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Synthesis, characterization, and evaluation of derivatives from cardanol as repellents/pesticides agents of *Liriomyza sativae* (Diptera: Agromyzidae) and *Bemisia tabaci* (Hemiptera: Aleyrodidae) on melon plants

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Maria Roniele Félix Oliveira
ORCID: https://orcid.org/0000-0002-1340-5678
Universidade Estadual do Ceará, Brazil
E-mail: ronielefelixmrfo@hotmail.com
Nìvia da Silva Dias-Pini
ORCID: https://orcid.org/0000-0002-3664-812X
EMBRAPA Agroindústria Tropical, Brazil
E-mail: nivia.dias@embrapa.br
Elaine Facco Celin
ORCID: https://orcid.org/0000-0001-7187-6913
Universidade Federal do Ceará, Brazil
E-mail: elainecelin@yahoo.com.br
katiany do Vale Abreu
ORCID: https://orcid.org/0000-0001-8605-1359
Universidade Estadual do Ceará, Brazil
E-mail: katyanyabreu@yahoo.com.br
Nádylla Régis Xavier de Oliveira
ORCID: https://orcid.org/0000-0001-5561-0445
Universidade Federal do Ceará, Brazil
E-mail: nadyllaregis@hotmail.com
Airis Maria Araújo Melo
ORCID: https://orcid.org/0000-0002-1082-1430
Universidade Estadual do Ceará, Brazil
E-mail: airismelo@hotmail.com
Marília de Albuquerque Oliveira
ORCID: https://orcid.org/0000-0001-8605-1359
Universidade Estadual do Ceará, Brazil
E-mail: marilia.albuquerque@uece.br
Atanu Biswas
ORCID: https://orcid.org/0000-0002-8112-1968
USDA Agricultural Research Service, USA
E-mail: atanu.biswas@ars.usda.gov
Huai Nan Cheng
ORCID: https://orcid.org/0000-0001-8647-057X
USDA Agricultural Research Service, USA
E-mail: hn.cheng@ars.usda.gov
Carlucio Roberto Alves
ORCID: https://orcid.org/0000-0001-7164-7467
Universidade Estadual do Ceará, Brazil
E-mail: carlucio.alves@uece

Abstract
Hydrogenated cardanol derivatives were evaluated as potentially active insecticides/repellent agents against insect pests such as *Liriomyza sativae* (Diptera: Agromyzidae) and *Bemisia tabaci* biotype B (Hemiptera: Aleyrodidae).

Benzoylation, acetylation, and methylation were made on the hydrogenated cardanol aromatic ring to synthesize active ingredients with potential insecticide action. Nuclear magnetic resonance spectra (^{13}C and ^{1}H NMR) and infrared (IR) proposed the chemical elucidation of the products. The production of melon seedlings was made and each synthesized products, benzoylated 3-pentadecylphenol, acetylated 3-pentadecylphenol, and methylated 3-pentadecylphenol were applied to seedlings by glass sprayer. Phytotoxicity evaluations were carried out, noting that all the plants showed burnt leaves except for concentration of 1% which the leaves remained healthy look. The insecticidal efficacy as well as the repellent and dissuasive effects were evaluated by a completely randomized design (DIC) and a randomized complete block design (DBCA) with six treatments, five and four replicates respectively. The compounds derived from cardanol did not present insecticidal activity in DBCA for *L. sativae*; however, they had a noticeable effect on oviposition index (IPO). Ovicidal effect and nymphal mortality only were found for *B. tabaci* by methylated 3-pentadecylphenol and Mospilan® in terms of dissuasion and oviposition effects in the two bioassays evaluated and, methylated 3-pentadecyl showed a similar behavior as the effect of Abamectin® on the confinement test, with 53.5% - 65.4% repellence of adults, an important data to emphasize that the products synthesized from cardanol are promising in preventing the pests attack such as *L. sativae* and, *B. tabaci*.
Keywords: Deterrent effect; Insecticidal effect; Repellence; White fly; Leafminer fly.

Resumo
Derivados hidrogenados do cardanol foram avaliados como inseticidas/repelentes potencialmente ativos contra insetos-praga como *Liriomyza sativae* (Diptera: Agromyzidae) e *Bemisia tabaci* biótipo B (Hemiptera: Aleyrodidae). Benzoilação, acetilação e metilação foram feitas no anel aromático do cardanol hidrogenado para sintetizar ingredientes ativos com potencial ação inseticida. Espectros de ressonância magnética nuclear (^{13}C e ^{1}H RMN) e infravermelho (IV) propuseram a elucidação química dos produtos. A produção de mudas de melão foi feita e cada produto sintetizado, 3-pentadecilfenol benzoilado, 3-pentadecilfenol acetilado e 3-pentadecilfenol metilado foram aplicados às mudas por pulverizador de vidro. Avaliações de fitotoxicidade foram realizadas, observando-se que todas as plantas apresentaram folhas queimadas, exceto na concentração 1% em que as folhas permaneceram com aspecto saudável. A eficácia inseticida, bem como os efeitos repelentes e dissuasores foram avaliados por um delineamento inteiramente casualizado (DIC) e um delineamento de blocos completos randomizados (DBCA) com seis tratamentos, cinco e quatro repetições, respectivamente. Os compostos derivados do cardanol não apresentaram atividade inseticida em DBCA para *L. sativae*; no entanto, eles tiveram um efeito perceptível no índice de oviposição (IPO). Efeito ovicida e mortalidade ninfal foram encontrados apenas para *B. tabaci* por 3-pentadecilfenol metilado e Mospilan® em termos de efeitos de dissuasão e oviposição nos dois bioensaios avaliados e, o tratamento com 3-pentadecil metilado apresentou comportamento semelhante ao efeito do Abamectin® no teste de confinamento, com 53,5% - 65,4% de repelência de adultos, dado importante para ressaltar que os produtos sintetizados a partir do cardanol são promissores na prevenção do ataque de pragas tais como *L. sativae* e *B. tabaci*.
Palavras-chave: Efeito dissuasor; Efeito inseticida; Repelência; Mosca branca; Mosca minadora.

