Essential oil microencapsulated fumigant: an innovative technology against *Zabrotes subfasciatus* (Boheman, 1833) (Coleoptera: Chrysomelidae: Bruchinae)

David Jossue Lopez Espinosa
Karen Oliveira de Menezes
Alice Maria Nascimento Araújo
Valdemir da Costa Silva
Irinaldo Diniz Basílio Júnior
Roseane Cristina Predes Trindade
rcpt@ceca.ufal.br

https://orcid.org/0000-0001-5714-9453

Short Report

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Abstract

The fumigating effect of essential oils on the control of stored-grain pests has been widely studied. However, bioactive compounds with low molecular weight, high volatility, and sensitivity to light and heat; thus, the development of new technologies, such as the microencapsulation process, is needed to improve their use in agriculture. The objective of this research was to develop and characterize a microencapsulated fumigant based on citronella essential oil (MFCEO), *Cymbopogon nardus* L., for the management of *Zabrotes subfasciatus* (Boheman, 1833) (Coleoptera: Chrysomelidae), one of the main storage pests of beans. The resulting microparticles were a white powder with 100–1300 μm, 3.29% moisture content, a bimodal distribution, and smooth circular particles. The fumigant activity showed LC_{50} 0.46 $g.L^{-1}$ and 6.49 $\mu L.L^{-1}$ of air and LC_{99} of 7.6 $g.L^{-1}$ and 12.94 $\mu L.L^{-1}$ of air for MFCEO and essential oil, respectively. Thus, this is the first report of a MFCEO for the management of *Z. subfasciatus*.

INTRODUCTION

The insecticidal potential by fumigant action for citronella essential oil (CEO), *Cymbopogon nardus* L. (Poaceae) in stored-grain pests, as well as its insecticidal and repellent activity against *Zabrotes subfasciatus* (Boheman, 1833) (Coleoptera: Chrysomelidae), considered the main pest responsible for reducing the commercial value of grains in the post-harvest (Brito et al. 2019; Mesele et al. 2019; Caballero-Gallardo et al. 2021), has been reported as a viable alternative in pest control.

Essential oils consist of bioactive compounds with low molecular weight, highly unstable and sensitive to oxygen, light, moisture, and heat (Majeed et al. 2015); therefore, the development of improvement methods is needed it increase their use in agriculture.

Microencapsulation can be used to increase the efficacy of essential oils because it can keep the active ingredient inside a core, with the volatilization of aromatic compounds controlled by means of particles designed to gradually release the compounds, diffusing the matrix of fully biodegradable particles (Workman et al. 2020).

Therefore, this research aimed to develop and characterize a microencapsulated fumigant based on citronella essential oil (MFCEO), *C. nardus*, to manage *Z. subfasciatus*. The MFCEO was characterized by the size and humidity of the microparticles, polydispersity index, morphological analysis, thermogravimetric analysis (TGA), and scanning electron microscopy (SEM).

MATERIAL AND METHODS

The CEO was purchased from Quinarí®, Brazil, and chemically analyzed through gas chromatography coupled with mass spectrometry (GC/MS). Components were identified by Wiley 7 lib and NIST 08 lib database and Kovats Retention Index (El-Sayed 2011).

Microencapsulation was performed in the spray dryer device model LABMAC MSD 100, at an inlet temperature of 180°C, air flow of 80, and rotation speed of 25 rpm (Nascimento et al. 2022a). The encapsulating agents (maltodextrin, gum arabic, and starch) corresponded to 80% and the CEO to 20%, in addition to 300 mL of absolute ethyl alcohol P.A. and distilled water. The proportion of each encapsulation was based on the density of the essential oil (0.881 mg), corresponding to maltodextrin (48%), starch (24%), and gum arabic (8%).

The size and polydispersity index of the microparticles were determined by the dynamic light scattering technique, wherein 10 mg of the MFCEO was solubilized in 10 mL of distilled water, thus obtaining a stock solution of 1 mg/mL. The moisture content was obtained by the gravimetric method, using the electronic moisture balance modelShimadzu-MOC-120H ®, with a precision of 1 mg and an infrared dryer.

