. These methods can be by steam distillation, hydrodistillation or pressing (SIMÕES e SPITZER, 2004). These processes result in an aromatic concentrate, considered as a source of active substances, of extremely powerful and precise action (BUTNARIU, 2018). These aromatic compounds contain 85–99% of low molecular weight volatile components, including terpenoids, terpenes, and other aromatic and aliphatic constituents. (GONZÁLEZ-MAS *et al.*, 2019).

Essential oils are highly complex natural liquids, aromatic, volatile, hydrophobic and oily, consisting of several compounds synthesized by aromatic plants as secondary metabolites. (SWAMY, 2016). Terpenes are the most abundant compounds in essential oils (CAZELLA *et al.*, 2019), Terpenes are the most abundant compounds in

essential oils. They are generally toxic to physiological and biochemical processes in insects (PASSREITER *et al.*, 2004; *ROH et al.*, 2020), such as thymol, which can be found in essential oils of several plant species, including *Thymus vulgaris*, and has considerable toxicity against *Spodoptera litura* (Lepdoptera: Noctuidae) (MARCHESE *et al.*, 2016).

Rodrigues *et al.* (2019) analysing the chemical composition of the essential oil *C. blanchetianus* from Euphorbiaceae family, verifyed that 49.5% of the constituents were terpenoids, with 39.2% being monoterpenes and 10.3% sesquiterpenes. Pinheiro (2016) when analysing the chemical composition of *Sida rhombifolia* essential oil (Malvaceae), found 19.9% of monoterpene compounds. Investigating the chemical composition and insecticidal potential of essential oils from plant species is an important and necessary activity in order to obtain new compounds that can be used in pest management.

Intending to identify control methods of insect pests alternative to chemical control, we determined the yield, chemical composition of *C. blanchetianus* (Euphorbiaceae) and *S. cordifolia* (Malvaceae) essential oils and its possible. Com o objetivo de identificar métodos de controle de insetos-praga alternativos ao controle químico, determinamos o rendimento, a composição química dos óleos essenciais de *C. blanchetianus* (Euphorbiaceae) e *S. cordifolia* (Malvaceae) and its possible insecticide effect by means of biofumigation tests on the cowpea weevil (*C. maculatus*).

II. MATERIAL AND METHODS

2.1 Insect breeding

The creation of C. maculatus was carried out on Vale do São Francisco's Federal University (UNIVASF) Laboratory of Beekeeping and Meliponiculture, Agricultural Sciences Campus. The insects were kept in 2.0 L glass containers, covered with a thin "tulle" containing cowpea (V. unguiculata) grains. Adult insects were kept in the grains for eight days after copulation and oviposition. After this infestation period, the insects were removed, and the system was left with only the eggs to obtain the first generation. The same procedure was performed to obtain the next generations. The insects were kept in an airconditioned chamber, at a 25°C ± 2°C temperature, relative humidity of $60\% \pm 10\%$, and photophase of 12h.

2.2 Plant Material

The plant species C. blanchetianus and S. cordifolia used in this study were identified by the team of professionals from the Reference Center for the Recovery of Degraded Areas in Caatinga, at the Vale do São Francisco's Federal University (Centro de Referência para Recuperação de Áreas Degradadas da Caatinga, of Universidade Federal do Vale do São Francisco -CRAD/UNIVASF). Three samples of ach plant were deposited on the Herbarium Vale do São Francisco (HVASF), and the exsiccates of C. blanchetianus and S. cordifolia were registered with the numbers 24.231 and 24.232, respectively. The material for extracting the EOs consisted on fresh leaves of C. blanchetianus (latitude -9.26707946 and longitude -40.43271789) and S. cordifolia. (latitude -9.26678400 and longitude -40.43245554), which were collected in March 2021, at 9:00 am, on Santana's farm - Petrolina-PE city, Northeast region of Brazil.

2.3 Essential oil's extraction and performance

For the EOs extractions, it was weighed in triplicate, 100g of fresh leaves, which were transferred to a volumetric balloon of 2 Liters, with subsequent addition of 1.5 L of water. This balloon was connected to a Clevenger apparatus for hydrodistillation according to the method recommended by the European Pharmacopoeia (1983). At the process' end, the essential oil was measured directly in the extraction burette. Gravimetric analysis to obtain the oil yield (%, w/w) was performed, based on the fresh weight, according to the following equation: RO% = (Mo*100) / Bm. Where: RO%, Mo e Bm, are the essential oil yield, extracted essential oil mass and plant biomass, respectively. Após a extração, os óleos foram armazenados em frasco âmbar e conservados a 4°C até o uso.

