Article

Preparation and characterization of Nanocapsules for application as pesticides

Preparación y caracterización de Nanocápsulas para su aplicación como plaguicida

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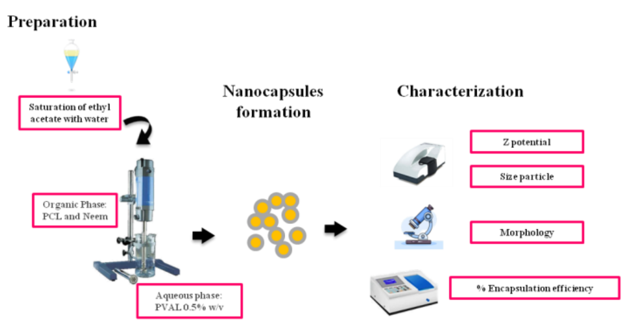
Abstract

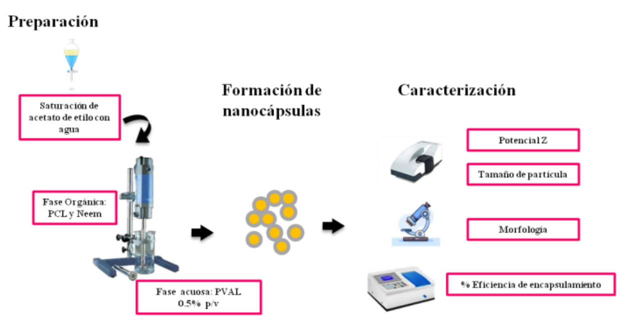
In the present work, polymeric nanocapsules of polycaprolactone loaded with neem extract were prepared. The nanocapsules were prepared by the emulsification-diffusion method. The nanocapsules ranged in size from 254 to 369 nm. An encapsulation efficiency of up to 84.5% was achieved.



Nanopesticides, Neem Encapsulation, Polycaprolactone Nanoparticles

Resumen

En el presente trabajo, se prepararon nanocápsulas polimericas de policaprolactona cargadas con extracto de Neem. Las nanocápsulas se prepararon por el método de emulsificación-difusión. Las nanocápsulas obtuvieron un tamaño de 254-369 nm. Se obtuvo una eficiencia de encapsulación de hasta el 84.5%.



Nanoplaguicidas, Encapsulación de Neem, Nanoparticulas de Policaprolactona

Area: Promotion of frontier research and basic science in all fields of knowledge

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1. Introduction

The excessive use of chemicals in crop protection has led to serious problems for human health and the environment. Moreover, their use has failed to eliminate or reduce crop pests. The constant application of chemicals has caused insects and other organisms to become resistant to these substances, leading to the use of higher doses and eventually to a saturation and/or accumulation of chemical pesticides in agriculture, causing great environmental impact.

The WHO has classified pesticides according to their acute toxicity, in order to warn farmers of the degree of danger [Table 1]. Pesticides can reach the final consumer in two ways, the first via residues on vegetables or via the food chain, concentrating and causing irreparable and permanent damage to human health. [Brechelt A., 2004].

Box 1

Table 1

Toxicological categories of pesticides

Category toxicological		LD50 for the rat [mg/kg of body weight			
		Oral		Dermal	
		Solids	Liquids	Solids	Liquids
Ia	Extremely dangerous	5 or less	20 or less	10 or less	40 or less
Ib	Highly dangerous	>5-50	>20-200	>10-100	>40-400
II	Moderately dangerous	>50-500	>200-2000	>100-1000	>400-4000
III	Slightly dangerous	> 500	> 2000	> 1000	> 4000

Taken from Brechelt, 2004.

On the other hand, the UN General Assembly established the 2030 Agenda for Sustainable Development, with the aim of favouring the planet, as well as strengthening universal peace. Within this framework, Mexico established a set of actions aimed at achieving at least 10 of the 17 goals of the 2030 Agenda. One of the actions established is to promote agroecology with the support of the Food and Agriculture Organisation of the United Nations [FAO, 2015].

Synthetic pesticides represent one of the main and most effective weapons for the management of agricultural pests, however, they generate high economic costs, environmental pollution, reduction of beneficial organisms and wild species, intoxications, negative effects on applicators and people related to their management and the development of pest resistance [Gutiérrez-Ramírez et al., 2013].

Agriculture requires, among other things, the increased use of chemicals for pest control. In this part, scientific advances have shown that nanomaterials have innovative properties that can be used in the development of a new generation of substances that have been called nano-pesticides. The consideration of nano-pesticides in pest management and control is considered as a first example, the use of colloidal silver which has nanometric particle sizes and has had an increase in its antimicrobial properties due to its large surface area. Nanotechnology applied to pesticides allows the formulation of products that are more specific and less toxic to non-target organisms. The encapsulation of active ingredients in nanoparticles improves the efficacy of pesticides, allowing a lower application dose and reducing pest resistance [Martez L., 2022]. In addition, the use of pesticides at the nanoscale can minimise the risks associated with human and environmental exposure by facilitating a controlled and targeted release of the active ingredient [Vázquez-Núñez, 2022].