The thermogravimetric curves of the MFCEO and encapsulating agents were obtained using the Shimadzu device model TGA50H at the Instrumental Analysis Laboratory of the Chemistry Coordination of the Federal Institute of Alagoas with an initial sample of 5.0 ± 0.5 mg. Thermoanalytical data were analyzed using the TA-60WS Collection Monitor software (Nascimento et al. 2022b).

A sample of the MFCEO powder was covered with gold by the QUORUM metallizer Q150R ES and submitted to a SEM, model TESCAN VEGA3, with a working distance of 15.97 mm and voltage acceleration equal to 20 kV (Maciel et al. 2019).

For the fumigation test, chambers consisting of glass containers with 1.3 L capacity were used, containing 20 adult *Z. subfasciatus*, non-sexed, 0 to 24 h old in 40 g of beans. The CEO treatments were fixed inside the lid of the containers, while for the MFCEO treatments, the powder was placed in a Polypropylene-TNT sachet, both at the top of the arena. After 48 h, the percentage of mortality was evaluated for the treatments.

Then, preliminary tests with different concentrations were carried out to determine the Upper Limit (LS) with approximately 95% mortality and Lower Limit (LL) with approximately 5% mortality, using the Bliss formula (1934). The following concentrations were tested for the CEO (3.66, 4.48, 5.47, 7.0, 8.17, and 10.0 µL), the MFCEO (0.050, 0.082, 0.135, 0.222, 0.36,5 and 0.600 mg) and the highest concentration of the MFCEO containing only the polymers (white test). The data were subjected to Probit analysis (SAS program) to estimate the lethal concentrations of LC_{50} and LC_{95}.

RESULTS

The chromatographic profile of the CEO found the following major compounds: Citronellal (47.12%), Citronellol (18.56%), and Geraniol (11.07).

The MFCEO was a fine white powder with 3.29% moisture content, yielding 48.0%. The polydispersity index was equal to 0.365. The MFCEO particles had a bimodal distribution, with a diameter of 100–1300 μm. The thermal stability studies presented five decomposition events: (I) from 25 to 150°C, with mass loss of 6.48%; (II) from 150 to 305°C, with mass loss of 38.63%; (III) from 305 to 450°C, with mass loss of 33.85%; (IV) from 450 to 720°C, with mass loss of 15.82%; (V) from 720 to 900°C, with mass loss of 0.1%. The total analysis showed a mass loss of 94.88%, leaving 5.12% ash content. The SEM micrographs illustrate the spherical-shaped microparticles, agglomerated in different sizes, with predominantly smooth surfaces, without apparent cracks (Fig. 1).

The CEO and MFCEO treatments showed fumigation toxicity for *Z. subfasciatus* adults, verified by the estimates of lethal concentrations, which fit the probit model (P = < .0001): LC_{50} of 6.49 $\mu L.L^{-1}$ of air and LC_{99} of 12.94 $\mu L.L^{-1}$ of air for CEO; and LC_{50} 0.46 $g.L^{-1}$ and CL_{99} of 7.6 $g.L^{-1}$ and, for MFCEO, respectively. The assay only with the polymers did not exhibit any fumigant effect.

DISCUSSION

The analysis of MFCEO through PDI, particle size and diameter, TGA, and SEM micrographs showed that this is a promising formulation due to its release of volatile chemical compounds even after the high-temperature process of the spray dryer. Therefore, the process was registered as the patent BR 1020240153235.

The protection resulting from the microencapsulation of essential oils can increase the stability of the compounds, which makes the active material more effective and allows its use at low concentrations (Veiga et al. 2019).

The control of MFCEO was neutral to Z. *subfasciatus*, showing that the coating materials used in the microencapsulation process do not interfere with the insect’s behavior and confirming that the essential oil caused the fumigation. The choice of the coating material can guarantee efficient encapsulation and stability of the microcapsules. The physical-chemical properties of the material to be encapsulated and the desired properties of the microcapsules must considered (Gharsallaoui et al. 2007).

This is the first report of a microencapsulated fumigant of spray-drying citronella essential oil for *Z. subfasciatus* management.