2.4 GC-MS Essential Oil Analysis

The chemical analysis of the essential oil was performed using the SHimadzu QP 2020 equipment, which consists of a GC-2010 Gaseous Chromatograph, connected to a quadrupole mass spectrometry analyser. The column used was an RTX 5MS (crosslinked 5% phenyl methyl siloxane) with 30 m \times 0,25 mm internal diameter (DI), film thickness of 0,25 μm . The carrier gas was helium, with a column flow of 1,82 ml.min-1. The amount of samples injected was 1 μL with a split of 5:1. The oven temperature was initially 50°C, increased at a rate of 3°C. min-1 to 280°C. The quadrupole mass spectrometer was swept along the range 37 to 660 u (atomic mass unit) with an Electronic Impact voltage of 70 eV. The main components of the EOs were identified based on mass spectra compared with data from the Mass Library database NIST14.

2.5 Evaluation of insecticide activity

The evaluation of the insecticidal activity of EOs on *C. macullatus* was performed through biological assays with insects in adulthood, using the methodology of biofumigation in glass jars. Fifteen adult non-sexed insects were introduced into glass jars (750mL) containing 10g of cowpea. To perform the biofumigation tests in order to determine the lethal concentrations for the essential oil of *C. blanchetianus*, concentrations of 0; 1,38; 1,90; 2,63; 3,62; 5,0 (μg/750mL) were used. For the essential oil of *S. cordifolia*, only a preliminary test was performed involving the following oil concentrations 0;3;6;9;12 and 15 (μg/750 mL).

The application of the essential oil was performed by introducing a strip of filter paper (2x3 cm) inside the container lid being impregnated with different volumes of essential oil with the aid of a micropipette. To avoid direct contact of the essential oil with insects, the paper strip was suspended on a thin fabric, "tule" type, positioned at the top of the bottle. For the control treatment, strips of paper impregnated with distilled water were used, using the volume corresponding to the highest dose of the essential oil. After applying the treatments, the vials were closed in order to avoid the outflow of gases. Subsequently, the insects were kept in a climatic chamber, with $25^{\circ} \pm 2^{\circ}$ C, relative humidity of $60\% \pm 10\%$ and photophase of 12h. The evaluation of mortality was performed 48 hours after the installation of the experiment, recording the number of dead insects.

2.6 Statistical Analysis

The data were submitted to analysis of variance and regression study with the help of the Sisvar software. (FERREIRA, 2014). Lethal Doses (LD) were calculated by PROBIT (FINNEY, 1971), analysis using the R software.

III. RESULTS AND DISCUSSION

3.1 Essential oil extraction and yield

At the beginning of the distillation of the leaves of *C. blanchetianus* it was possible to observe two phases, an aqueous (aromatic water) and an organic (essential oil) of yellowish colour. In addition, it was possible to feel the presence of a characteristic aroma, the same detected in the field, at the time of collection of this plant material. After two hours of distillation, it was observed that the extraction of the oil was practically irrelevant. For species S. cordifolia, the biphasic mixture (aromatic water and essential oil) was only observed from the first hour of distillation. The essential oil presented a transparent colour preserving the aroma of fresh leaves collected in the field.

Table 1 shows the average yield of essential oils of *C. blanchetianus* and *S. cordifolia*, which reached values of 0.50 % and 0.03%, respectively.

Table 1. Average yield of essential oil obtained from leaves of C. blanchetianus e S. cordifolia.

| | C. blanchetianus | S. cordifolia |
|--------------------|------------------|---------------|
| Mass of leaves (g) | 100 | 100 |
| Oil mass (g) | 0,50 | 0,03 |
| Yield (%) | 0,50 | 0,03 |
| | | |

The yield of essential oil from fresh leaves of C. blanchetianus has already been determined in some studies, which demonstrated by the results that this species has a high content of volatile oil in its leaves. Angélico (2011), obteve um rendimento de 0,72%, já Melo (2011), relatou 0,70%. Angelico (2011), obtained a yield of 0.72%, Melo (2011), reported 0.70%. Pereira (2017), described an income of 0.40%. This study demonstrated an income close to the works cited here for C. blanchetianus. Regarding the species of the genus Sida, of the Malvaceae family, it is observed that they have a low content of volatile oils, since Pinheiro (2016) using the aerial parts of S. rhombifolia, obtained a yield of only 0.005%. Nunes (2006) using fresh leaves of S. cordifolia, obtained a yield of 0.06%, higher than the result of this study, as shown in Table 1. The respective variation in yield during oil extraction may be related to environmental factors such as climate, soil, altitude, wind, rain, as well as the time of collection.