Resumen
Los derivados del cardanol hidrogenado se evaluaron como agentes insecticidas/repelentes potencialmente activos contra plagas de insectos como *Liriomyza sativae* (Diptera: Agromyzidae) y *Bemisia tabaci* biotipo B (Hemiptera: Aleyrodidae). Se realizó benzoilación, acetilación y metilación sobre el anillo aromático de cardanol hidrogenado para sintetizar principios activos con potencial acción insecticida. Los espectros de resonancia magnética nuclear (^{13}C y ^{1}H RMN) e infrarrojo (IR) propusieron la elucidación química de los productos. Se realizó la producción de plántulas de melón y cada uno de los productos sintetizados, 3-pentadecilfenol benzoilado, 3-pentadecilfenol acetilado y 3-pentadecilfenol metilado, se aplicaron a las plántulas mediante un rociador de vidrio. Se realizaron evaluaciones de fitotoxicidad, observándose que todas las plantas presentaron hojas quemadas a excepción de la concentración 1% en la cual las hojas mantuvieron un aspecto saludable. La eficacia insecticida así como los efectos repelente y disuasorio se evaluaron mediante un diseño completamente al azar (DIC) y un diseño de bloques completos al azar (DBCA) con seis tratamientos, cinco y cuatro repeticiones respectivamente. Los compuestos derivados del cardanol no presentaron actividad insecticida en DBCA para *L. sativae*; sin embargo, tuvieron un efecto notable en el índice de oviposición (IPO). Solo se encontró efecto ovicida y mortalidad ninfal para *B. tabaci* por 3-pentadecilfenol metilado y Mospilan® en cuanto a los efectos de disuasión y oviposición en los dos bioensayos evaluados y el tratamiento con 3-pentadecilo metilado mostró un comportamiento similar al efecto de Abamectin® en la prueba de confinamiento, con 53.5% - 65.4% de repelencia de adultos, dato importante para resaltar que los productos sintetizados a partir de cardanol son prometedores en la prevención del ataque de plagas. tales como *L. sativae* y *B. tabaci*.
Palabras clave: Efecto disuasorio; Efecto insecticida; Repelencia; Mosca blanca; Mosca minadora de hojas.

1. Introduction

In recent years, the main melon-producing and exporting regions of northeastern Brazil had curtailed the cultivation of

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melon (Cucumis melo L.) due to the increase in the incidence of pests, especially the leafminer fly, *L. sativae* (Diptera: Agromyzidae) (Oliveira et al., 2017), and the white fly, *B. Tabaci* (Gennadius) (Hemiptera: Aleyrodidae) biotype B (Baldin et al., 2012). These pests cause serious economic loss with considerable reductions in melon yield and quality. The damage is especially serious in the Rio Grande do Norte and Ceará states of Brazil (Alcântara et al., 2019; Lemos et al., 2021; Silva et al., 2020).

Pests have been highlighted as the main phytosanitary problem in crops (Lamichhane et al., 2016; Silva et al., 2020). Chemical insecticides are used as the main strategy for pest control; however, this option favors the situation of widespread outbreaks. Because of the low selectivity, some insecticides can not only eliminate the intended targets but also contribute to the development of resistant populations. (Lacey et al., 2015; Nunes et al., 2013; Santos et al., 2018). The most effective insecticides in the control of these species are abamectin, spinosade, and cyromazine (Chang et al., 2022; Padilha et al., 2019; Santos et al., 2023; Zhang et al., 2022). There have been reports that demonstrated differential resistance with different synthetic insecticides (Capinera, 2020; Carrière & Onstad, 2023; Denholm & Devine, 2013; Somers et al., 2015).

Avoid problems and to reduce the level of highly toxic synthetic insecticides in the agricultural environment, new control methods have been used, e.g., biological compounds derived from natural products and genetically modified plants (Birchfield & McIntosh, 2020; Devos et al., 2022; Khursheed et al., 2022; Mendes Hacke et al., 2022; Naegeli, 2023; Zhu et al., 2021). The synthesis of selective and more efficient active ingredients can provide new bioinsecticides that meet the challenges in pest management (Klassen et al., 2023; Pirzada et al., 2020; Singh et al., 2020). In melon agricultural production, the use of cardanol derivatives as a precursor of new molecules with insecticidal activity appears attractive because it is a renewable raw material obtained from the waste of the cashew nutshell industry (Anacardium occidentale).

Chemical modifications of the benzene ring or the side chain of cardanol can produce substances with different activities and increase their potential use, as indicated in earlier studies of structure/activity relationships (Chang et al., 2022; Lim et al., 2018; Liu et al., 2016; Lomonaco et al., 2009). This approach allows insecticidal products with high specificity to be generated and satisfies the demand required in certain crops, such as the conservation of beneficial entomofauna and the control of pests with high infestation power and resistance to chemical insecticides.

In this work, we aimed to evaluate the insecticidal activity, deterrent effect, and repellence of molecules derived from cardanol (in the emulsion form), obtained by the appropriate synthesis and modifications of hydrogenated cardanol, against two insects (*L. sativae* and *B. Tabaci*) in the melon plant.

2. Methodology

2.1 Synthesis and chemical characterization

The reagents for the reactions and solvents for the silica gel column and thin layer chromatography were purchased from commercial sources (Vetec, Synth) and were used without further purification. Silica gel-60 chromatographic column was used (70-230 mesh ASTM, Merck). All molecules synthesized were monitored by thin-layer chromatography on Camlab-Polygram Silk/UV254 silica gel plates with 0.25 mm thickness. The structural confirmation of the synthesized compounds was carried out with nuclear magnetic resonance (NMR) and infrared (IR) spectroscopy. The ^{1}H and ^{13}C NMR spectra were obtained on an Agilent 500/54 spectrometer, operating at frequencies of 399.88 and 100.56 MHz, respectively. The IR spectra were obtained with KBr tablets on a Shimadzu IRAffnity-1 spectrometer.

2.2 Chemical synthesis

Three chemical modifications were made on the hydrogenated cardanol aromatic ring (benzoylation, acetylation, and methylation). For the benzoylation reaction, the cardanol was reacted with 5% sodium hydroxide solution (Vetec, 97%) and benzoyl chloride (Vetec, 98-100.5%). The acetylated product was obtained via the reaction of the cardanol with acetic anhydride (19.5 mL, Vetec, 97%) and pyridine (39 mL, Vetec, 99%); the reaction product was extracted with ethyl acetate (Dinamica, 99.8%). The reaction of cardanol with sodium hydroxide (Vetec, 97%) and dimethylsulfate (Sigma-Aldrich, ≥ 99%) provided the synthesis of the methylated product. Thus, from the hydrogenated cardanol three active ingredients with potential insecticidal action were synthesized: benzoylated 3-pentadecylphenol, acetylated 3-pentadecylphenol, and methylated 3-pentadecylphenol.

2.3 Bioassays and phytotoxicity

The bioassays were carried out in the entomology laboratory of Embrapa Agroindústria Tropical, Ceará, Brazil. *L. sativae* and *B. Tabaci* were obtained from mass rearing at the laboratory and greenhouse.

The production of melon seedlings was made in a greenhouse. The melon seeds were planted in polyethylene trays with 150 cells, containing coconut fiber as a substrate and vermiculite in a 2: 1 ratio. The transplant was performed on the tenth day after sowing onto polyethylene pots with 7.5 cm in height × 10.5 cm in diameter and a capacity of 0.5 kg with a substrate of humus and sand (3:1 ratio). Irrigation was done by dripping and, after the transplant, the plants remained in the greenhouse at a standardized distance of 50 cm, with humidity of 70 ± 10%, a temperature of 27 ± 3°C and, 12:12 h L:D photoperiod until they reached their vegetative development with three definite leaves formed.