Declarations

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Competing Interests

The authors declare no competing interests.

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Figures

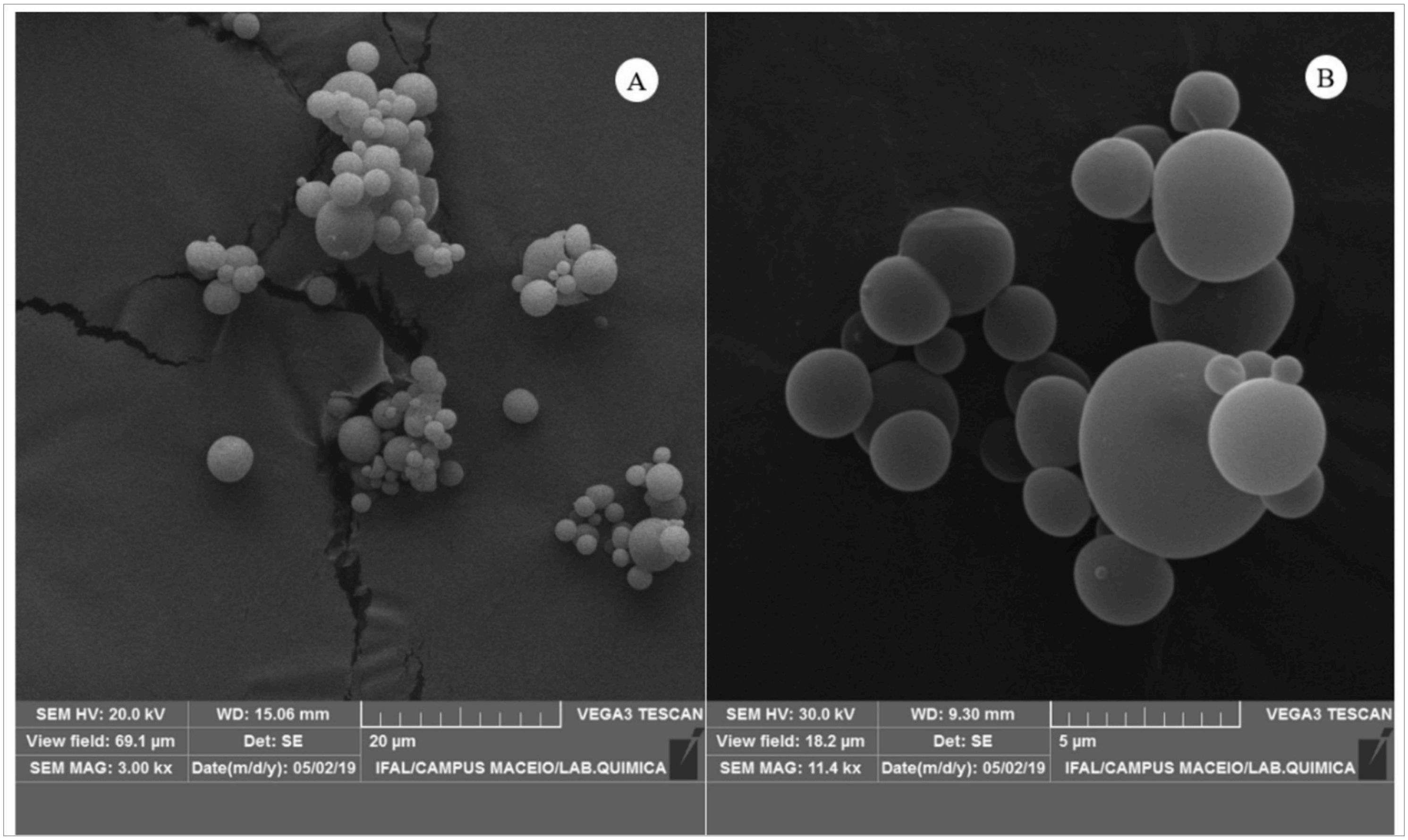

Figure 1

Photomicrographs of microencapsulates of *Cimbopogon nardus* essential oil: **A.** shapes of the microcapsules 3000× and **B.**surface of microcapsules 11400×.