3.2 GC-MS analysis of essential oil

The analysis by CG-MS of the essential oils of *C. blanchetianus* and *S. cordifolia* generated 20 peaks in both samples, where it was possible to identify by comparing the mass spectrum with the database of the Mass Library NIST14, 20 components in the first (Figure 1) and 12 in the second (Figure 3).

3.2.1 C. blanchetianus Essential oil

After comparing with the GC-MS database and known retention indices, it was possible to identify twenty EO compounds of *C. blanchetianus*, mostly monoterpenes, totalling 63.59% of their total composition (Table 2). The major constituents were monoterpene hydrocarbons 1,8-cineol (32.89%) and α -pinene (12.98%), in addition to sesquiterpene spathutulenol (13.87%), and its sum corresponded to a total of 59.74%. *C. blanchetianus*, like most species of the genus *Croton*, synthesise essential oils, which chemical composition is abundant in mono,

sesquiterpenoids and phenylpropanoids (PALMEIRA-JUNIOR et al., 2006).

Ribeiro *et al.*, (2018) performed the chemical analysis of the essential oils and obtained from the leaves (three species of the genus Croton collected at different times of the day, including *Croton blanchetianus Baill*) identified spatulenol as the majority constituent at 8 and 12 noon, and anethol at 8 pm. The same authors observed that for this species, spatulenol (second most abundant constituent of this study), is absent at 8pm. Oliveira (2008) identified in the chemical composition of the essential oil of *C. blanchetianus*, α -pinene (10.5%) as the majority compound, followed by β -pinene (1.4%) and β -myrcene (1.9%), while Rodrigues et al., (2019), observed 1,8 cineol (16.9%) as the main compound, followed by β -caryophyllene (15.9%) and germacrene D (14.5%).

The analysis of the chemical constituents of the essential oil's fresh leaves of *C. blanchetianus*, according to the data of the present study and previous reports, showed a variation in the majority compound, and only in the work of Rodrigues et al., (2019) similarities were found with the components referenced here. The authors also identified 1,8-cineol as the most abundant constituent. This compound is a monoterpene naturally identified in several aromatic plants of the genera *Eucalyptus*, *Croton*, *Hyptis*, *Pectis*, *Rosamarinus* e *Salvia* (ARAÚJO, 2003; VILELA, 2009). In this study, 1,8-cineol and α -Pinene as were considered major monoterpenes in *C. blanchetianus*, Ebadollahi essential oil (2020). It was also identified in the *Eucalyptus*' essential oil, recognized for its insecticide action, the 1.8-cineol (51.6%) and α -Pinene (15.8%).

These differences in the chemical composition of essential oils are linked to the physiology of the entire plant. The constituents and their amounts depend mainly on the enzymes responsible for catalyzing the production of volatile compounds in an organ. Stage of development and abiotic stresses such as soil salinity, moisture and temperature. (SANGWAN *et al.*, 2001).

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| | <i>J</i> | | |
|---------------------|----------|-------------|-------|
| COMPOUNDS | SI* | RT (min.)** | % |
| α-Thujene | 96 | 6.384 | 1.35 |
| α-Pinene | 97 | 7.073 | 12.98 |
| Sabinene | 96 | 8.445 | 2.03 |
| Nopinene | 97 | 8.541 | 1.65 |
| Myrcene | 92 | 9.142 | 1.45 |
| Phellandrene | 97 | 9.611 | 1.10 |
| Cymene | 96 | 10.435 | 2.08 |
| Eucaliptol | 90 | 10.751 | 32.89 |
| Terpinene | 97 | 11.859 | 1.21 |
| Terpinolene | 96 | 13.110 | 1.21 |
| 4-Terpineol | 95 | 17.043 | 2.56 |
| α-Terpineol | 96 | 17.677 | 3.08 |
| Elemene | 91 | 24.154 | 2.31 |
| β-Elemene | 93 | 26.499 | 1.12 |
| Caryophyllene | 95 | 27.637 | 7.14 |
| Humulene | 96 | 29.003 | 1.39 |
| D-Germacrene | 94 | 30.143 | 1.66 |
| B-Germacrene | 93 | 30.793 | 4.58 |
| Spathulenol | 94 | 34.045 | 13.87 |
| Caryophyllene oxide | 90 | 34.170 | 4.34 |
| Monoterpenes (%) | | | 63.59 |