Before the bioassays, phytotoxicity tests were carried out on the insecticides registered for melon use in order to establish the maximum non-phytotoxic concentration. About 1 g of each synthesized molecule, benzoylated 3-pentadecylphenol, acetylated 3-pentadecylphenol, and methylated 3-pentadecylphenol was emulsified in 0.025 g fixed (0.023 mL or 0.023% v/v) Tween 20 (Vetec, Brazil) to obtain five concentrations (emulsions) at 1, 2, 3, 4 and 5% w/w (molecule/water) in 100 mL distilled water. The experiment was carried out with a completely random design, with 4 repetitions and 6 treatments (4 emulsions, Control: distilled water only, and a blank: Tween 20 and distilled water). For the applications of the products, a glass sprayer was used at approximately 20 cm, it was possible to spray the entire 10 cm diameter without wasting the applied volume, which remained at 5mL. The seedlings remained in the greenhouse, and the phytotoxicity evaluations were made for 48 h, observing the presence of lesions on the surface of the leaves.

2.4 Evaluation of the insecticidal effect in the larval stage of the *L. sativae*

To evaluate the molecules insecticidal effect in the larval stage, a completely randomized experimental design (DIC) was used with three different emulsions at 1% (w/v) (benzoylated 3-pentadecylphenol, acetylated 3-pentadecylphenol, methylated 3-pentadecylphenol), Abamectin® (50 mL/100 L of water; v/v), control (distilled water), and blank (Tween 20 in distilled water; 0.023% v/v); each treatment was composed of five repetitions (plants). The application followed the above method (2.3). The percentage of larval mortality and the percentage of effectiveness of the treatments was determined by using Eq. 1 of Schneider-Orelli (1947).

Initially, 100 melon plants (with two true leaves) were used to perform the infestation caused by each pest (8 individuals/plant). The infestation was performed in an entomological cage for 15 h. After this time interval, the plants and flies were removed from the cages and the emulsions were applied (at different concentrations of the cardanol compounds). The plants remained in the greenhouse until the development of the larvae; they were then taken to the laboratory for the collection of the pupae and the observation of their later development up to the adult stage.

$$MC(\%) = \left(\frac{\text{Mortal.}(\%)\text{in T} - \text{Mortal.}(\%)\text{in C}}{100 - \text{Mortal.}(\%)\text{in C}}\right) * 100 \quad (1)$$

Where: MC (%) = corrected mortality; C = mortality in the control, and T = mortality after the treatment. For the calculation, the highest value between the mortality recorded in the control and the blank were used as the mortality in the control.

2.5 Evaluation of the deterrence for oviposition on the *L. sativa*

The treatments with insects were evaluated through a free-choice test and a confinement test. Melon seedlings (29 days after sowing) were sprayed with each emulsion at 1% v/v (benzoylated 3-pentadecylphenol, acetylated 3-pentadecylphenol, methylated 3-methyl pentadecylphenol), Abamectin® (50 mL/100 L of water, recommended dilution), control (distilled water), and blank (Tween 20 in distilled water; 0.023% v/v). For the applications of the products, a glass sprayer was used following the method of item 2.3. After fifteen minutes of the application, the melon seedlings were placed in cages for infestation for 15 h, with 8 individual insects/plants.

For the free-choice test, an experimental design with randomized complete block design (DBCA) was used, with six treatments (4 emulsions, control, and blank) and four repetitions (plants), and with 4 blocks and one repetition for each treatment. For the confinement test, DIC was used, with six treatments, four repetitions, and the experimental unit being the entomological cage. The preference for oviposition of the *L. sativae* was measured by the number of mines in each treatment. The oviposition preference index (IPO) was also calculated according to Eq. 2 (Fenemore, 1980)

$$\text{IPO}=\left[\frac{(T-P)}{(T+P)}\right] * 100 \quad (2)$$

Where: T = number of larvae in the treatment evaluated, and P = number of larvae in the control (distilled water). The index varies from +100 (very stimulating) to –100 (total deterrence), and the zero value being indicative of neutrality.

2.6 Evaluation of insecticidal effect in the nymph stages of egg and *B. tabaci*

The experiment was carried out in DIC with six treatments: the three cardanol molecules emulsified at 1% individually, Mospilan® (25 g/100 L), control, and blank with five repetitions. The flies, not sexed, that were present in the leaves of cabbage were randomly distributed in each entomological cage. The infestation of the melon seedlings was carried out in a greenhouse for a period of 3 h for oviposition. Later, the plants were placed in the laboratory for the counting of the eggs on the underside of the leaflets with the help of a magnifying glass. Each follicle was pulverized five times, with their respective treatments, and then the seedlings were kept in the greenhouse for nine days. After this period, when the nymphs were 2-3 days of age, the mortality in the egg stage was evaluated by counting the number of hatched nymphs and the number of viable eggs in the leaflet. On the tenth day, a second spray was carried out on the leaflets containing only nymphs. After seven more days in the greenhouse, the nymphal mortality was evaluated. With the percentages of mortality in the egg stage on the ninth day and nymphal mortality on the seventeenth day, the insecticidal efficacy was determined by Eq.1 of Schneider-Orelli (1947).

2.7 Evaluation of repellency and deterrence of cardanol derivatives in *B. tabaci*

The effect of repellency and oviposition was evaluated in relation to *B. tabaci* in melon plants, where six treatments were established (pulverization at different concentrations of cardanol derivatives having already been mentioned), with free-choice and confinement tests being carried out. After 15 min of the emulsion application, the melon seedlings were placed in entomological cages. The same experimental procedure was carried out with the deterrence test for the oviposition of the *L. sativae*. The repellency was evaluated at 6, 24 and 48 h after the release of insects, and the number of adults present on the back of the leaflets was counted. Subsequently, the repellency index (IR) was calculated with Eq. 3. The determination for oviposition was made by counting the number of eggs in each leaflet, after the third count (48 h) of the number of adults. The leaves were sectioned and analyzed in the laboratory, with the help of a magnifying glass, to determine the number of eggs present on the underside of the leaf, and the oviposition preference index was determined (Eq. 2).

$$IR = \frac{2T}{(T+P)} \quad (3)$$

Where: IR = repellence index; T = number of adults in the treatment evaluated, and P = number of adults in the control (distilled water). The IR values vary between zero and two, with IR = 1 indicating similar attraction among the evaluated molecules in 1% emulsion and the control (distilled water), IR < 1 corresponding to greater repellency by the extract, and IR > 1 indicating less repellency per extract, evaluated in comparison to control.

