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# Preparation and evaluation of pheromone slow-release dispensers of grape vine moth and brinjal fruit and shoot borer

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**Preparation and evaluation of pheromone slow-release dispensers of grape vine moth and brinjal fruit and shoot borer**

**Abstract:** The inclination towards pest free production in agriculture pose a greater challenge to farmer practices. Mating disruption (MD) has become an integral component of the IPM system which interferes with insect mating. MD of Lepidopteran insects using active and passive dispensers with various pheromone loads and release patterns are reported. The longevity of passive dispensers with mesoporous silica nanoparticles (MSN), carbon nanospheres (CNS) and their 1:1 admixture embedded with synthetic pheromones were evaluated. The synthesized MSN and CNS showed no agglomeration as evident from Zeta potential of -16.8 mV and -42.12 mV respectively. The blend of dispenser materials showed the highest EE (entrapment efficiency) and LC (loading capacity) with respect to (E, Z)-7,9-dodecadienyl acetate and (E)-11-hexadecenyl acetate, in the range of 90-92 % and 45–46 % respectively. The residual pheromone analysis of slow-release dispensers showed 47-57 % release of brinjal fruit and shoot borer pheromone and 89-90 % *Lobesia* pheromone release in 120 days and in comparison, pheromone wax emulsions showed 80 % release in 40 days. Statistical analysis of variance showed significant difference in release of *Lobesia* from different dispenser materials. The high $R^2$ for first order kinetics with 92 % EE supports slow release of pheromones from the selected materials.

**Key words:** synthetic pheromone, entrapment efficiency (EE), integrated pest management (IPM), mating disruption (MD), mesoporous silica nanoparticles (MSN), carbon nanospheres (CNS).

**Priprava in vrednotenje počasnih feromonskih razpršilcev pri zatiranju trtnega molja in brinjalskega sadnega in poganjkovega zavijača.**

**Izvleček:** Strmenje k pridelavi brez škodljivcev predstavlja v kmetijstvu vse večji izziv za kmetijsko prakso. Motnja parjenja (MD) je postala sestavni del sistema integriranega uravnavanja škodljivcev (IPM), ki moti parjenje žuželk. Obstajajo poročanja o motnjah parjenja žuželk iz reda Lepidoptera, kjer se uporabljajo aktivni in pasivni razpršilniki z različnimi vsebnostmi in vzorci sproščanja feromonov. Ocenjena je bila dolgoživost pasivnih razpršilnikov z nanodelci mezoporoznega silicija (MSN) z ogljikovimi nanosferami (CNS) in njihove mešanice v razmerju 1:1 z vključenimi sintetičnimi feromoni. Sintetizirana MSN in CNS nista pokazala kopičenja kot je razvidno iz Zeta potenciala -16,8 mV oziroma -42,12 mV. Mešanica materialov razpršilnika je pokazala največjo učinkovitost ujetja (EE) in obremenitveno zmogljivost (LC) glede na E, Z-7,9-dodekadienil acetat in E-11 heksadecenil acetat, v območju 90–92 % oziroma 45–46 %. Analiza ostankov feromonov v razpršilnikih s počasnim sproščanjem je pokazala 47-57 % sproščanje feromona brinjalovega zavijača plodov in poganjkov ter 89-90 % sproščanja feromona vrste iz rodu *Lobesia* v 120 dneh, za primerjavo pa so emulzije feromonskega voska pokazale 80 % sproščanje v 40 dneh. Statistična analiza variance je pokazala pomembno razliko v sproščanju fermonov vrste iz rodu *Lobesia* iz različnih materialov za doziranje. Velik $R^2$ za kinetiko prvega reda z 92 % EE podpira počasno sproščanje feromonov iz izbranih materialov.

**Ključne besede:** sintetični feromon, učinkovitost ujetja (EE), integrirano zatiranje škodljivcev (IPM), motnje parjenja (MD), mezoporozni nanodelci silicija (MSN), ogljikove nanosfere (CNS).

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## 1 INTRODUCTION

The integrated pest management (IPM) approach using behavior modifying chemicals like pheromones for mating disruption (MD) has been fruitful in the last two decades to control pest population in fruit and vegetable crops (Ioriatti & Lucchi, 2016, Huang et al., 2024) compared to the conventional methods using pesticides (Riegel, 2021; Van Dijk et al., 2021). To reduce the implementation costs of this technique research work on the optimization of the pheromone used, quantity and release mode are essential.

Pheromones are vulnerable to degradation when exposed to the atmosphere unless protected. The development of pheromone formulations involves considerations such as cost, frequency of dispenser replacement, and their sensitivity to elevated temperatures (Mafra-Neto et al., 2014).

Use of carrier material is the choice in formulation development. Among different carriers employed to dispense pheromones plastic dispensers, aerosol devices, rubber septa, rope dispensers, wax emulsions (Atterholt, 1999), sprayable dispensers, natural gums as wall forming material (Chen et al., 2007), polyethylene vials (Chamberlain et al., 2000) were extensively used. These carrier materials present issues such as high costs, increased need for source points, and frequent refilling depending on the dispenser type (Ortiz, 2021; Johansson, 2001). All current dispensers require large amounts of pheromone for season-long pest control, substantially raising deployment expenses (Vacas et al., 2015).

Wax preparations as emulsions or as SPLAT formulations were effective in controlling yellow stem borer affecting rice (Badari Prasad et al., 2022), pink boll worm infesting cotton (Sreenivas et al., 2021), oriental fruit moth affecting apple orchards (Stelinski et al., 2006, Atterholt et al., 1999), mealybug affecting tangerine and apple orchards with source points ranging from 750 (Ballesteros et al., 2021) to 1000 (Badari Prasad et al., 2022). The efficacy of these formulations was found to be within 52 to 100 days (Atterholt, 1999; Stelinski et al., 2006). Various formulations have been developed as sprayable agents, providing the benefit of convenient application (Desauziers et al., 2022). These formulations have demonstrated effective release periods ranging from two weeks to one month (Knight et al., 2004; Il'ichev, 2006).