Table 2. Constituents of C. blanchetianus'essential oil leaves.

Sesquiterpenes (%)

Total Componentes:

(**) Retention time.

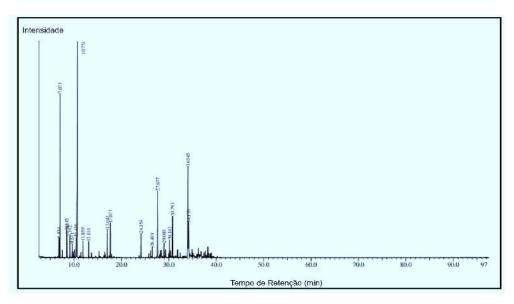


Fig.1. Chromatographic profile of the essential oil of C. blanchetianus' leaves.

^(*) Similarity Index based on NIST14 Mass Library mass spectra.

$$\begin{array}{c} \text{CH}_3 \\ \text{CH}_4 \\ \text{CH}_4 \\ \text{CH}_5 \\$$

Fig.2. Constituintes majoritários identificados no óleo essencial de C. Blanchetianus.

3.2.2 S. cordifolia's essential oil

For the essential oil *S. cordifolia* (Malvaceae), the Chemical analysis allowed the identification of 12 compounds by CG-MS, all of them sesquiterpenes, totaling 83.81% of its constitution (Table 4). The major compounds were spathulenol (31.76%) and caryophyllene oxide (24.88%), and its sum corresponded to a total of 56.64% (Figure 4). On the other hand, some authors identified monoterpenes compounds in plant species on *Malvaceae's* family, such as Pinheiro (2016), who analising the *Sida*

rhombifolia's essential oil, found out the linalol (15.1%), α-terpinol (2.6%), geraniol (1.7%) e o α-Pinene (0.5%). Oliveira (2021), in turn, analising the Chemical composition of *Malva Sylvestris*' essential oil, identified the monoterpene eugenol (14,9%). The constituents of the EOs' monoterpenes group, such as 1-8-cineol, carvacrol e eugenol, are the ones with high pesticidal properties Against stored product pests (AJAYI, 2014). So, the absence or low amount of terpenoids might result in low insecticidal activity.

Table 3. Constituintes do óleo essencial de folhas de S. Cordifolia.

| COMPOUNDS | SI* | TR (min.)** | % |
|---------------------|-----|-------------|-------|
| β-Elemene | 94 | 26.498 | 3.40 |
| Caryophyllene | 95 | 27.599 | 5.79 |
| Humulene | 95 | 28.997 | 1.73 |
| Aromadrendene | 94 | 29.287 | 1.07 |
| Humulene Epoxide | 91 | 32.926 | 1.20 |
| Spathulenol | 94 | 34.010 | 31.76 |
| Caryophylene Oxide | 92 | 34.160 | 24.88 |
| Ledol | 95 | 34.474 | 1.59 |
| Viridiflorol | 95 | 34.900 | 3.67 |
| Humulene Oxide II | 90 | 35.120 | 3.35 |
| α-Cadinol | 90 | 36.827 | 2.04 |
| Dioctyl Phtalate | 93 | 44.309 | 3.33 |
| Monoterpenes (%) | | | - |
| Sesquiterpenes (%) | | | 83,81 |
| Total Components: | | | 12 |
| I NYCET () () () | | | |

^(*) Similarity Index based on NIST14 Mass Library mass spectra.

^(**) Retention time.