2.8 Statistical analysis

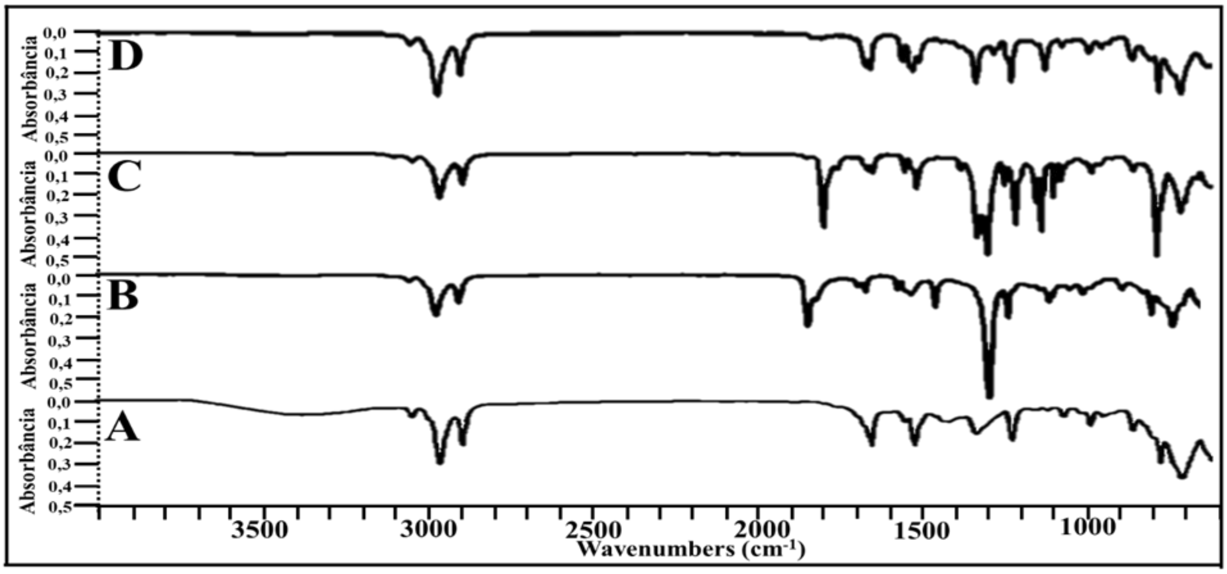
The data from this experiment were statistically evaluated by analysis of variance (ANOVA). Data transformation was conducted according to the optimal power method (Box-Cox), and averages were compared using the F test at the 5% probability level with the statistical program SASM-Agri. The data that did not show differences through the application of ANOVA were analyzed with the nonparametric test of Kruskal-Wallis and Friedman, with the statistical program ASSISTAT.

3. Results and Discussion

3.1 Spectroscopic characterization

The IR spectrum of the cardanol (Figure 1A) is compared with the IR spectrum of acetylate 3-pentadecyl phenol, an extra peak was observed at 1750 cm^{-1} (Figure 1B). Meanwhile, the characteristic absorption of the OH band, presented in the cardanol spectrum, at about 3300 cm^{-1}, disappeared. The presence of the acetate group was confirmed by the band at 1750 cm^{-1}, which corresponded to the functional group of the ester C = O. The benzoylated IR spectra exhibited a strong peak at about 1730 cm^{-1} (Figure 1C), assigned to the benzoate ester C = O. The absence of the absorption band at 3300 cm^{-1} of the OH group, confirmed this information. The methylated spectrum presented a weak absorption band at 2860-2800 cm^{-1} (Figure 1D) that overlap with cardanol peaks; however, it was evident that the absorption band at 3300 cm^{-1} (OH) disappeared, which seems to confirm the methylation.

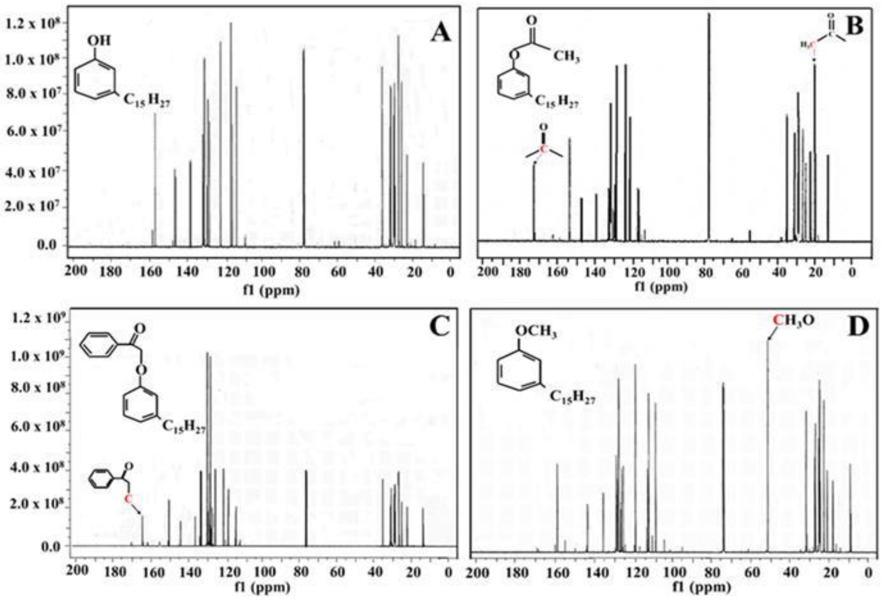
Figure 1 - Spectra in the infrared region. (A) Hydrogenated cardanol; (B) Acetylated 3-pentadecyl; (C) Benzoylated 3-pentadecyl; (D) Methylated 3-pentadecyl phenol.



Source: Authors.

The products derived from cardanol were synthesized and Analysed via ^{13}C NMR, ^{1}H NMR, and IR spectra of the synthesized compounds to evaluate the structures of the respective products. The ^{13}C NMR spectrum of acetylated 3-pentadecyl phenol (Figure2B) presented the acetate peaks at 22 and 169 ppm; these peaks were not present in the ^{13}C NMR cardanol spectrum (Figure 2A). This result was verified by the presence of $COCH_3$ peak at 2.3 ppm in the ^{1}H NMR spectrum (Figure 3B).

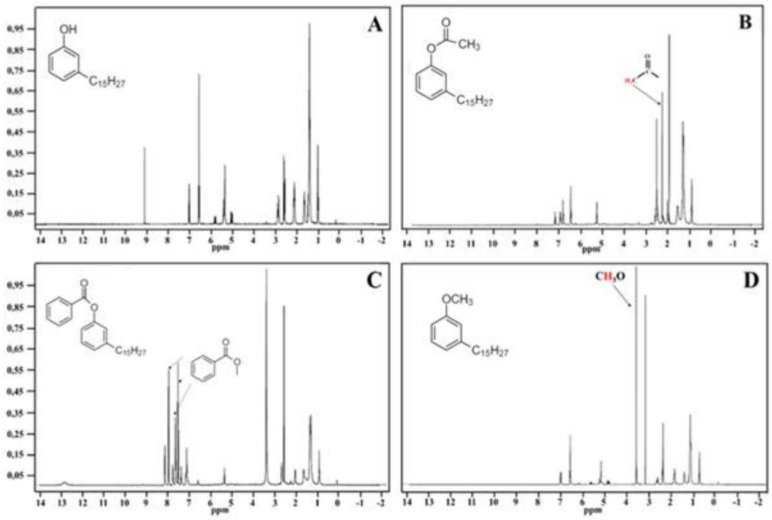
Figure 2 - ^{13}C-NMR Spectroscopy: (A) Hydrogenated cardanol; (B) Acetylated 3-pentadecylphenol; (C) Benzoylated 3-pentadecylphenol; (D) Methylated 3-pentadecylphenol.