A versatile method for controlling these pest populations is using pheromone-based passive dispensers that provide an effective way to control pest populations (Evenden, 2016). Commonly, hand-applied dispensers such as twist tie ropes, twin ampoules, and membranes are used; these plastic or silicone rubber containers are filled or impregnated with pheromones that are passively released through their walls (Ferrer, 2011).These dispensers with a LC ranging from 200-500 mg release the pheromone completely in 56-72 days (De Lame et al., 2007; Lo Verde et al., 2020).

*Lobesia botrana* ([ Denis & Schiffermüller,1775]) affects grape berries during all three generations and can facilitate secondary infections by other pests such as *Botritis cinerea* Pers.(1794), leading to quality issues and economic losses of up to 20–30 % (Guidotti, 2023). According to Vassiliou (2011), 56–69 % of damage occurs to grape berries if not properly treated. MD is a widely adopted strategy for managing the European grapevine moth (*Lobesia botrana*, Denis and Schiffermüller; Lepidoptera: Tortricidae) through the deployment of passive dispensers or aerosol devices. Current MD methods utilize various dispenser types, including aerosol devices (Lucchi et al., 2018), biodegradable dispensers (Anfora et al., 2008), polyethylene tube dispensers (Gordon et al., 2005), twist-tie rope and ampoule dispensers (Gavara et al., 2022), as well as nano emulsions (Benelli et al., 2020). The synthetic pheromone loading for *Lobesia botrana* in these dispensers ranges from 1.72 g (Ceballos et al., 2022), to 220–300 mg per dispenser (Gordon et al., 2005), and 170–210 mg in rope and ampoule dispensers (Gavara et al., 2022). Most currently available dispensers offer relatively high pheromone loading capacities.

*Leucinodes orbonalis* Guenée, 1854, known as the Brinjal fruit and shoot borer (BFSB), is a major pest in brinjal cultivation, affecting flowering, pod development, and inflorescence stages. It can cause up to 90 % yield loss or total crop failure if uncontrolled (Rahman MM, 2007). While traditionally managed with plant products (Gahukar, 2017) and traps (Alam et al., 2003), BFSB can be effectively controlled using pheromones (Jacob & Revathi, 2019; Mathur et al., 2012; Cork et al., 2005).

This study evaluated the release of synthetic pheromones for *Lobesia botrana* and BFSB from MSN and CNS. The effectiveness of slow-release dispensers was tested in semi-field conditions, demonstrating improved loading and sustained release for cost-effective pest management throughout the crop season.

## 2 MATERIALS AND METHODS

### 2.1 CHEMICALS

The pheromone of European grape vine moth (EGVM), *Lobesia botrana* '(E, Z)-7,9-dodecadienyl acetate) and BFSB, *Leucinodes orbonalis* 'E-11 hexadecenyl acetate' were obtained from ATGC biotech pvt. Ltd, Hyderabad (India). The other materials used like tetraethyl orthosilicate, pluronic 123 surfactant, HCl, $H_2SO_4$,

methanol, ethanol, DCM, ethyl acetate, glucose, butylated hydroxytoluene (BHT), polyvinylpyrrolidone K30 (PVP K30) are of analytical grade. All the chemicals and solvents obtained were used as received without further purification.

## 2.2 SYNTHESIS OF DISPENSER MATERIALS

### 2.2.1 Synthesis of MSN

Mesoporous silica was synthesized using tetraethyl orthosilicate as the precursor and pluronic 123 as the surfactant via the sol-gel method (Zhao, 1998; Zhu, 2011). Porosity was imparted using an economical method and the resulting MSN was characterized (Vakkanti and Guntuku,2024).

### 2.2.2 Synthesis of CNS

Hydrothermal carbonization was used to prepare CNS from Glucose. 100 g of glucose was dissolved in 300 ml of water by stirring until a clear solution was obtained. The pH of the solution was adjusted between 4-5 with concentrated $H_2SO_4$. The solution was transferred into a 2-liter autoclave and heated to 180 $^0$C for 4 h The obtained carbonaceous solid material was separated by centrifugation at 10000 rpm in 10 minutes. The resulting solid material was subjected to washing with water and ethanol for about 10 times. The obtained solid was dried in vacuum oven at 80 $^0$C for 12 h (Qi et al., 2014).

## 2.3 LOADING OF PHEROMONES IN DISPENSER MATERIALS

The loading of both actives, *Lobesia* pheromone and BFSB blend, was conducted using a solvent evaporation with solid dispersion method. Approximately 20 g of the active ingredient was diluted at a 1:1 ratio with DCM to facilitate dissolution of the pheromone. Dispersions of pheromone were prepared with at least 50 % pheromone concentration (Bansal, 2010; Zanoni et al., 2019). An antioxidant, BHT, was added at 1 % relative to the pheromone concentration. This mixture was referred to as Solution A. Solution B was made by granulating about 20 g each of MSN and CNS separately with 5 % PVP K30 as a binder. Solution A and Solution B were thoroughly combined to form a slurry, which was then sonicated for approximately 5 minutes at 30 °C. The solvent was removed by drying at 30 °C for 5 to 10 minutes. A 1:1 ratio of pheromone to material dispenser was used, consistent with RPW commercial lures. For further studies, 200 mg of each loaded particle sample was placed in polyethylene vials (PE) (Li et al., 2008).

### 2.3.1 Loading efficiency-preliminary analysis using TLC

TLC was utilized to evaluate the presence of sample loading. A single vial from each loaded material was gathered, then subjected to sonication by immersion in 10 ml of ethyl acetate for 15 minutes at 30 °C. The resulting supernatant was collected and analyzed using TLC. Precoated silica gel plates were appropriately marked. The TLC chamber was saturated with a solvent system consisting of hexane and ethyl acetate in a 9:1 ratio. The Rf value of the supernatant sample was compared to that of the standard, employing an iodine chamber as developing system.

