Encapsulation of Pheromones and Essential Oils

INTRODUCTION

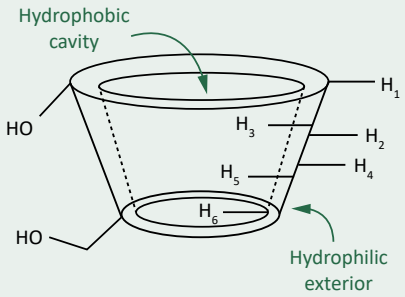
Encapsulation technology is revolutionizing the agricultural industry by offering innovative methods to **enhance the effectiveness and longevity of active ingredients**. This process involves encasing bioactive substances within a protective matrix, allowing for **controlled release and targeted delivery**. Such advancements are particularly crucial in modern agriculture, where there is an increasing need for sustainable and efficient solutions to crop protection and enhancement. Encapsulation not only improves the stability of these compounds but also ensures their **optimal performance with minimal environmental impact**.

Roquette, a global leader in plant-based ingredients, is committed to driving the agricultural sector forward. Our innovative solutions address challenges such as pest control, nutrient management, and crop health. By leveraging our expertise and technology, we aim to advance sustainable agricultural practices, ensuring superior outcomes and promoting environmental stewardship.

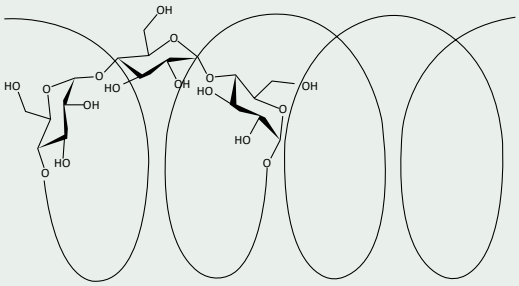
ENCAPSULATION AGENTS

KLEPTOSE® beta-cyclodextrin (BCD) and Linecaps are advanced encapsulation agents used in agricultural formulations, enhancing product stability, solubility, and shelf life.

KLEPTOSE® BCD enhances the stability of hydrophobic molecules and lipophilic parts, and protecting them from oxidation, hydrolysis, temperature degradation, and photolysis. This significantly increases shelf life and reduces toxicity. It transforms liquid actives into solid complexes, ideal for stable agricultural formulations. Although its low solubility limits its use in liquid form, **KLEPTOSE® BCD** is plant-based, biodegradable, thermally stable, and robust in alkaline conditions, making it a reliable encapsulation agent for a variety of agricultural applications.



Linecaps pea maltodextrin, forms inclusion compounds with flexible chain diameters and molar ratios for improved solubilization. Its plant-based, biodegradable composition features a helical structure with a hydrophilic exterior and hydrophobic cavity. Easily soluble in cold water, it exhibits low viscosity and weaker interaction compared to cyclodextrins. **Linecaps** ensures safe handling and adapts to various guest molecules, making it a versatile choice for diverse agricultural applications.



MOLECULAR ENCAPSULATION OF ESSENTIAL OILS WITH ROQUETTE PRODUCTS

Molecular encapsulation is crucial in agriculture for **protecting essential oils used as plant protection solutions. Essentials oil** have beneficial properties for pest control, but their volatility and sensitivity to environmental factors limit their effectiveness. Encapsulation with Roquette products enhances their stability and controlled release, making them more effective as plant protection agents.

Case Study: Molecular encapsulation of Orange Oil, comparison of Linecaps and GLUCIDEX® range in combination with CLEARGUM®

The primary compound in orange essential oil is limonene (60-90%), a hydrophobic aromatic compound that faces challenges due to its volatility and oxidation. Using Linecaps (pea maltodextrin) with CLEARGUM® CO 01 (OSA starch) as an emulsifier enhances the stability and retention of limonene, making it more effective for various applications.

Table 1: Encapsulation composition of orange oil

Ingredients	Quantity (%)	Mass quantity (g) for 40% dry matter
Maltodextrin	21	105g
CLEARGUM® C0 01	15	75g
Limonene	4	20g
Water	60	300g

The encapsulation process involves dissolving 21% of maltodextrin, 15% CLEARGUM® CO 01, 4% limonene, and 60% demineralized water at 70ºC to form a pre-emulsion. A limonene solution preheated to 30ºC is added and stirred vigorously. The pre-emulsion is subjected to high shear forces at 5ºC using a POLYTRON PT 45/2M device at 24,000 rpm for 10 minutes. The mixture is then atomized with a Lab Plant SD05 atomizer at 60ºC and an inlet temperature of 175ºC. Encapsulation efficiency is measured by evaluating the retention rate of limonene. The powder obtained is washed with hexane to isolate surface-bound limonene, while encapsulated limonene is solubilized. Quantification is done using gas chromatography (VARIAN 8200 CX autosampler, column DB1, temperature gradient from 60 to 250°C at 7°C/min, He vector gas). To determine the protective efficacy against external environmental aggressions, a measurement of limonene oxidation over time is conducted. The encapsulated limonene is incubated at a temperature of 70°C in an oven for 20, 40 and 60 days. The limonene and limonene oxide are dissolved in acetone, then purified and measured by gas chromatography (VARIAN 8200 CX autosampler under the same conditions as previously).