Source: Authors.

The ^{13}C NMR benzoylated 3-pentadecyl phenol spectrum (Figure 2C) showed a peak at 165 ppm that could be assigned to the benzoate group. The other benzoate peaks were hidden among the aromatic peaks of cardanol. In the ^{1}H spectrum, the peaks at positions 7.43, 7.62, and 7.92 ppm (Figure 3C) confirmed the presence of the new aromatic ring in the benzoylated compound. The methylated compound of 3-pentadecyl phenol (Figure 2D) was evidenced by the methoxy group peak at 55 ppm. In the ^{1}H NMR spectrum (Figure 3D), a line at 3.75 ppm was characteristic of the methoxy group.

Figure 3 - ^{1}H NMR Spectroscopy: (A) Hydrogenated cardanol; (B) Acetylated 3-pentadecylphenol; (C) Benzoylated 3-pentadecylphenol; (D) Methylated 3-pentadecylphenol.



Source: Authors.

However, the multiple sp^{2} ^{13}C resonances between delta 110–170 strongly (Figure 2) suggest that not just that none of the three derivatives was prepared as pure entities, possibly as a consequence of the starting material itself lacking purity. Given implicit evidence of impurity, and thus uncertainty as for added contaminants. Even though, the products of the synthesis were used to evaluate their repellent and pesticide action.

3.2 Bioassays and phytotoxicity

The melon plants showed susceptibility to the three cardanol derivatives at 5, 4, 3, and 2%. 1% emulsions of derived molecules did not damage the leaves due 100% of healthy leaves were found with an external appearance similar to the control group. As a relation to insecticidal effect on *L. sativae* larvae, the benzoylated, acetylated, and methylated products of 3-pentadecylphenol and the active ingredient Abamectin®, in minimal doses, were ineffective. Low mortality and low efficacy were observed, and statistically equal observations were made without differences between the control groups (distilled water and Tween) and the blank group (distilled water) (Table 1).

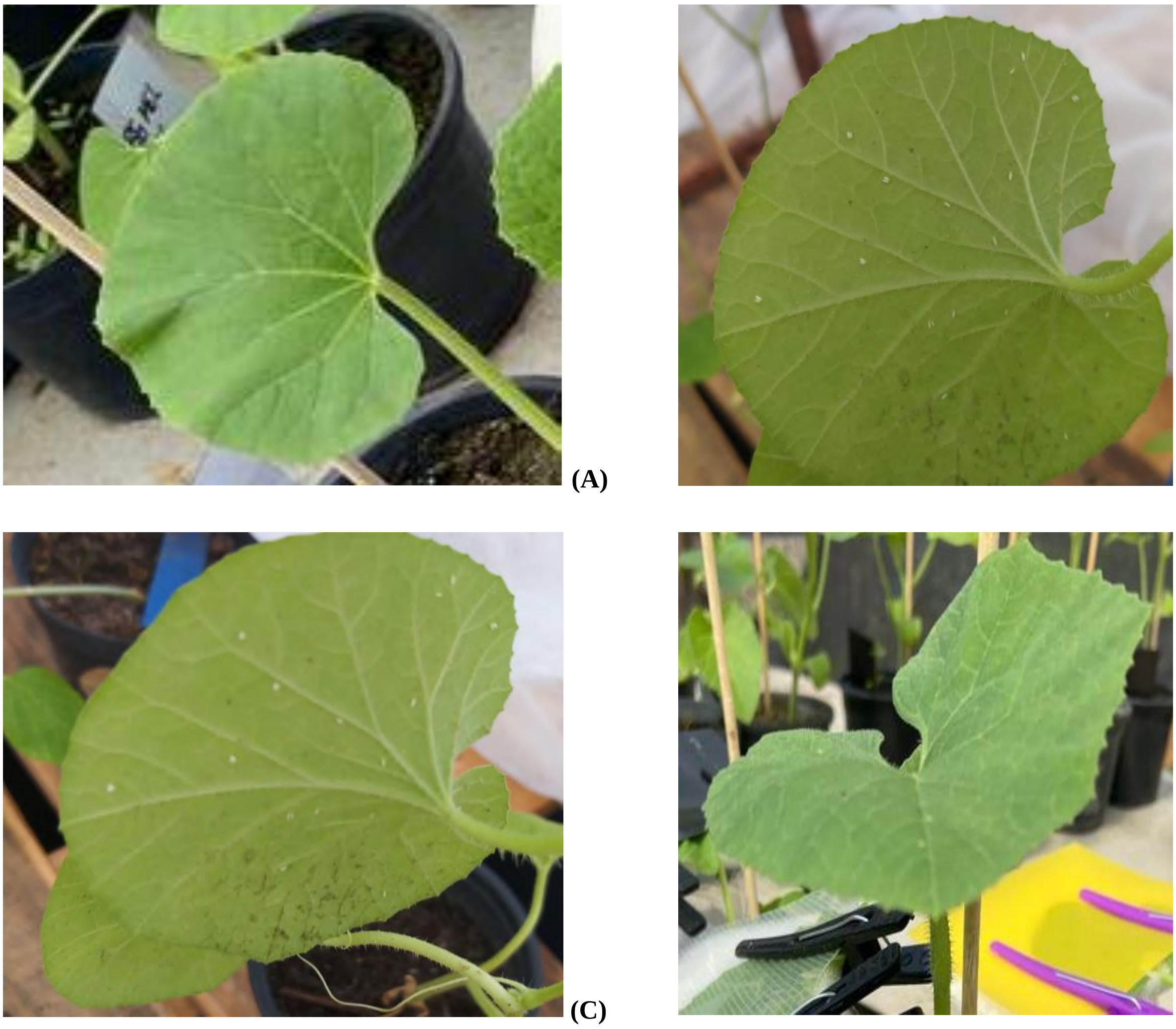
Table 1 - Average (±SD) of the mortality rate of the *L. sativae* larvae after emulsion´s application (1% Benzoylated 3-pentadecyl, 1% acetylated 3-pentadecyl, and 1% methylated 3-pentadecyl phenol) in the melon cultivation maintained in the greenhouse.