### 2.3.2 LC and EE

A Randomized replicated factorial-mixture design was utilized to evaluate the LC and EE of MSN and CNS particle formulations and their mixtures at three ratios (1:1, 0.5:1, and 1:1.5). Each preparation was tested in triplicate (n = 3). The 1:1 ratio of loaded particles was found optimal for preparing the blend (Table 1). The LC and EE were calculated using the equation below according to Rabima & Sari (2019).

$$Loading\ Capacity(LC)\% = \frac{Amount\ of\ pheromone\ entrapped\ in\ the\ material}{(mass\ of\ the\ pheromone\ loaded\ particles\ )} X\ 100$$

$$Entrapment\ efficiency(EE)\% = \frac{Amount\ of\ pheromone\ entrapped}{Total\ pheromone\ added} X\ 100$$

## 2.4 CHARACTERIZATION OF PARTICLES SYNTHESIZED

The prepared and loaded materials, MSN and CNS were characterized for the functional groups using Fourier Transform Infrared (FTIR) spectroscopy (FT-IR -BRUKER) using KBr pellet method (1:100 w/w of the sample was taken with respect to KBr) in the scan range of 400–4000 cm$^{-1}$ at a resolution of 4 cm$^{-1}$.The surface morphology of materials before and after loading was analyzed using SEM (HITACHI S-3700 )at an accelerated voltage of 15 KV. The stability and loading efficiency of the materials was assessed by TGA (PerkinElmer TGA 8000) by taking 6.0 ± 0.2 mg of sample. The temperature was raised from about 40 °C to about 250 °C at a heating rate of about 10 °C min$^{-1}$. The nitrogen flow was about 40 ml min$^{-1}$. (Zanoni et al., 2019). The X-ray diffraction (XRD) patterns were measured at room temperature using X-ray diffractometer (Bruker AXS) (Nguyen et al., 2020). The stability of particles was assessed by determin-

ing the zeta potential using Zetasizer nanoS (Malvern Instrument, Malvern, UK). The particles were analyzed at 25 $^0$C using PBS as dispersion medium. Approximately 1 ml sample was placed in a cuvette for analysis (Malvern Instruments Ltd., 2012). Thin layer chromatography (TLC) was performed to confirm the presence of pheromones in the nanoparticles. These analyses were carried out on representative single samples.

## 2.5 INSTALLATION OF PE VIALS IN SEMI-FIELD CONDITIONS

A total of 200 mg of each type of loaded particle containing *Lobesia* pheromone and BFSB pheromone were dispensed into PE vials in triplicate. These vials were then deployed under semi-field conditions by placing them outdoors, exposed to direct sunlight on an elevated platform at a height of 0.8–1.0 meters to closely simulate field environments. Average weather parameters throughout the study period were documented, with data sourced from the Indian Meteorological Department (IMD) in Begumpet, Hyderabad (https://mausam.imd.gov.in). Control PE vials containing only pheromone without any carrier material were also set up in triplicate for comparative analysis. All lures were arranged in triplicate, and samples were collected at regular intervals of 0, 10, 20, 30, 40, 60, 90, and 120 days. The triplicate lures collected were preserved for subsequent analysis using gas chromatography.

## 2.6 ACTIVE QUANTIFICATION

Gas chromatography (GC) was used to quantify pheromone LC and EE in PE vials. Samples collected at regular intervals were stored at -20 °C for later analysis. Residual pheromone in different formulations was also quantified by GC.

### 2.6.1 Residual pheromone analysis by gas chromatography

To assess the residual pheromone content and analyze pheromone release from MSN, CNS and 1:1 admixture of MSN and CNS, a randomized replicated factorial-mixture design was employed. The residual pheromone content in each of the PE vials collected at regular time intervals was analyzed by gas chromatography. The technique was carried out using dodecyl acetate as internal standard and TGAI (Technical grade active ingredient) as reference standard. The GC equipment Agilent 8890 with flame ionization detector was used. It consists of an auto sampler fitted with Innowax column 30 m x 0.32 mm x 0.25-micron film thickness using split ratio 50:1 at 250 $^0$C at the injection port. Temperature was programmed at 100 °C for one minute and then the final temperature was raised to 250 °C at the rate of 10 °C min$^{-1}$ and held for 10 minutes using nitrogen as carrier gas. The total runtime was about 25 minutes. The residual amount of pheromone was expressed as percentage of the initial loading of active (Gavara, 2020).

### 2.6.2 Statistical analysis

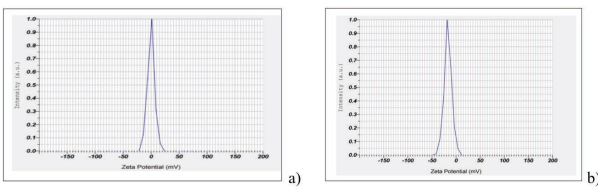
Statistical significance of material type and pheromone on LC and EE was assessed using two-way ANOVA. Release data for *Lobesia* and BFSB pheromones from three dispenser materials were fit to kinetic models (zero order, first order, Higuchi, Korsmeyer-Peppas). Kinetics were compared with control and proprietary wax formulations. Triplicate means and standard deviations were calculated; error bars indicate SD. The model with the highest $R^2$ was selected as best fit. Two-way ANOVA evaluated particle-pheromone effects on cumulative percent release over time, using a significance level of 0.05 and reporting 95 % confidence intervals when applicable. Tukey's HSD test performed post hoc pairwise comparisons. Analysis was carried out using Excel Analysis Tool Pak.

