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Coacervation Process for Preparation of Encapsulate Lambda-Cyhalothrin

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ABSTRACT

In the present study, a new type of an encapsulate pesticide was prepared by Coacervation method. Lambda-Cyhalothrin was encapsulated due to interaction between two oppositely charged materials, gelatin and gum Arabic as shell in a complex Coacevation process at the presence of 10 mol etoxylated nonyl phenol (NPE) which is a kind of a non-ionic emulsifier to enhance the homogenization of the product and size control. Spherical shape of capsules were scanned by Scanning Electron Microscopy (SEM) and optical microscope. The encapsulation efficiency was achieved 96% and analyzed by Gas chromatography equipped with FID detector (GC-FID). From the results of release study, 49, 63, 89% of encapsulated materials were released from their shell after 15, 30,180 min respectively. The present study, applies a convenient and inexpensive method to introduce a pesticide, which can be exploited in public health field.

Keywords: Lambda-cyhalothrin, Coacervation, Encapsulation, Release study, Encapsulation efficiency

Introduction

Lambda-cyhalothrin belongs to a group of pesticides called pyrethroids which is registered by the U.S. Environmental Protection Agency (EPA) in 1988. It is offered as an alternative for Cyhalothrin about which is consisted of two diastereoisomeric pairs of isomers of Cyhalothrin. It is widely used as an effective insecticide in agriculture and public health domain such as hospitals and houses in different forms of formulations such as wettable powders, emulsion concentrates and capsule suspensions. Lambda-cyhalothrin and generally pyrethroids paralyze and extirpate insects by disrupting their nervous system [1, 2, 3].

Microencapsulation is known as a process of which small droplets of a kind of core material in an emulsion bulk entrap into a shell. The shell or wall materials which cover the core materials can be chosen from polymers, polysaccharides, lipids or proteins. Core materials such as volatile materials, flavors, essential oils and pesticides are capsulated to become slow release or being released over a long period of time to take full advantage of them and also being protected against environmental influences such as light, oxygen and variety of other conditions [4, 5, 6].

Abundant of methods have been widely carried out to protect the core materials during encapsulation process, which are divided into three main categories: chemical, physico-chemical and physico-mechanical methods among, which Coacervation technique that is a subcategory of physico-chemical method, have been using and developing since 1930 for many purposes [7]. Meticulously and simply, Coacervation will occur in circumstances as bellow:

a) Preparation of a homogenous emulsion of a core material in a solution of shell materials in water which are carrying opposite charges to have the maximum interaction between positive and negative charges to

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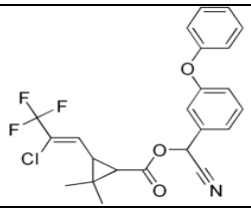
fold the core material by colliding with each other, for example gelatin with negative and positively charged acacia positively charged at the presence of an emulsifier.
b) Mild heating and agitation as well as pH adjustment, encapsulation takes place in this stage.
c) Cooling and adding cross linking materials such as formaldehyde or glutaraldehyde, the prepared capsules from stage 2 will be hardened by adding cross linking materials [8, 9, 10, 11].
The present study offers a slow-release insecticide product based on Coacervation method by gelatin/gum Arabic as shell materials and Lambda-cyhalothrin as core material, in which the core material releases over a long time, not only to increase the effectiveness, but also to enhance durability and stability.

Experimental

Materials and devices

A type gelatin and gum Arabic were prepared from chemical loacal market in Tehran, Kenon 10® or 10 mole etoxlytated nonyl phenol was from Kymiagaran Emrooz Industry. Solveso 200 ND was prepared from Hydro carbon Iran, Hydrochloric acid, Sodium hydroxide and glotaraldehide were purchased from Merck Company, Germany. Lambda-cyhalothrin was prepared from Gharda Company, India. Characteristic of Lambda-cyhalothrin is shown in table1. All solutions were prepared in distillated water. For investigation of release behavior of capsules, GC model Agilent 7890B and roller mixer model PLMA 001 Farzaneh Arman were applied. SEM model XL 30 Philips in Tarbiat Modares university and optical microscope model OLYMPUS CX23 Japan was used for scanning and detecting the shape of obtained capsules. The pH measurement was recorded by pH meter model AZ Taiwan.

Table 1
Chemical and physical properties of Lambda-cyhalothrin.