Treatments	**Number of mines ***	**Mortality *[1] (%)**	**Efficiency[2] (%)**
Benzoylated [(*)]	12.0 ± 6.9 a	6.4 ± 14.2 a	2.8
Acetylated [(*)]	16.0 ± 4.5 a	9.6 ± 11.4 a	6.1
Methylated [(*)]	13.6 ± 6.5 a	14.8 ± 8,7 a	11.5
Abamectin®	18.0 ± 5.6 a	12.4 ±12.5 a	9.1
Blank	16.8 ± 6.2 a	3.7 ± 3.8 a	-
Control	13.8 ± 4.1 a	3.6 ± 8.1 a	-
VC[3]	38.4%	95.1%	

[*] Averages followed by the same letter in the columns do not present differences by the F test at 5% probability.
[(*)] Benzoylated 3-pentadecyl, acetylated 3-pentadecyl, and methylated 3-pentadecyl phenol.
[1] Data transformed by the optimal Box-Cox power method with $\lambda = \arcsin \sqrt{(x/100)}$.
[2] Determined by the formula of Schneider-Orelli (1947).
[3] Variation Coefficient. Source: Authors.

Factors such as time, application method, and dosage may have influenced the penetration and toxicity of the Abamectin® active ingredient and the other treatments. Because of these factors, the results in the present study were contradictory to those found in the literature, in which the use of the commercial product described presented 100% mortality (Figure 4) in larvae of *L. sativae* (Araujo et al., 2012). The case of *L. sativae* resistance to Abamectin® has been reported in California and Georgia (Ferguson, 2004). The study by Wei et al., (2015) evaluated the resistance of *L. sativae* to Abamectin under laboratory conditions 39 times after 20 cycles of selection and 25 generations.

Figure 4 - Presentation of melon leaves after 48 h of application (A) Abamectin® 5 mL/10 L of water and cardanol derivatives emulsified at a concentration of 1% (v/v): (B) benzoylated; (C) acetylated and, (D) methylated 3-pentadecyl phenol under the conditions of item 2.3 (used at approximately 20 cm, it was possible to spray the entire 10 cm diameter without wasting the applied volume, which remained at 5mL).



(A) **(B)** **(C)** **(D)**

Source: Authors.

The ineffectiveness of the molecules synthesized against the *L. sativae* during the development period could be correlated with the low activity of (Figure 4B) benzoylated, (Figure 4C) acetylated and (Figure 4D) methylated 3-pentadecyl phenol, that was verified by the low effect of these active principles at the beginning of the pest biological development process (Celin et al., 2017; Ware & Whitacre, 2004). The presence of mines on the leaves sprayed with the three products derived from cardanol and the use of the commercial product (Abamectina®) indicated that the molecules derived from cardanol (1% emulsion) did not inhibit 100% of the oviposition of the *L. sativae*. However, the methylated 3-pentadecyl emulsion showed a similar behavior as the effect of Abamectin® on the confinement test, with 53.5% and 65.4% repellence, according to the index preference for the oviposition index (IPO). In comparison, in the choice test the three products derived from cardanol had a deterrent effect superior to the Abamectin® effect, around 45-60%, against adults' action on the melon leaves. According to the values obtained by IPO, all treatments showed deterrence for the *L. sativae* oviposition (Table 2).

Table 2 - Mines/leaves of melon and index of preference for oviposition (IPO), after the application of each emulsion at 1% v/v (benzoylated 3-pentadecylphenol, acetylated 3-pentadecylphenol, methylated 3-methyl pentadecylphenol), Abamectin® (50 mL/100 L of water), control (distilled water), and, blank (Tween 20 in distilled water; 0.023% v/v).

	No choice			With choice		
Treatments	**Mines / leaf *[1]**	**IPO[2]**	**Classification[3]**	**Mines/leaf*[1]**	**IPO[2]**	**Classification[3]**
Benzoylated (*)	17.0 ± 8.1 a	-34.3	Deterrent	13.2 ± 12.9 a	-45.0	Deterrent
Acetylated (*)	25.0 ± 17.9 a	-16.3	Deterrent	12.5 ± 9.0 a	-47.3	Deterrent
Methylated (*)	10.5 ± 7.5 a	-53.5	Deterrent	9.0 ± 9.5 a	-59.0	Deterrent
Abamectin®	7.2 ± 2.0 a	-65.4	Deterrent	27.2 ± 18.4 a	-12.2	Deterrent
Blank	23.2 ± 7.6 a	-19.8	Deterrent	35.0 ± 22.2 a	0.0	Neutral
Control	34.7 ± 19.5 a	0.0	Neutral	35.0 ± 22.2 a	0.0	Neutral
VC[4]	25.5%			36.0%		

*Averageses followed by the same letter in the columns, do not present differences by the F test at 5% probability.
(*) Benzoylated 3-pentadecyl, acetylated 3-pentadecyl and methylated 3-pentadecylphenol.
[1]Data transformed by the Box-Cox optimal power method with λ = ln x.2
[2]IPO=[(T-P)/(T+P)]x100.
[3]This varied from +100 (very stimulating) to –100 (totally deterrent), with 0 being the value indicative of neutrality (Fenemore, 1980)
[4]Variation Coefficient.
Source: Authors.

As regards the insecticidal effect on eggs and nymphs, significant differences were observed between the control, blank, and the commercial product evaluated (Table 3). Mospilan® showed ovicidal activity (99.8%) in comparison to other treatments; nevertheless, molecules derived from cardanol (1% emulsion) showed intermediate mortality rates, in eggs at 6.5 - 13.5%, without statistical differences to control and blank. In the case of the nymphal activity, low efficacy values were obtained (0-5.3%), and the commercial product showed intermediate averages relative to the benzoylated, acetylated, and methylated products of 3-pentadecyl, thus exhibiting significant differences from the control and blank. The insecticidal effect was considered effective when the control percentage was higher than 80% (Cock et al., 1995), emulsions at 1% v/v (benzoylated 3-pentadecylphenol, acetylated 3-pentadecylphenol, methylated 3-methylpentadecylphenol) were considered ineffective in eggs and nymphs with the percentage below 80%. The insecticidal inefficiency may be due to the concentration of its products derived from cardanol, since Lomonaco et al., (2009) showed insecticidal activity of LCC products against Aedes aegyptis when they tested the CNSL, cardol, cardanol, and their hydrogenated analogs, as well as other studies already reported (Carvalho et al., 2019; Kubo et al., 2003; Kubo et al., 1999).

Table 3 - Mortality of egg and nymph stages of *B. Tabaci*, in melon, after applying the treatments. The infestation was caused by a period of 3h for oviposition and, counting of the eggs on the underside of the leaflets.