# 3 RESULTS AND DISCUSSION

## 3.1 CHARACTERIZATION OF DISPENSER MATERIALS

### 3.1.1 Zeta potential

MSN showed a zetapotential of -16.8 mV, indicating particle repulsion that enhances mesoporous stability. CNS showed a zetapotential of -42.12 mV. These negative values prevent agglomeration and burst release, supporting controlled release (Tao et al., 2018) and confirming the materials suitability as slow-release carriers. Figures 1a and 1b illustrate these results.




a) b)

**Figure 1:** Zeta potential of prepared MSN (a) and (b) CNS

### 3.1.2 XRD of CNS

Figure 2 presents the XRD analysis of CNS within the scan range of 10-80$^0$, displaying a characteristic peak at 2θ 220 (Ba et al., 2021), which suggests that CNS may be appropriate for handling pheromone load.

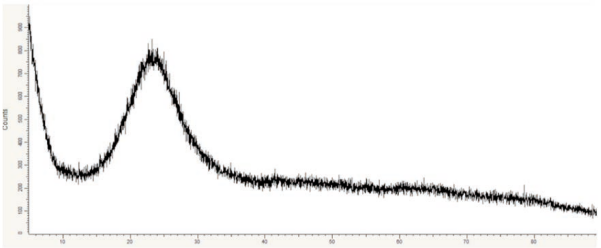


**Figure 2:** XRD pattern of CNS

### 3.1.3 FTIR of CNS

The FTIR spectra of CNS in Figure 3 display a peak at 1701 cm$^{-1}$ for C=O stretching (carboxyl groups), a broad band at 2950–3650 cm$^{-1}$ for OH stretching (hydroxyl groups), a 1359 cm$^{-1}$ peak for -C-O stretching in carboxyl, a 799 cm$^{-1}$ peak for -C-H deformation, and a 1608 cm$^{-1}$ peak indicating the carbon backbone. These results confirm typical functional groups present in CNS.

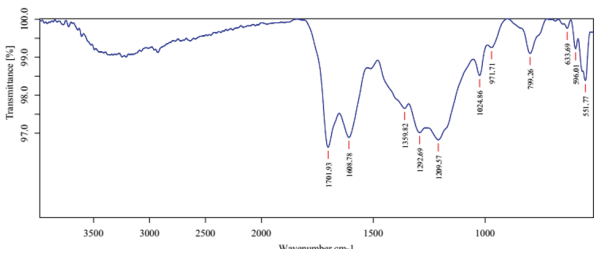



**Figure 3:** FTIR spectra of synthesized CNS

### 3.1.4 Characterization of active loading using FTIR

#### *3.1.4.1 Lobesia* pheromone loading in MSN

Figure 4 demonstrates that the peaks at 2856, 2929, and 3016 cm$^{-1}$ correspond to the C-H stretching vibrations of the *Lobesia* pheromone. The peak observed at 1649 cm$^{-1}$ is indicative of C=C stretching vibrations, while the sharp peak at 1740 cm$^{-1}$ represents the C=O stretching of the pheromone. A broad band at 3404 cm$^{-1}$is attributed to the OH stretching vibration of the silanol group. The presence of a peak in the region of 1020–1070 cm$^{-1}$ signifies C-O stretching. After loading *Lobesia* pheromone into MSN, the vibration shifted from 1073 to 1058 cm$^{-1}$, which can be attributed to interactions between the characteristic siloxane and the pheromone. The FTIR spectra clearly confirms the successful loading of *Lobesia* pheromone into the mesoporous framework of the synthesized MSN.

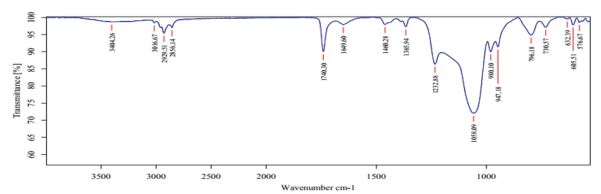



**Figure 4:** FTIR spectra of *Lobesia* pheromone loaded MSN

#### 3.1.4.2 BFSB pheromone loading in MSN

Figure 5 displays peaks at 2954, 2923, and 2853 cm$^{-1}$, indicating CH stretching in BFSB pheromone. A prominent peak at 1740 cm$^{-1}$ shows C=O stretching vibration characteristic of the pheromone. The peaks at 1645, 3378, and 1055 cm$^{-1}$ correspond to C=C, OH (silanol), and C-O stretching vibrations, respectively. The shift of the bond from 1073 to 1055 cm$^{-1}$ represents the interaction of the characteristic siloxane with the BFSB pheromone. Thus, the FTIR spectra shows the effective loading of the pheromone in MSN.

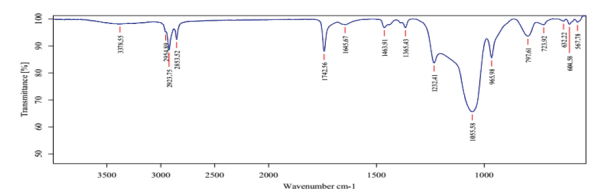



**Figure 5:** FTIR spectra of BFSB pheromone loaded with MSN

#### *3.1.4.3 Lobesia* pheromone loading in CNS

In figure 6 the peaks at 3013 2928 and 2858 cm$^{-1}$ correspond to C-H stretching vibration characteristic of

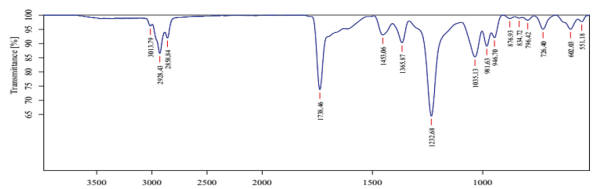



**Figure 6:** FTIR spectra of *Lobesia* pheromone loaded in CNS

*Lobesia* pheromone. The appearance of a sharp peak at 1738 $cm^{-1}$ is characteristic of -C=O stretching associated with *Lobesia* pheromone. The peak at 1035 $cm^{-1}$ depicts -C-O stretching vibration. Additionally, a peak at 1359 $cm^{-1}$ shows C–OH stretching vibrations characteristic of CNS. The characteristic peaks at 1365 and 796 $cm^{-1}$ reveal -C-O stretching vibration peak in carboxyl and -C-H deformity respectively, signifying the presence of CNS. Thus, the FTIR spectrum reveals the successful loading of *Lobesia* pheromone within CNS.