Chemical Name (IUPAC)	Structure	Molecular weight	Physical state
3-(2-chloro-3,3,3-trifluoro-1-propenyl) -2,2-dimethyl-cyano (3-phenoxyphenyl) methyl cyclopropanecarboxylate		449.85 gr/mol	Off white powder

Preparation of capsules

5.0 gr of gelatin powder and 5.0 gr of gum Arabic were added into 100 mL distillated water separately under a mild agitation and heating until obtaining a homogenous solution and then were stored in a refrigerator overnight to complete the hydrolyzing process. 5.1 gr of Lambda-cyhalothrin was dissolved in the minimum amount of Solveso 200 ND and 2 gr of NPE under a mild stirring and heating. For preparation of emulsion, the prepared solution of Lambda-cyhalothrin was added into gum Arabic solution under a mild heating at 40ºC and stirring for 30 minutes to break down the size of emulsified droplets of Lambda-cyhalothrin. Furthermore, the solution of gelatin was poured into Lambda-cyhalothrin emulsion, slowly and dropwise continually over about 30 min. pH was adjusted at 4 by adding acetic acid 20% (W/V) afterwards. The obtained mixture was cooled down to below 10ºC overnight in a refrigerator. In the end, the process of encapsulation was completed by pouring 10 mL glutaraldehyde, adding 200 mL water and fixing pH at 6 by adding NaOH 0.1M (the pH of Lambda-cyhalothrin's stability as FAO specification) then stirring was continued for 4 hours. Obtained milky viscous solution was tested to detect of the shape of capsules, micro encapsulation efficiency and release study.

Results and Discusion

Imaging of capsules by scanning electron microscopy and optical microscope

SEM model XL 30 Scanning Electronic Microscope (Philips) in Amir Kabir University, Tehran, Iran, was used to show the shape and approximate size of capsules. The microspheres were observed at an accelerating voltage of 25 kV. The image is shown in Figure 1.

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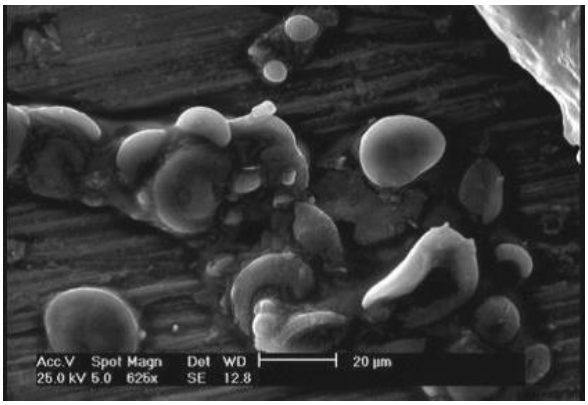

Figure 1. Spherical shape of Lambdacyhalothrin capsules by SEM

Optical microscope OLYMPUS CX23 was exploited to show the capsules shape as well. The result is shown in Figure 2.

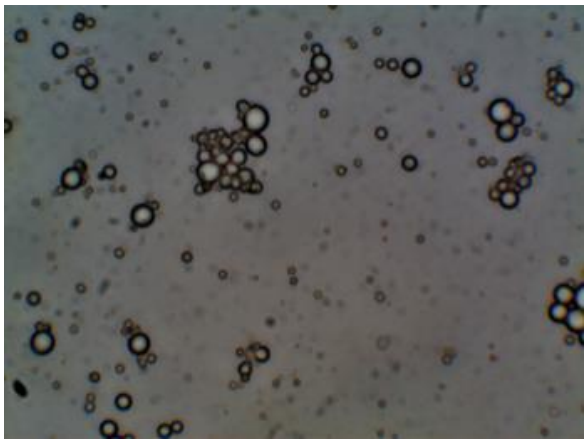

Figure 2. Image of Lambda-Cyhalothrin capsules by Optical microscope

Microcapsule efficiency

To measure the Microcapsule efficiency or encapsulation yield (the amount of capsulated Lambda-Cyhalothrin), 1 gr of the prepared sample (capsule solution) was transferred into a 60 mL glass tube, then 5-5.5 mL distillated water and 50 mL n-Hexan were added to the tube and the mixture was placed on a roller set at 70 RPM for 5 min. After 5 minutes (±10 seconds) the bottle was removed from the roller and immediately a sample was taken from hexane layer for GC analysis under defined conditions as Table 2, below. Results were compared with analytical standard Lambda-Cyhalothrin. The amount of released Lambda-Cyhalothrin was measured as equation 1. The amount of non-capsulated Lambda-Cyhalothrin was obtained 4% which means 96% of Lambda-Cyhalothrin was successfully entrapped in the capsules (Interim specification WHO/IS/CS/463/2002).

GC chromatogram is shown in Figure 3. a and b, in which, (a) is the chromatogram of analytical standard of Lambda-Cyhalothrin and (b) is belonged to the amount of capsulated Lamda-Cyhalothrin.

$$\text{Non-capsulated Lambda-Cyhalothrin} = \frac{\text{RMP}}{\text{R}\prime\text{W}}\ \text{g/Kg} \quad [1]$$

R = Total Lambda-Cyhalothrin peak ratio of released non-capsulated Lambda-Cyhalothrin

R'= Total Lambda-Cyhalothrin peak ratio of standard Lambda-Cyhalothrin

M = Mass of Lambda-Cyhalothrin Standard

W = Mass of the solution of encapsulated Lambda-Cyhalothrin sample

P = Purity of the Lambda-Cyhalothrin Standard

Table 2

GC-FID condition: Defined conditions for determination of the amount of released Lambda-Cyhalothrin in capsules.