Treatments	**Egg / Leaf *[1]**	**Mortality*[2]**	**Efficiency [3]**	**Nymph/Leaf ***	**Mortality*[2]**	**Efficiency [3]**
Benzoylated [(*)]	136.0 ± 0.1 b	9.9 ± 7.9 ab	7.7	124.0 ± 1.5 bc	0.8 ± 0.9 ab	-4.7
Acetylated [(*)]	263.6 ± 0.2 ab	7.9 ± 13.5 ab	5.7	249.2 ± 3.9 b	0.1 ± 0.2 ab	-5.5
Methylated [(*)]	484.4 ± 0.0 a	6.7 ± 6.5 ab	4.4	452.8 ± 1.8 a	1.0 ± 0.9 ab	-4,5
Mospilan®	662.4 ± 0.2 a	99.8 ± 0.3 b	99.8	1.6 ± 0.5 c	0.0 a	-5,6
Blank	173.8 ± 0.2 b	2.4 ± 2.3 a	-	170.4 ± 3.2 b	1.6 ± 1.1 ab	-
Control	181.8 ± 0.3 b	1.4 ± 1.2 a	-	178.6 ± 3.1 b	5.3 ± 1.3 b	-
VC[4]	8.1%	66.2%		19.9%		

[*] Averages followed by the same letter in the columns, do not present differences by the F test at 5% probability.
[(*)] Benzoylated 3-pentadecyl, acetylated 3-pentadecyl, and methylated 3-pentadecyl phenol
[1] Data transformed by the Box-Cox optimal power method, with λ = 10 x log (number of eggs/leaf).
[2] Nonparametric data, null hypothesis is rejected at the level of 5% statistics made with the test of KRUSKAL-WALLIS (Silva & Azevedo, 2009).
[3] Determined by the formula of Schneider-Orelli, (1947).
[4] Variation Coefficient.
Source: Authors.

After application of the treatments in the confinement test at 6, 24 and 48 h, it was observed that the deterrence of the products derived from cardanol was significantly different from the number of individuals in the leaves containing the commercial product Mospilan®, presenting significant differences in relation to control and blank (Table 4). Although plants sprayed with benzoylated, acetylated and methylated 3-pentadecylphenol did not show differences in their average values compared to the control and blank after six hours of application, they presented intermediate values relative to the commercial product after 24 and 48 h, indicating that they had been less efficient than the Mospilan® in relation to *B. tabaci* repellency.

As we evaluated the number of whiteflies on the melon leaves in the range of 6, 24 and 48 h after spraying of the products in the free-choice test, significant differences were found. Mospilan®, in general, showed greater effectiveness in the preventive control of the pest, with noted differences in relation to the control and blank. The acetylated and methylated had similar characteristics as Mospilan®, in the range of six hours of application, and the benzoylated already had an intermediate value relative to the commercial product, but there were no differences in relation to the control. At 24 and 48 h, the benzoylate product showed an intermediate value relative to the other two products derived from cardanol.

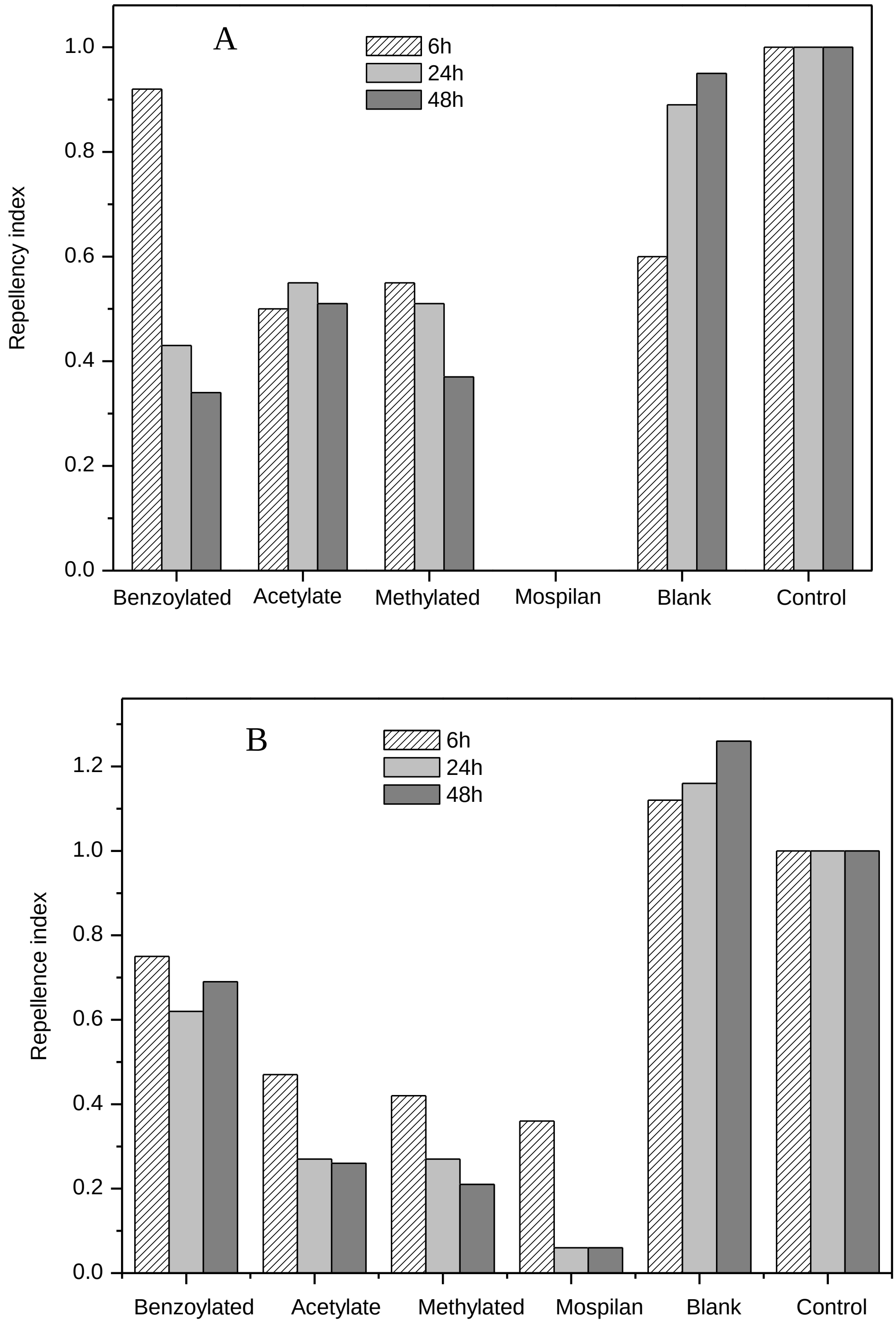
Table 4 - Average number (± SD) of adults of *B. Tabaci* attracted by sprayed melon leaves previously sprayed with different emulsion at 1% v/v (benzoylated 3-pentadecylphenol, acetylated 3-pentadecylphenol, methylated 3-methyl pentadecylphenol), Abamectin® (50 mL/100 L of water), control (distilled water), and, blank (Tween 20 in distilled water; 0.023% v/v) in different confinement and, free-choice test.