3.1.4.4 BFSB loading in CNS

The peaks at 2923 and 2854 $cm^{-1}$ indicate -CH stretching vibrations of the BFSB pheromone. Appearance of a sharp peak at 1741 $cm^{-1}$ represents -C=O stretching vibration, while 1233 $cm^{-1}$ corresponds to -C-O stretching in the carbonyl group. A peak at 1365 $cm^{-1}$ shows C–OH stretching vibrations characteristic of CNS. The C=C vibration peak shifted from 1645 to 1614 $cm^{-1}$ and decreased in intensity, indicating pheromone entrapment within CNS. Thus, the FTIR spectrum in Figure 7 confirms successful loading of BFSB pheromone into CNS.

### 3.1.5 Thermogravimetric studies

3.1.5.1 TGA of MSN and CNS

Figures 8a and 8b show no significant inflection point in the particle thermogram, indicating strong stability above 250 °C. Mesoporous silica exhibits no mass loss up to this temperature, while CNS lost less than 2 %, which is negligible.

3.1.5.2 TGA of *Lobesia* and BFSB synthetic pheromones

The thermal decomposition profiles of the TGAI of *Lobesia* and BFSB pheromones are illustrated in Figures 9a and 9b, respectively. The thermograms demonstrate distinct inflection points at 120 °C and 190 °C. The *Lobesia* thermogram exhibited an 80 % mass loss, whereas the BFSB thermogram showed a 35 % mass loss.

3.1.5.3 TGA of *Lobesia* loaded MSN and CNS

The MSN-pheromone thermogram shows a 40 % loss of mass (Figure 10a), versus 80 % without loading, indicating increased stability of the loaded *Lobesia* pheromone. The thermogram for carbon sphere-loaded *Lobesia* displays an inflection at 120 °C due to surface pheromone degradation (Figure 10b), and a major inflection

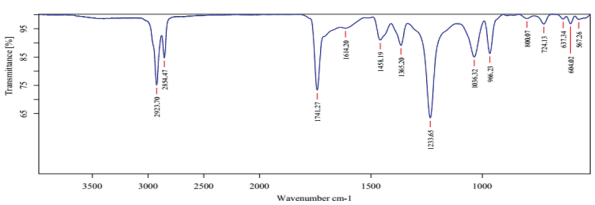



**Figure 7:** FTIR spectra of BFSB pheromone loaded in CNS

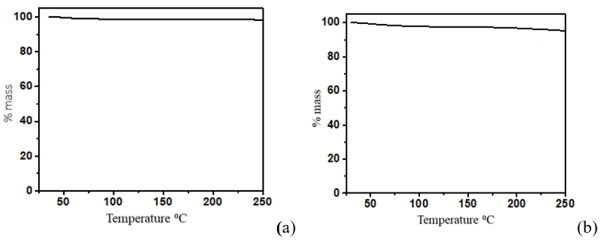



**Figure 8:**TGA thermograms of MSN(a) and CNS (b) showing stability

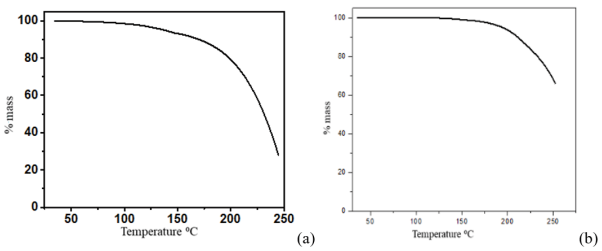



**Figure 9:** Comparison of TGA thermogram of *Lobesia* pheromone (a) and BFSB pheromone (b)

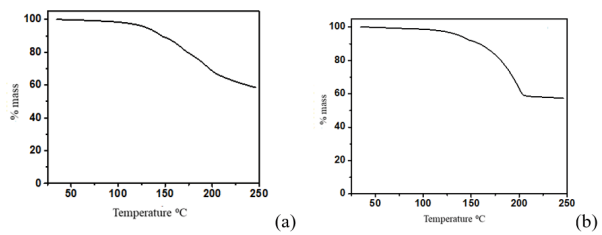



**Figure 10:** Comparison of TGA thermogram of *Lobesia* pheromone loaded in MSN(a) and CNS(b)

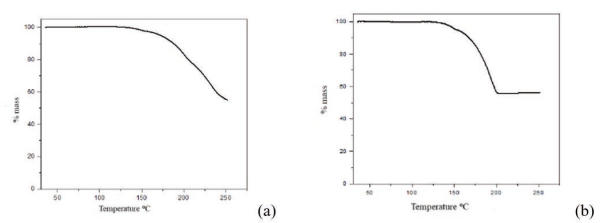



**Figure 11:** TGA curves of BFSB pheromone loaded in MSN (a) and CNS(b)

at 200 °C likely resulting from entrapped pheromone degradation.

3.1.5.4 TGA of BFSB pheromone loaded MSN and CNS

*Lobesia* pheromone-loaded MSN shows an inflection point above 150 °C, confirming pheromone stability (Figure 11a). CNS loaded with BFSB display two degradation steps (Figure 11b). SEM images of the loaded carbon show pheromones interacting with the surface of the spheres.

**3.1.6 SEM analysis**

3.1.6.1 SEM of CNS

The SEM image of CNS shown in Figure 12 displays a smooth, spherical arrangement with no apparent agglomeration.

3.1.6.2 SEM of loaded materials

SEM images reveal that mesoporous silica pores are blocked, indicating successful entrapment of pheromone within the hexagonal pores as seen in Figure 13 a. The loaded CNS display rough surface confirming drug loading. There was no agglomeration or change in particle shape observed confirming the stability even after the pheromone loading. A slight increase in particle size confirms effective loading of the pheromone (Figure 13 b).