Table 2: Retention level of limonene

Maltodextrin		
Base origin	Grade	% Limonene
Corn	GLUCIDEX® 12	67
	GLUCIDEX® 17	67
	Linecaps DE 17	63

Table 3: Evolution of the oxidation rate of limonene

Maltodextrin	% limonene oxide / 100% limonene			
	T0	T20	T40	T60
GLUCIDEX® DE 12 Corn-based	0	1.79	1.79	1.80
GLUCIDEX® DE 17 Corn-based	0	1.79	1.94	1.98
Linecaps	0	1.90	2.22	2.41

The use of **Linecaps and CLEARGUM® CO 01** yielded **high encapsulation efficiency** for limonene, with retention rates comparable to GLUCIDEX® maltodextrin range. This combination offers several benefits, including **easy preparation of solutions, low viscosity even at high dry matter, efficient atomization, and no stickiness**. The resulting microcapsules exhibit **good stability and low oxidation rates**, making Linecaps a superior choice for protecting volatile compounds.

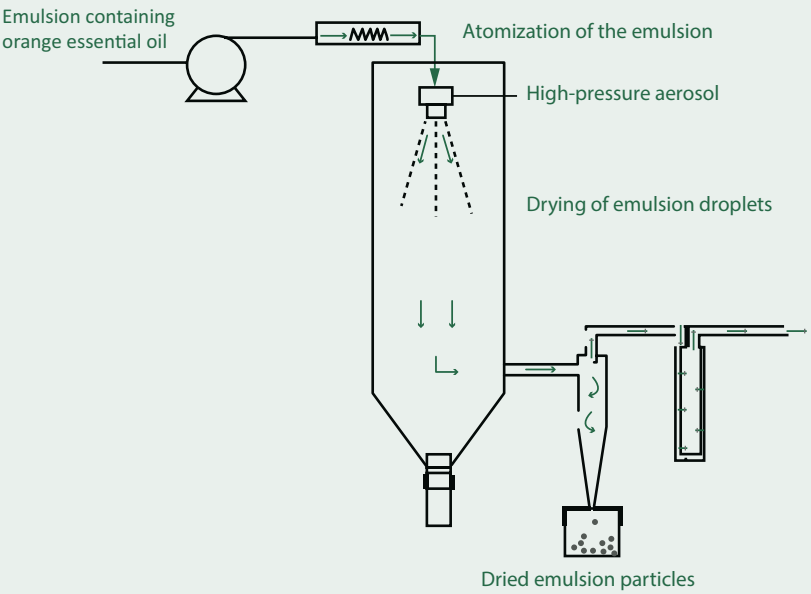


Figure 1: Spray drying of orange essential oil emulsion

MOLECULAR ENCAPSULATION OF PHEROMONE WITH ROQUETTE PRODUCTS

Case Study: Molecular Encapsulation of Pheromone with KLEPTOSE®

In agriculture, various encapsulation technologies are employed to enhance the stability, solubility, and controlled release of active ingredients such as pheromones. Another experiment has been conducted to investigate the formation of inclusion complexes between KLEPTOSE® beta-cyclodextrin and a mixture of pheromones.

Pheromones are chemical signals used by insects to communicate, and their encapsulation has gained significant attention in agriculture for pest control. Utilizing pheromones in a controlled manner can disrupt mating patterns of pests, thereby reducing their population without the need for harmful pesticides. This environmentally friendly approach leverages molecular encapsulation techniques to enhance the stability and effectiveness of pheromones when deployed in agricultural settings.

The experiment involves preparing KLEPTOSE® BCD: pheromone complex in molar ratio of 2:1. Using a laboratory kneader (HKD-T 06 D IKA High-performance laboratory kneader), KLEPTOSE® BCD is mixed with pheromones and water to form a smooth paste, which is then kneaded for five minutes, dried in an oven at 45 °C, and ground into a fine powder.