In the present research work, polymeric nanopcapsules containing Neem extract were developed for application as a nano-pesticide. Neem extract [*Azadirachta indica A. Juss*] is considered a bioinsecticide with a broad spectrum. Its most important active ingredient is azadirachtin, which has been tested against 220 species of insect pests of the orders *Blattodea*, *Caelifera*, *Coleoptera*, *Dermaptera*, *Diptera*, *Ensifera*, *Heteroptera*, *Isoptera*, *Lepidoptera*, *Phasmida*, *Phthiraptera*, *Siphonaptera and Thysanoptera* [López-Díaz y Estrada-Ortiz, 2005].

2. Methodology

2.1. Preparation of Neem Nanocapsules [NCs-PCL-Neem]

The organic phase was prepared with Polycaprolactone [PCL] and Neem Extract in ethyl acetate previously saturated with water.

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The aqueous phase consisted of 0.5% Polyvinyl alcohol [PVAL]. The organic phase was added to the aqueous phase with magnetic stirring at 10,000 rpm using a high speed homogeniser. Stirring was maintained for 10 minutes. Subsequently, deionised water was added to the system and the same stirring conditions were continued for a further 5 minutes. The solvent and as much water as possible were evaporated under reduced pressure in a rotary evaporator. The nanocapsules were recovered by centrifugation at 14600 rpm. Finally, the nanocapsules were dried under vacuum. The prepared systems are presented in table 2.

Box 2

Table 2

Nanoparticulate systems for the preparation of NCs-PCL-Neem nanocapsules

LOT	PCL	Extract Neem
1	200	
2	200	200
3	400	
4	400	200

2.2. Physico-chemical characterisation of NCs-PCL-Neem nanocapsules

2.2.1. Particle size and polydispersion index

Particle size and polydispersity index were determined by the dynamic light scattering technique using a Malvern Zetasizer Nano ZS90 [Malvern Instruments Ltd, UK] with a detection angle of 90° and a wavelength of 633 nm. One millilitre of each colloidal dispersion was diluted to 10 ml with distilled water to obtain a dilute dispersion.

2.2.2 Z-potential of NCs-PCL-Neem nanocapsules

The zeta potential was determined using a ZS90 Nano Zetasizer, based on the electrophoretic movement of the particles in dispersion using polycaprolactone dispersions as a reference. This parameter indicates the degree of repulsion between adjacent particles. The measurements were performed in triplicate.

2.2.3. Morphology of NCs-PCL-Neem nanocapsules

The FertyNeem nanocapsules were visualised under an optical microscope using 2500 x magnification to obtain their morphology.

2.2.4. Quantification of Neem extract from FertyNeem nanocapsules [NCs-PCL-Neem]

Encapsulation efficiency [E.E.] was determined using the indirect method based on quantifying the unencapsulated active compound [Sotelo-Boyás et al., 2017]. Briefly, 2 ml of each colloidal dispersion was centrifuged at 14 600 rpm for 60 min, to separate the nanocapsules and the stabiliser by removing the supernatant. To the sediment obtained, 1 ml of cyclohexane was added to dissolve the unencapsulated ethyl acetate, but not to dissolve the polymeric barrier in the nanocapsules. The concentration of unencapsulated β-carotene was determined using a UV vis spectrophotometer at a wavelength of 275 nm. The measurements were performed in triplicate and the encapsulation efficiency was determined using the following relationship:

E.E. = [mg Neem added - mg Neem not encapsulated] / mg Neem added **x** 100………... [1]

3. Results and Discussion

3.1. Development of the analytical method for the quantification of Neem extract

3.1.2. Determination of the UV vis absorption spectrum of Neem extract

The absorption spectrum of the Neem extract [Figure 1] shows that the wavelength of maximum absorption is 275 nm, a value that coincides with those reported by Macías-Alvarado and Pérez-Alvarado [2021].

Box 3

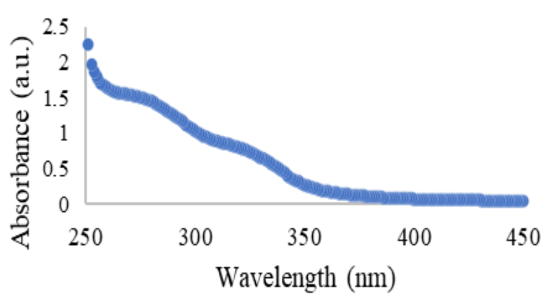

Figure 1

UV vis absorption spectrum of Neem extract in 1% Tween 20. =275 nm.

Source: [Own elaboration]

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3.1.3. Calibration curves for the quantification of Neem extract.

Figure 2 shows a linear relationship between the concentration of Neem extract and its absorbance in the concentration range of 1 to 10 mg/ml of Neem extract [$r^2 = 0.9994$], the equation of the linear relationship is: $y = 0.1419x - 0.0028$.