## 3.2 PRELIMINARY ANALYSIS OF ACTIVE LOADING USING TLC

The preliminary analysis conducted using TLC demonstrated effective incorporation of active compounds into the dispenser particles chosen for this study. As illustrated in Figure 14, the Rf values obtained from both the standard (St) and the sonicated sample (S) were identical.

## 3.3 EE & LC OF THE PHEROMONES

The EE % and LC % of *Lobesia* and BFSB pheromone were depicted in Table 1

The results of the two-way ANOVA examining the effects of the optimized ratio of dispenser material and pheromone type on LC % and EE % are tabulated in Table 2.

The combination of different dispenser materials and pheromone did not result in any significant difference statistically ($F = 0.966532$, $p < 0.05$). The pheromone type or dispenser material showed a highly significant effect on EE and LC of the material ($F = 23598.99$, $p < 0.001^{***}$). Post hoc analysis using Tukey's Honestly Significant Difference (HSD) test indicated that CNS and the blend showed significant differences, with the blend

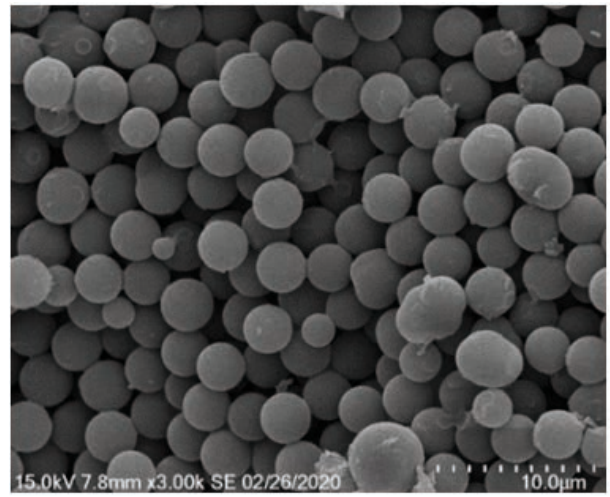



**Figure 12:** SEM image of synthesized CNS

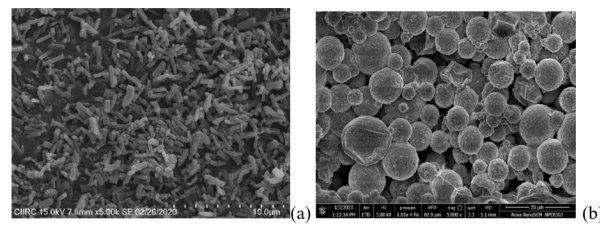



**Figure 13:** SEM image of loaded MSN (a) and CNS(b)

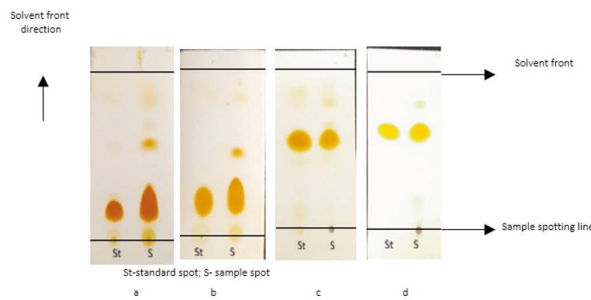



**Figure 14:** TLC of *lobesia* and BFSB pheromones extracted from MSN and CNS with respective standards. Plates: (a) Std *lobesia* + *lobesia extracted from MSN (Rf = 0.17,0.21); (b) Std lobesia* + *lobesia* extracted from CNS (Rf = 0.21,0.24); (c) Std BFSB + BFSB extracted from MSN (Rf = 0.54,0.54); (d) Std BFSB + BFSB extracted from CNS (Rf = 0.63,0.63)

**Table 1:** EE and LC of dispenser materials

| Pheromone | MSN: CNS | EE (%) | LC (%) |
|---|---|---|---|
| *Lobesia* | 01:00 | 89.86 ± 0.26 | 44.93 ± 0.4 |
| *Lobesia* | 00:01 | 85.96 ± 0.28 | 42.98 ± 0.86 |
| *Lobesia* | 0.5:1 | 88.22 ± 0.68 | 43.5 ± 0.55 |
| *Lobesia* | 01:01 | 91.63 ± 2.21 | 45.82 ± 0.65 |
| *Lobesia* | 01:01.5 | 88.09 ± 0.51 | 43.94 ± 0.14 |
| BFSB | 01:00 | 90.95 ± 0.28 | 45.48 ± 0.24 |
| BFSB | 00:1 | 87.70 ± 0.34 | 43.85 ± 0.36 |
| BFSB | 0.5:1 | 87.45 ± 0.87 | 44.08 ± 0.23 |
| BFSB | 01:1 | 92.23 ± 0.24 | 46.12 ± 0.87 |
| BFSB | 01:1.5 | 88.93 ± 0.78 | 44.97 ± 0.36 |

demonstrating higher efficiency. MSN did not exhibit any significant difference from CNS and blend. This suggests increased interaction of the pheromones with the physical admixture of MSN and CNS, which may be attributed to properties of the physical admixture of the two materials. The added advantage of more surface area and enhanced interaction with hydrophobic molecules of MSN and CNS respectively were effectively exploited by this combination resulting in higher EE. This could potentially result in extended release which may also depend on the physicochemical properties such as vapor pressure differences, volatility among the selected pheromones. The LC % was stable across all the three dispenser materials and the blend of MSN and CNS showed no statistically significant difference in loading of both the pheromones.

## 3.4 WEATHER CONDITIONS

The average weather conditions at different timelines in the semi field studies were recorded and subsequently tabulated (Table 3).

## 3.5 QUANTIFICATION OF RESIDUAL PHEROMONE BY GC

Samples collected at intervals of 10, 20, 30, 40, and up to 120 days were analyzed and quantified using GC. The residual *Lobesia* and BFSB pheromones against time were represented in figure 15 & 17 as percentage of the initial loading of active.