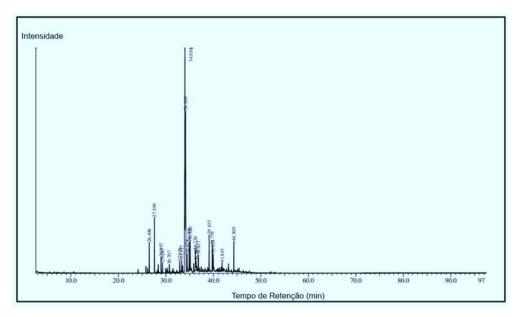


Fig.3. Chromatographic profile of essential oil from S. cordifolia leaves.

Fig.4. Major constituents identified in the essential oil of S. Cordifolia.

3.3. Insecticidal Activity

While the *C. blanchetianu's* essential oil showed greater bioactivity apresentou maior bioatividade on *C. maculatus* (CL50 = $2,44\mu$ L/750 mL e CL90 =

 $3,41\mu L/750$ mL, respectively) (Table 4), *S. cordifolia* essential oil showed low actividade Against the insect, causig low mortality, which reached at the maximum of 5,3% at the highest dose used ($15\mu L/750$ mL). So, it was not possible to determine the LC50 and LC 90 in this case.

Table 4. Estimates of DL50 e DL90 (μg/750 cm³) of Croton blanchetianus' essential oil on Callosobruchus maculatus.

| Essential oil | Angular Coefficient (± EP) | DL50 (IC) ^(a) | DL90 (IC) | χ ^{2 (b)} | p- value |
|------------------|-------------------------------|--------------------------|-----------------------|--------------------|-------------|
| C. blanchetianus | 8,73 ± 2,02 | 2,44 (2,24 - 2,65) | 3,41 (3,08 – 4,01) | 21,08 | 0,9999 |

^a IC: Confidence interval at 95% probability.

The mortality effect of the EO *C. blanchetianus* on *C. maculatus* was significative and was increasingly shown to increase the dose, which demonstrates the potential of this product for the control of *C. maculatus*, when applied via

fumigation on the insect. Corroborating the results of the present study, Silva *et. al*, (2020) observed in contact toxicity tests, the insecticidal action of the *C. blanchetianus*'s essential oil on *C. maculatus* (CL50 = 7,14

^b χ²: calculated chi-square value.

 μ L/20g of cowpeas and CL90 = 14,85 μ L/20g cowpeas respectively). Besides, the authors also demonstrated that the action of *C. blanchetianus* essential oil on oviposition and *C. maculatus* born alive is dose-dependent. The lowest and highest concentration caused an average reduction in the amount of 16.7% eggs and 95.52%, respectively, in addition to reduce the emergence of insects, which reached values of 15,75%. This result potentializes the efficiency of the EO from this specie on the *C. maculatus* since it shows insecticidal action by contact and fumigation. According to Silva et al., (2020) the EL from this specie shows Insecticidal activity for a long storage period, however, because it is biodegradable, its insecticidal activity decreases over time.

Other species of the genus *Croton* also have insecticidal activity, such as *C. conduplicatus* and *C. sonderianus* species, which, according to Oliveira et al. (2019), reduces oviposition and feeding punctures of the vegetable leaf miner, Liriomyza sativae, in melon.

Many studies have demonstrated insecticide effect of the essential oils on C. Macularia, acting in different ways. This demonstrates the abundance of plant species producing compounds with biocidal properties. The Lippia citrodora Kunth., Rosmarinus officinalis L., Mentha piperita L. and Juniperus Sabina L. essential oils show excellent fumigation action against C. maculatus, while the essential oil of wormwood from Judaea (Artemisia judaica L.) reduces the fecundity and emergence of adults. The Wedelia trilobata L.'s Essential oil, rich of α-pinene, also shows high potential for effectiveness in controlling C. maculatus (MAHMOUDVAND et al., 2011; ABD-ELHADY, 2012; SATONGROD et al., 2021)

Essential oils, through the diversity of constituents, have multiple mechanisms of action, connecting to different target sites at biochemical and physiological levels in insects. (TRIPATHI ARUN, 2016). Terpenoid compounds obtained from EOs reveal various insecticidal activities against pests of stored products, flies, cockroaches, and mosquitoes (TRIPATHI ARUN, 2016). From the main constituents of essential oils that have insecticidal properties against pests of stored products, monoterpenes 1,8-cineol and carvacrol are the most toxic, with a high insecticidal activity against C. maculatus. The compounds β-pinene and α-pinene also have activity on these insects. However, with a lower toxicity (AJAYI, 2014). The 1,8-cineol is used as a fumigant for C. maculatus adults (AGGARWAL al., 2001), and (RYAN acetylcholinesterase enzyme inhibitor and BRYAN, 1988).