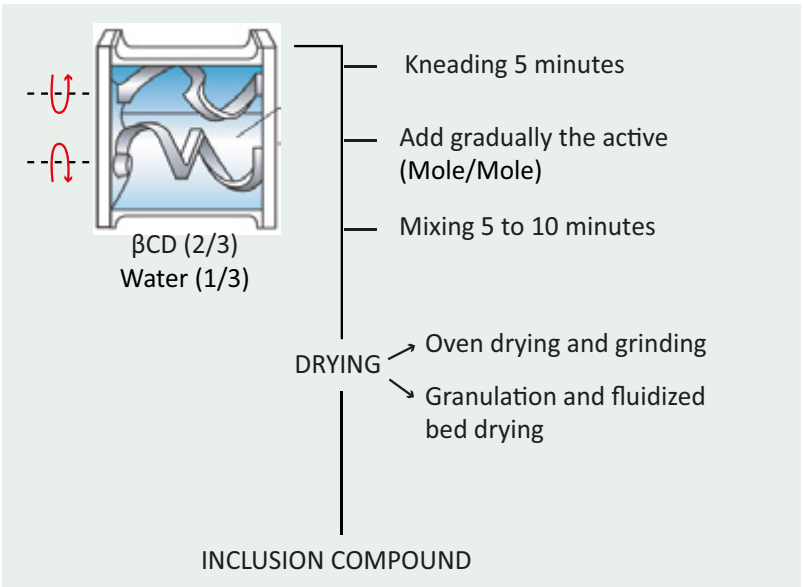
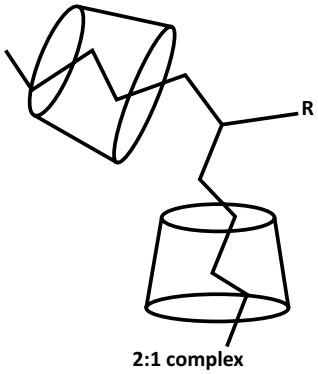


Table 4: Encapsulation composition of pheromones (insecticide) with KLEPTOSE® BCD

Ingredients	MW (g/mol)	Quantity (g)
KLEPTOSE® BCD (14% water content)	1135	187.69
Pheromone mixture	245	17.77
Water (1/3 BCD)	18	62.56
Total		268.03

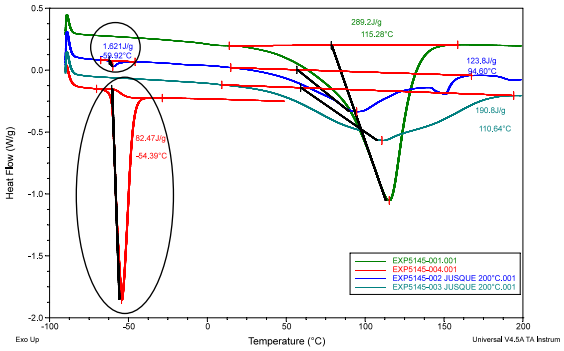
Molar ratio BCD:AI=2:1



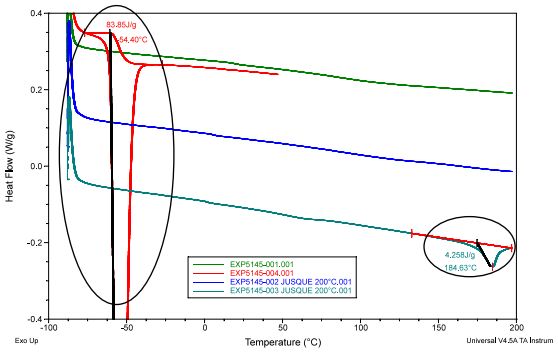
Differential Scanning Calorimetry (DSC) thermograms and DRX diffractograms confirm the formation of inclusion complexes between KLEPTOSE® BCD and pheromones, particularly in the presence of water, which facilitates the formation of these complexes and enhances the encapsulation process. DRX diffractograms shows diffraction lines at 11.6 ° and 17.6 ° which are specific to inclusion complexes with BCD. On the DSC thermograms, pheromone endotherms are absent in the 2:1 complex spectrum made with water (light blue), unlike the complex without water (dark blue), which shows a melting endotherm at -54 °C (83 J/g) and a crystallization endotherm at -75 °C (71 J/g). Additionally, the presence of a new crystalline species during the cooling phase (crystallization at 164 °C, 7.8 J/g) and the second heating phase (melting at 185 °C, 4.3 J/g) further confirms the successful encapsulation and formation of inclusion complexes. These findings indicate that water significantly enhances the encapsulation efficiency and alters the physical properties of the KLEPTOSE® BCD and pheromone mixture.