### *3.5.1 Lobesia* release

The control vial released its entire contents within one month. A burst release i.e 60 % of active was released within 10 days from control compared to 30-40 % via dispenser materials. The blend demonstrated more effectiveness than MSN and CNS up

**Table 2:** ANOVA of EE and LC of dispenser materials

| Source of Variation | SS | df | MS | F | P-value | F crit |
|---|---|---|---|---|---|---|
| Rows | 2.482628 | 5 | 0.496526 | 0.966532 | 0.481981 | 3.325835 |
| Columns | 24246.49 | 2 | 12123.24 | 23598.99 | ***4.27E-19 | 4.102821 |
| Error | 5.137189 | 10 | 0.513719 | | | |
| Total | 24254.11 | 17 | | | | |

*** $p < 0.001$ (indicates level of statistical significance)

**Table 3:** Weather conditions at different timelines in the study

| Days | Average temperature (°C) | Average wind speed(km/h) | Average humidity (%) | Average precipitation (mm) |
|---|---|---|---|---|
| 10 | 29.32 | 10.23 | 42 | 5.3 |
| 10-20 | 30.65 | 11.45 | 39 | 3.1 |
| 20-30 | 32.42 | 11.33 | 40 | 11.32 |
| 30-40 | 36.64 | 11.86 | 43 | 7.2 |
| 40-60 | 38.65 | 12.32 | 38 | 36 |
| 60-90 | 40.32 | 14.54 | 40 | 42.2 |
| 90-120 | 36.34 | 12.06 | 58 | 104 |

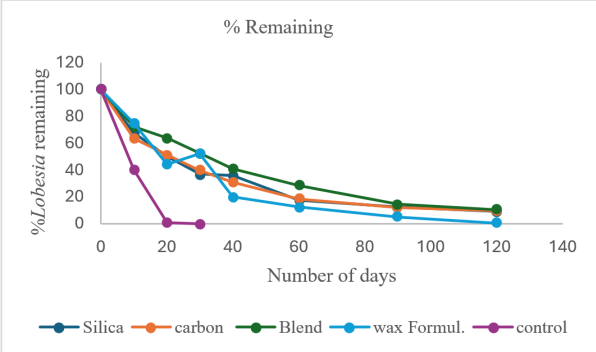



**Figure 15:** Residual *Lobesia* pheromone against different time points

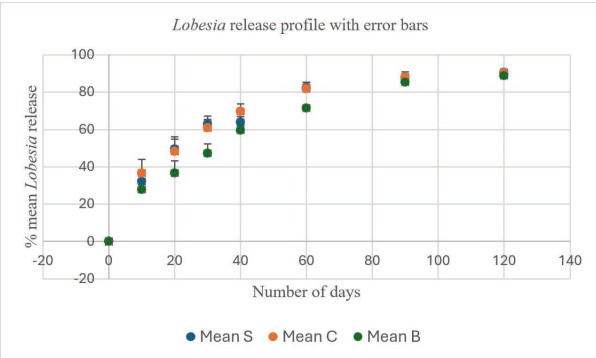



**Figure 16:** Mean *Lobesia* release at different time points. Error bars represent SD from triplicate measurements (n = 3)

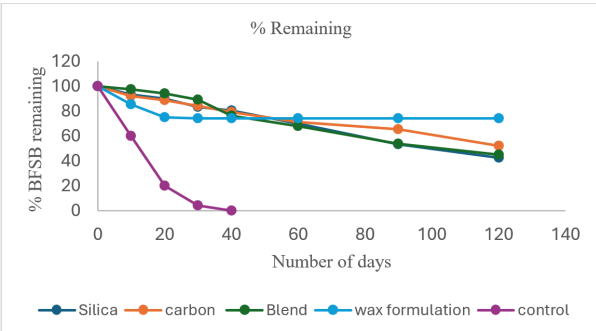



**Figure 17:** Residual BFSB pheromone against different time points

to two months. After 3 months all the three dispenser particles retained similar pheromone content. More than 10 % of active remained in the particles compared to wax formulation which showed no pheromone after 110 days. Even after 120 days 10 % of *Lobesia* pheromone was still retained in the dispenser particles. As shown in Figure 16, there is high variability in error bars at early time points, likely due to the initial burst release; beyond 60 days, the release profile became more consistent.

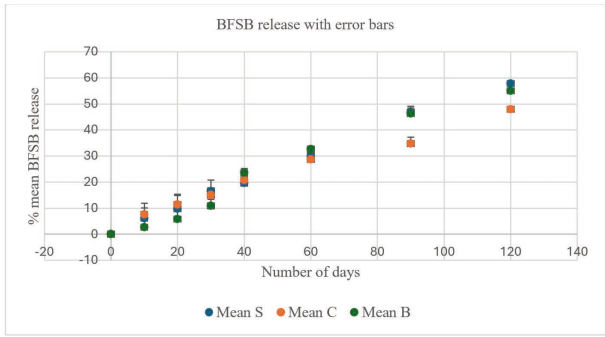



**Figure 18:** Mean BFSB release at different time points. Error bars represent SD from triplicate measurements (n = 3)

### 3.5.2 BFSB release

All the three dispenser materials exhibited equivalent release patterns. 89 % of BFSB active is still retained by the blend after one month. 83-84 % retention was observed for MSN and CNS. 90 % of active was lost in control within a month. The wax formulation showed release until 40 days only and no further release was observed which shows its ineffectiveness after 40 days. CNS showed a better retention capacity than the other two materials. Error bars indicated consistent BFSB pheromone release beyond 40 days, as depicted in Figure 18. An initial burst release was noted at earlier time points. Greater variability in CNS release may be attributed to the initial release of surface-bound pheromone followed by the subsequent release of entrapped pheromone.