Box 4

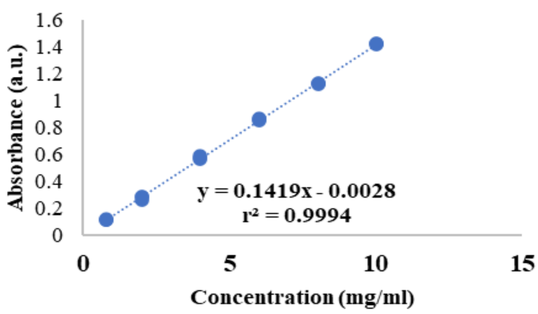

Figure 2
Calibration curve for Neem extract in 1% Tween 20 [□=275 nm]
[Own elaboration]

3.1.3. Preparation of NCs-PCL-Neem Nanocapsules

Neem extract, PCL nanocapsules were prepared by the emulsification-diffusion technique. This technique is based on the formation of nanocapsules prepared by emulsification-diffusion, based on the diffusion of the polymer molecules towards the aqueous phase, originating local regions of supersaturation, from which new polymer globules or aggregates are formed that are not completely desolvable.

The stability of these aggregates by the stabiliser is important to avoid their coalescence. This stability allows the formation of nano-sized particles after complete diffusion of the organic solvent [Quintanar-Guerrero, et al., 1997].

The diffusion emulsification technique allowed us to prepare FertyNeem nanocapsules with spherical morphology and an average size of 250-450 nm [Figures 3 and 4].

Box 5

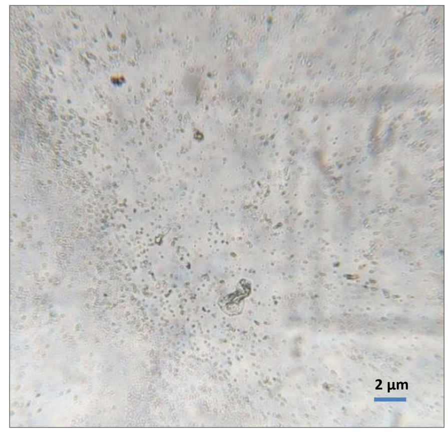

Figure 4
Optical micrograph of NCs-PCL polymeric nanocapsules. Images taken with optical microscopy equipment at 2500x
Source: [Own elaboration]

Box 6

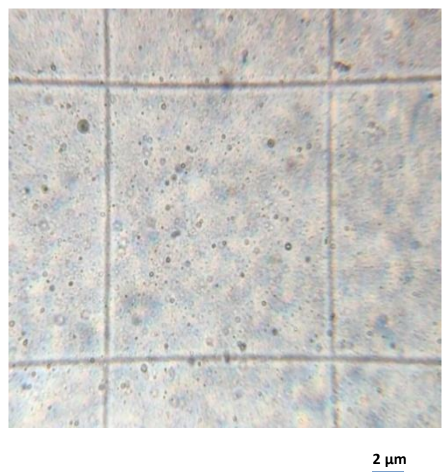

Figure 5
Optical micrograph of NCs-PCL-Neem polymeric nanocapsules. Images taken with an optical microscope at 2500x.
Source: [Own elaboration]

The values for particle size, Z-potential and encapsulation efficiency are shown in table

Box 7

Table 3
Nanoparticulate systems for the preparation of NCs-PCL-Neem nanocapsules.

LOT	PCL [mg]	Neem Extract [mg]	Proportion PCL:Neem	Average size [nm]	PDI	Potential Z [mV]	Encapsu-lation efficiency of Neem [%]
1	200			254.8	0.197	-6.56	
2	200	200	1:1	305.4	0.214	-3.56	78.43
3	400			287.6	0.145	-4.65	
4	400	200	2:1	369.8	0.187	-2.78	84.5

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The results shown in table 3 indicate that with the 2:1 polymer-Neem ratio, there is greater encapsulation efficiency, due to the fact that there is a greater amount of polymer in the formulation, which gives the nanocapsules greater stability. With respect to the charge, a decrease in the Z a potential is observed in those formulations that have Neem, which may be due to the presence of an amount of Neem close to the surface of the nanocapsules. This decrease in charge is presented by Pasquoto-Stigliani et al. [2017], who encapsulated Neem extract in nanoparticulate formulations of PCL.

4. Conclusions

Polymeric PCL nanocapsules were prepared from Neem extract by the emulsification-diffusion technique with an encapsulation efficiency of up to 84.5%. Physicochemical characterisation tests showed the formation of spherical nanocapsules, with an average size of 250-369 nm.

Declarations

Conflict of interest

The authors declare that they have no conflicts of interest. They have no known competing financial interests or personal relationships that might have appeared to influence the article reported in this paper.

Authors' contribution

Aquino-Martínez, Alejandro: Contributed the project idea, performed the preparation and characterisation of the polymeric nanocapsules and drafted the manuscript.

Hernández-Santana, Jhair Alejandro: Contributed to the physicochemical characterisation studies.

Nieto-Ruiz, Jessica: Contributed to the physicochemical characterisation studies.

Martínez-Pérez, Beatriz: Contributed to the supervision of the preparation and characterisation studies, as well as assisting in the drafting of the manuscript.

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Abbreviations

FAO: Food and Agriculture Organization of the United Nations
NCs: Nanocapsules
PCL: Polycaprolactone
WHO: World Healt Organization

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Background

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Discussions

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