Treatments	**No choice**			**With choice**		
	6:00 h*	**12:00 h***	**48:00 h***	**6:00 h*[1]**	**12:00 h*[1]**	**48:00 h*[1]**
Benzoylated (*)	4.5 ± 5.8 a	3.7 ± 6.2 ab	2.7 ± 4.8 ab	11.7 ± 1.7 ab	11.1 ± 17.1 bc	12.5 ± 4.4 bc
Acetylated (*)	1.7 ± 0.9 a	5.2 ± 4.6 ab	4.5 ± 4.3 ab	6.0 ± 4.3 b	4.0 ± 2.9 cd	3.5 ± 2.8 cd
Methylated (*)	2.0 ± 4.0 a	4.7 ± 3.5 b	3.0 ± 1.8 ab	5.2 ± 4.0 b	4.0 ± 1.4 cd	2.7 ± 2.2 cd
Mospilan®	0.0 b	0.0 a	0.0 b	4.2 ± 0.9 b	0.7 ± 0.9 d	0.7 ± 0.5 d
Blank	2.2 ± 2.0 a	11.0 ± 6.3 b	12.0 ± 6,3 a	24.7 ±14.7 a	36.0 ± 21.2 a	40.2 ± 24.1 a
Control	5.2 ± 3.7 a	13.7 ± 10.4 b	13.3 ± 11.6 a	19.5 ± 5.2 a	26.0 ± 8.1 ab	26.2 ± 6.9 ab
VC[2]	84.7%	53.0%	58.4%	27.3%	28.0%	33.7%

* Averages followed by the same letter in the columns do not present differences by the F test at 5% probability.
(*) Benzoylated 3-pentadecyl, acetylated 3-pentadecyl and methylated 3-pentadecyl phenol.
[1] Data transformed by the Box-Cox optimal power method with $\lambda = \ln x$ (6 hours), $\lambda = \sqrt{(x+k)}$, k=0.1 (24 and 48 hours).
[2] Variation Coefficient.
Source: Authors.

The repellency indexes (RI) obtained after 6, 24 and 48 h of the application of the treatments by the non-choice method were less than one (RI < 1) for all treatments, except for the RI of benzoate after six hours that can be considered neutral. The Mospilan® stood out from the others, presenting total repellency on the adult *B. tabaci* in all evaluations (Figure 5A). The RI obtained after 6, 24 and 48 h by the choice method was less than one (RI < 1) for all treatments. The acetylated and methylated showed a similar efficacy as Mospilan®, while the RI of the benzoylate presented intermediate characteristics relative to the commercial product. In general, corresponding to the molecules synthesized showed repellency to *B. tabaci* (Figure 5B).

Figure 5 - Benzoylated 3-pentadecyl, acetylated 3-pentadecyl and methylated 3-pentadecylphenol repellency index (RI - no choice) in adults of *B. Tabaci* after 6, 24 and 48 h of application by the (A) nonchoice, and (B) choice method.



Source: Authors.

When comparing the average number of *B. tabaci* eggs in the melon leaves in the two bioassays, all the synthesized products were considered deterrents, with emphasis on the methylated product with IPO higher than 80 (0.3 eggs/cm^2), unlike controls (3.76 and 4.01). eggs/cm^2), which had the highest averages of deposited eggs (Table 5).

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Table 5 - Average number of eggs/cm^2 of *B. Tabaci* counted in leaves of melon and index of preference for oviposition (IPO) obtained after 48 h of the application of insecticides by the method without choice and with choice.

Treatments	No choice			With choice		
	Eggs/cm^2	**IPO[1]**	**Clasification[2]**	**Eggs/cm^2**	**IPO[1]**	**Clasification[2]**
Benzoylated (*)	1.7	-40.0	Deterrente	2.0	-20.0	Deterrente
Acetylated (*)	1.5	-32.4	Deterrente	0.7	-66.1	Deterrente
Methylated (*)	0.3	-82.3	Deterrente	0.3	-85.5	Deterrente
Mospilan®	0.0	-100	Deterrente	0.1	-94.1	Deterrente
Blank	2.2	-13.6	Neutral	2.5	-7.1	Neutral
Control	3.7	0.0	Neutral	4.0	0.0	Neutral

(*)Benzoylated 3-pentadecyl, acetylated 3-pentadecyl and methylated 3-pentadecylphenol
[1]IPO = ((P-T))/((T + P))]*100.
[2]The values vary from + 100 (very stimulating) to -100 (total deterrence), 0 being the value indicative of neutrality (Silva et al., 2012).
Source: Authors.

Methylated 3-pentadecyl product was similar to the commercial product in the two repellency and deterrence tests of cardanol derivatives and therefore is also less preferred for oviposition. Benzoylated and acetylated maintained intermediate characteristics in relation to the treatments and were considered efficient for the deterrence in oviposition. The study by (Santos et al., 2010) reported that methylation of hydroxyl group could lead to an increase in insecticidal potential, in addition, other studies state that the biological activity of phenols increases with the addition of alkyl groups, by making them more amphiphilic (Kubo et al., 2003; Nagabhushana et al., 2002).

During the evaluation of the deterrent effect in the confinement tests and with a chance of choice for the *B. tabaci*, an increase in the number of attracted adults after 24 h was observed due to the polymerization of the substances and, consequently, to the reduction of the repellent activity in the *B. tabaci*. It was likely that, after the first day of application, the treatments showed a reduction in their effects due to the propensity of the substances to polymerization, thereby decreasing their bioactivity and attracting a larger number of adult *B. tabaci*.

The oviposition behavior of the specie *B. tabaci* with the treatments applied suggests that these products are polar compounds that can serve as deterrents for oviposition. According to Arab & Bento, (2006), deterrent substances prevent the insect from continuing its oviposition due to the presence of volatiles. Based on the IPO values obtained, all the synthesized products were deterrent, compared to the control.

4. Conclusion

The synthetic products did not present insecticidal activity for the control of the larvae of the *L. sativae* and the eggs and the nymphs of the *B. tabaci*. However, the 1% 3-pentadecylphenol emulsion showed greater deterrence efficiency for the oviposition of the mentioned pests. Methylated 3-pentadecyl showed a similar behavior as the effect of Abamectin® on the confinement test, with 53.5% - 65.4% repellence of adults for the values obtained by the IPO showed deterrence for the *L. sativae* oviposition in all treatments. The structural modifications in the side chain of the phenolic lipid can lead to different modes of action and mutual synergistic relations, bearing a more specific evaluation of the mutual relations of the individualized effect of each modification is of paramount importance to strengthen the perspectives of the analogs of cardanol as an active ingredient environmentally. It is therefore important to emphasize that the products synthesized from cardanol have shown promise for the preventive management of the studied pests and are suitable for use in integrated pest management programs, although it is necessary to carry out studies of related chemical structures to improve its effectiveness as well as evaluating the selectivity of the emulsions concerning other non-target pests.

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Statements and Declarations

The authors declare no conflict of interest. The founding sponsors had no role in the design of the study; in the collection, analysis, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results. The authors contributed equally to the manuscript.

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