The variation in vapor pressure and volatility between *Lobesia* and BFSB pheromones influenced the release patterns from the selected material dispensers. The observations indicate a greater interaction of BFSB with the material dispensers compared to *Lobesia botrana* pheromone. No significant differences among the materials were observed in terms of BFSB release. Statistical analysis confirmed significant effect of different time points and dispenser materials on the release of *lobesia* as well as BFSB pheromones. Two-way ANOVA results of *Lobesia* pheromone ($F = 10.92$, $p < 0.001^{***}$) and BFSB pheromones ($F = 0.903851$, $p > 0.05$) from different materials and at different time points for *lobesia* ($F = 241.95$, $p < 0.001^{**}$) and BFSB release ($F = 77.54$, $p < 0.05^{**}$) were consistent with the obtained release data.

## 3.6 RELEASE KINETICS OF PHEROMONES

The release trend of the two selected pheromones in this study across dispenser materials were determined using different kinetic models and the coefficients of determination ($R^2$) obtained are tabulated (Table 4). The re-

**Table 4:** Kinetics of pheromone release from dispenser materials

| | *Lobesia* | | | BFSB | | |
|---|---|---|---|---|---|---|
| Pheromone | Dispenser material | | | Dispenser material | | |
| Kinetic model | MSN | CNS | Blend | MSN | CNS | Blend |
| | | | Coefficient of determination ($R^2$) | | | |
| Zero Order | 0.7508 | 0.7407 | 0.8654 | 0.9965 | 0.9795 | 0.9744 |
| First order | 0.9506 | 0.9461 | 0.9898 | 0.9911 | 0.9845 | 0.9897 |
| Higuchi | 0.9492 | 0.948 | 0.9871 | 0.9139 | 0.9538 | 0.8914 |
| Korsmeyer-Peppas | 0.9367 | 0.8853 | 0.8296 | 0.9922 | 0.9915 | 0.9698 |

sults represented in table 4 confirm that first order kinetic model best fits the release of *Lobesia* pheromone from all three materials as represented by the highest coefficient of determination. The first order equation shows that the release is concentration dependent. The release of BFSB Pheromone from blend of MSN and CNS was found to be concentration dependent but the release from the individual materials shows the best fit by Korsmeyer-Peppas model. The model showed a release exponent of < 0.5 and 0.5-1 in the release of *Lobesia* and BFSB respectively which supports that *Lobesia* release is purely diffusion controlled following Fickian diffusion and BFSB follows anomalous transport. This release pattern can be attributed to absence of swelling in MSN causing *Lobesia* release concentration dependent and there may be swelling or relaxation of the material dispensers in BFSB release thus exhibiting anomalous transport.

The $R^2$ values for the first order release show that it is the best fit model to support the release kinetics of *Lobesia* pheromone from MSN, CNS as well as blend. The release of BFSB pheromone from CNS and blend was best fit by the first order kinetic model as evident from highest $R^2$ values but the release from silica materials was best fit by zero-order kinetic model which is ideal. EE was statistically significant for CNS and physical admixture (1:1). There was no significant difference in LC % across all the three dispenser materials. This shows that there was a better interaction of pheromones with that of CNS and physical admixture of MSN and CNS thus exhibiting high EE %.MSN as a single dispenser material showed no significant entrapment comparatively. The enhanced EE promotes slow release of pheromones. The slow-release materials used in the present study are effective in controlling the release of active ingredients. Mesoporous silica was utilized to prolong the release of bee repellant pheromone (Zanoni et al., 2019). CNS with specific surface area, defined pore size and high compression strength offer wide applications like adsorption of various chemical compounds (Tripathi N. K.2018). The bioavailability of drug celecoxib was improved by employing uniform mesoporous carbon as carrier (Wang et al., 2016). Pheromones can be highly effective at very low concentrations of < 100 ng $m^{-3}$ to disrupt the mating of pests (Gavara 2020). The selected pests for the study exhibit a peak flight period lasting for about 8 months in case of *Lobesia botrana* (Loriatti et al., 2011) with average life cycle lasting for 30-50 days depending on temperature and 8-10 generations per year for BFSB with average life cycle 0f 38-40 days (Kumar & Kumar, 2010). After the study period of 120 days, a residual amount of 40 % BFSB and 10 % *Lobesia* pheromones in the vials can effectively be used for season long control. The PE vials with effective loading can be used to control release of pheromone up to 45 days (Johansson et al., 2001). MD was effectively achieved using passive dispensers such as PE vials and twist -tie ropes(Ricciardi et al., 2022) .MD of *Grapholita funebrana* Treitschke, 1835 was effective till 72 days with a loading of 253 mg per each polyethylene vial dispenser (Lo Verde et al., 2020). Polyethylene tubes with a load of 220 mg were effective in MD of *Lobesia botrana* for 4 months (Gordon et al., 2005). Thus, the present study with the developed materials having 100 mg load of pheromone can be used effectively with one replacement of loaded dispenser vials for season long control of *Lobesia botrana* and BFSB. The decreased amount of pheromone compared to earlier works of others can decrease the cost of sexpheromone based IPM.

The release of pheromones from polymer systems generally follows first order release trend (Hellmann 2024). *Lobesia botrana* pheromone release was measured at 0.65 mg to 1.02 mg per day (Gavara et al., 2022) suggested that a loading of 97.5 mg is sufficient for effective MD; Thus in the present study each particle dispenser contains 100 mg. *Lobesia* pheromone shows Fickian diffusion from all the dispenser materials as the release might be purely diffusion based depending on the concentration of the pheromone whereas the BFSB pheromone exhibited anomalous transport which might be attributed to the interaction of BFSB with dispenser wall material.

## 4 CONCLUSIONS

The synthesized MSN, CNS and the blend of these released the *Lobesia botrana* and BFSB pheromones for a prolonged period. The blend showed better release profile for both the pheromones by effectively retaining the pheromones. The release of BFSB was much prolonged which might be due to its physicochemical properties. The materials used showed better results than reference and control. The first order release model best fits the kinetics of pheromone release. These materials can serve as promising delivery systems for season long control of European grape vine moth and brinjal fruit and shoot borer.

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