Differential Scanning Calorimetry (DSC)

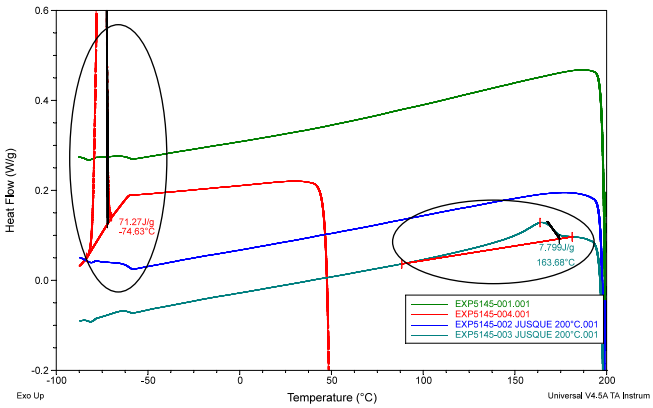
First heating



Second heating



Cooling



pheromones mixture
ß-cyclodextrin
ß-cyclodextrin: pheromones (2:1) (physical)
ß-cyclodextrin: pheromones (2:1) (kneading)

Figure 2: DSC thermograms of (2:1) BCD:pheromones inclusion complexes with and without water: a) first heating ; b) cooling ; c) second heating

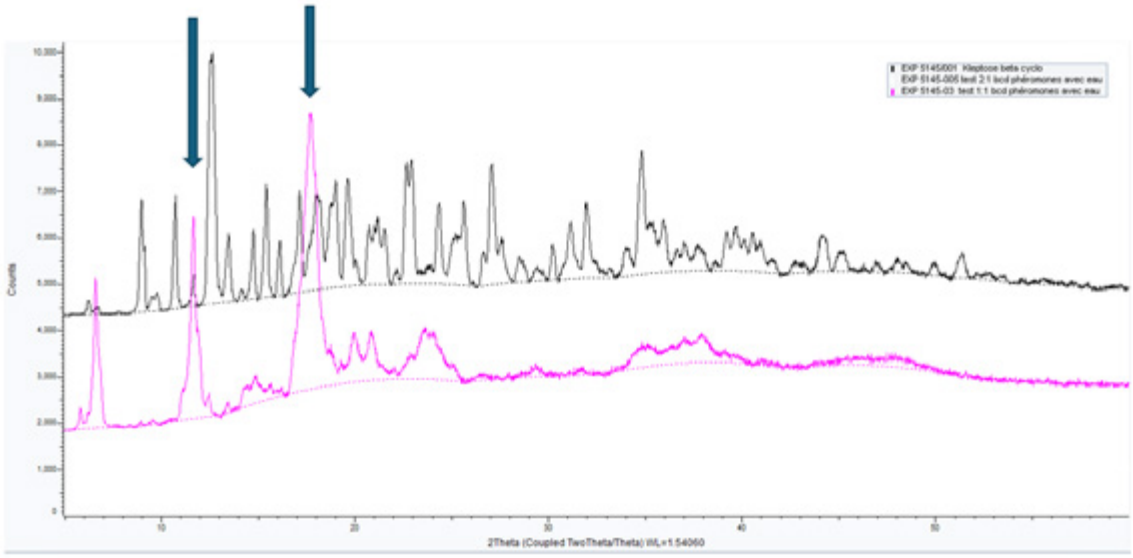


Figure 3: DRX diffractograms of BCD : pheromones (2:1) inclusion complexes

A DEDICATED RANGE OF SOLUTIONS FOR ENCAPSULATION FORMULATIONS

Roquette offer a specialized range of high-quality ingredients designed for encapsulation formulations in agricultural applications. Our selection includes advanced encapsulating agents and emulsifiers that ensure the stability and effectiveness of active ingredients. These components optimize release properties and longevity, providing environmentally friendly solutions that reduce reliance on harmful pesticides.

Product	Type	Grade	Encapsulant - Carrier	Emulsifier	Viscosity	Solubility	pH
GLUCIDEX®	Maltodextrin	17,19 or 21	✓		Low	Fully (600g/L)	~5
CLEARGUM®	Emulsifying starch	CO A1, CO 03, or CO 01	✓	✓	Low – Mid - High	High	Low (3-4)
Pea maltodextrin		Linecaps 17	✓		Low to High (250 - 2000 mPa.s by conc)	High	Low (~5)
KLEPTOSE®	beta-cyclodex-trin	BCD or HP	✓		Low ~90 mPa.s at 500 mM solution	Low	Neutral (5-8)
NUTRALYS® W	Wheat protein hydrolysate	W	✓	✓	Low to High (by conc.)	Low	Neutral (5-7)

To find out more about our plant-derived products for plant nutrition and protection products and how they can help you formulate an optimized plant care solution for your needs, check our website and contact us

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