Some studies propose that acetylcholinesterase (AChE) might be a potential target of insecticidal activity since the respective enzyme exerts a critical action in the regulation of nerve transmissions. (LIU J, 2021). It has already been demonstrated that insecticides induce mortality in pests by stopping AChE (BHAVYA et al., 2018). The monoterpenes 1,8-cineol and α-pinene (two of the major constituents of the essential oil of C. blanchetianus in this study), have previously been described as having inhibitory activity of AChE (DOHI et al., 2009). The monoterpenoids of numerous plant species' essential oils are, in fact, the main responsible for insecticidal activity (TONG and COATS, 2010), identified as fumigants and contact toxic for many insect pests. (RICE and COATS, 1994).

Some essential oils' constituents, especially the majority ones, are able to exert insecticidal action alone. However, this biocidal effect is potentiated by the joint action of the numerous constituents present, which end up promoting synergism. Rosemary essential oil has a potent insecticidal action, in which 1,8-cineole and camphor are two of its main constituents. Tak and Isman (2015) demonstrated that part of the Insecticidal action of rosemary essential oil is due to the synergism of 1,8-cineol and camphor, in which the first substance alone is more toxic than the second when applied topically. However, together, they cause a more significant insecticidal action, due to 1,8-cineol interaction with the lipid layer of the insect cuticle, causing increased solubility of camphor, potentiating its penetration and, consequently, a more lethal toxic effect.

It is observed that plant EOs, due to their less aggressive components, are able to replace conventional insecticides, because of their minimal harmful effects on the environment and human health. (GONZALEZ-MAS *et al.*, 2019).

Considering the numerous reports of the insecticidal action of the monoterpenes 1,8-cineol and α -pinene, it is believed that these constituents are the main actors in the fumigant insecticidal action of the EO of *C. blanchetianus* against *C. maculatus*. Besides, it is noticed that the maximum effect occurs due to the synergism of all elements, including the sesquiterpene spatulenol. The α -pinene e and the spatulenol have already been listed as potent insecticidal agents (SILVA et al., 2012; ANDRADE, 2016; LAWAL et al., 2018). Therefore, the *C. blanchetianus*' EOs due to its chemical composition, can become an alternative to chemical pesticides, because it is effective in controlling *C. maculatus*.

About the fumigant effect of the *S. cordifolia* essential oil, this study found no significant differences for the concentrations tested when compared to the control

treatment. The oil extracted from the leaves of these species caused low mortality of insects, reaching the maximum value of 5.3% in the highest dosage used $(15\mu\text{L}/750 \text{ mL})$. The chemical analysis of *S. cordifolia*'s essential oil did not identify any monoterpenoids. These constituents have biocidal activity and are usually found in large proportions in essential oils of aromatic plants with insecticidal activity. Therefore, it is believed that this fact can explain the results found in this study in relation to the species *C. blanchetianus* and *S. cordifolia*.

IV. CONCLUSION

The main volatile compounds of the essential oil obtained from the leaves of C. blanchetianus collected in Petrolina-PE city are the monoterpenes 1,8-cineol and α -pinene, in addition to the sesquiterpene spatulenol. For the S. cordifolia's essential oil, at the same city, the major components are spatulenol and caryophyllene oxide. The yields of essential oils from fresh leaves of C. blanchetianus and S. cordifolia were 0.50% and 0.03%, respectively.

From the essential oils evaluated, the essential oil of C. blanchetianus has fumigation toxicity, promoting high mortality in C. maculatus choleopteran adults, with relatively low lethal concentrations when compared to other analogue studies. The strong insecticidal activity of the essential oil C. blanchetianus may be, mainly, due to the monoterpenes 1,8-cineol e α -pinene and also to the synergistic interactions of mono and sesquiterpenes. The essential oil S. cordifolia, without the identification of any monoterpene constituent, promoted low mortality, with weak insecticidal potential

Our results emphasise the potential of *C. blanchetianus* as a source of essential oil for application in the control of *C. maculatus* in stored grains, and it may become a viable management option to replace synthetic insecticides.

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