Inlet (°C)	Oven (°C)	Detector (°C)	Carrier flow (N_2) (mL/min)
250	280	320	0.9

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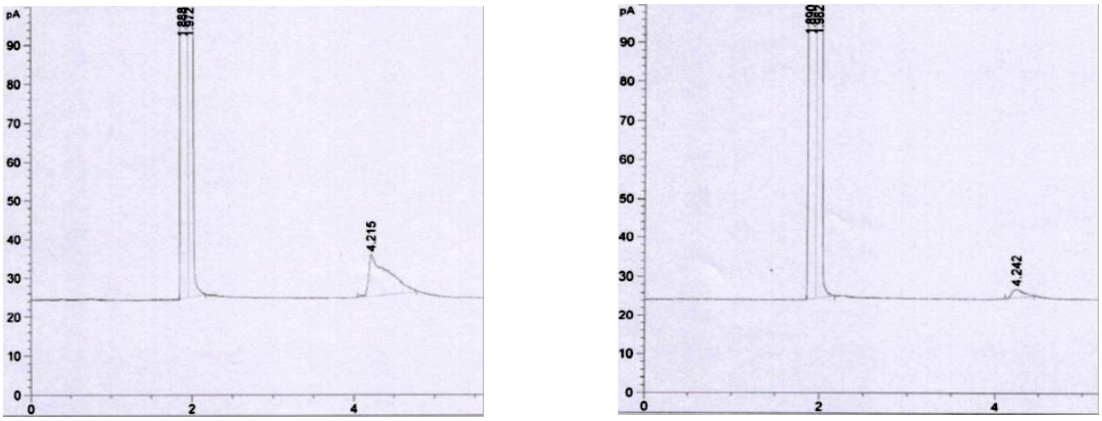

(a) (b)

Figure 3. (a) chromatogram of analytical standard of Lambda-Cyhalothrin and (b) the amount of non-capsulated Lamda-Cyhalothrin

Release study

For investigation of releasing behavior of Lambda-Cyhalothrin in capsules, about 0.7-0.8 gr of solution was weighted then 5-5.5 gr distillated water was added. The mixture was mixed with 90 mL of n-Hexan and 10 mL ethanol in a 150 mL glass tube. The glass tube was sealed and put on a roller mixer at 70 RPM for 15, 30, 180 minutes. After aforementioned times the sample was taken from n-Hexan phase to analyze and calculate by the equation 1 which is given in section 3.2, by GC-FID [12].

Release of lambda-cyhalothrin from the capsules, determined by the given method, was included:
at 15 min, 49% of that released at 180 min;
at 30 min, 63% of that released at 180 min; and
at 180 min, a minimum of 89% of the total lambda-cyhalothrin content determined according to the method.
The results from GC chromatogram is shown in Figure 4. are the amount of released Lamda-Cyhalothrin at 15,30, 180 min respectively.

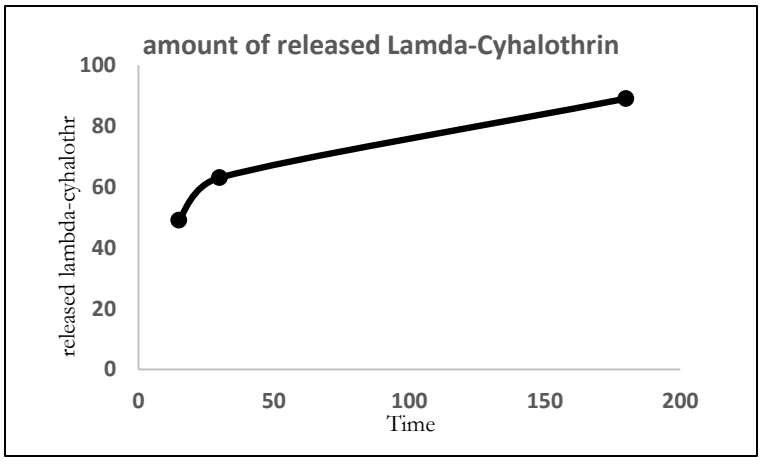

Figure 4. The amount of released Lamda-Cyhalothrin at 15,30, 180 min

Conclusion

From the present reseach a simple and applicable method for encapsulation of Lambda-Cyhalothrin as a pyrothroid pesticide is offered which does not need for any special instrument to produce. From SEM pictures, the micrometric size of capsules were achieved by using an appropriate shell materials and also the time of mixing to have a homogeneous emulsion by Kenon 10®, before adding gelatin and complation of encapsulation process. It can be easily seen from Figure 4 and extracted data from the chromatoghraphs, the release behaviour of capsules shows a suitable adoption with the data, which is given in WHO standards. Encapsulation efficiency seems to be acceptable for this method as it can be resulted from Figure 3, more

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than 96% of Lambda-Cyhalothrin was encapsulated, because of having a strong interaction between oppositely charged gelatin and arabic gum as shell materials.

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