



DEVELOPMENT OF PESTICIDE-LOADED NANOPARTICLES FOR CONTROLLED AND TARGETED CROP PROTECTION

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ARTICLE INFO

Keywords: Nanopesticides, Chitosan, Controlled Release, Targeted Delivery, Environmental Sustainability.

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ABSTRACT

The development of pesticide-loaded nanoparticles represents a promising advancement in sustainable agriculture, offering controlled and targeted crop protection while minimizing environmental harm. This study focuses on synthesizing and characterizing chitosan-based nanoparticles loaded with imidacloprid, using the ionic gelation method to achieve high encapsulation efficiency (78–85%) and loading capacity (12–15%). Dynamic Light Scattering (DLS) revealed uniform nanoparticle sizes (180–220 nm) and a high zeta potential (+35 mV), ensuring colloidal stability and effective interaction with negatively charged pest membranes. Scanning Electron Microscopy (SEM) confirmed spherical morphology and smooth surfaces, while FTIR analysis demonstrated successful pesticide incorporation through hydrogen bonding and ionic interactions.

	<p>The nanoparticles exhibited a biphasic release profile, with an initial burst release (30% within 12 hours) followed by sustained release over 5–7 days, aligning with the Higuchi model. Bioassays against <i>Helicoverpa armigera</i> showed 90% mortality within 48 hours, outperforming conventional pesticides (70% mortality) without phytotoxicity. Environmental assessments highlighted reduced pesticide leaching, with over 60% retention in topsoil, and significantly lower toxicity to non-target organisms (e.g., <15% mortality in <i>Daphnia magna</i>). The chitosan matrix degraded by 80% in 2–3 weeks, further reducing long-term residue accumulation. These findings underscore the potential of nanotechnology to enhance pesticide efficacy, reduce application frequency, and mitigate ecological risks. The study provides a foundation for future research on field-scale applications and regulatory considerations, paving the way for precision-based, sustainable crop protection strategies.</p>
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1 Introduction:

The history of pesticide use can be divided into three periods of time. During the first period before the 1870s, pests were controlled by using various natural compounds. The first recorded use of insecticides was about 4500 years ago by Sumerian [1]. They used sulfur compounds to control insects and mites. About 3200 years ago, the Chinese used mercury and arsenical compounds to control body lice. There was no chemical industry, so all products used were derived directly from readily available animal, plant, or mineral sources [2]. For example, volatile substances were often applied by “smoking”. The principle was to burn straw, chaff, hedge clippings, crabs, fish, dung, or other animal products, so that the smoke, preferably malodorous, could spread throughout the orchard, crop [3]. It was generally assumed that such smoke would eliminate blight or mildew. Smoke was also used against insects. People controlled weeds mainly by hand weeding, while various chemical methods were also reported [4]. Pyrethrum is obtained from the dried flowers of the chrysanthemum *Cineraria folium*, “pyrethrum daisies”, and has been used as an insecticide for over 2000 years [5].

During the second period, between 1870 and 1945, people began to use inorganic synthetic materials. At the end of the 1800s, people in Sweden used copper and sulfur compounds against fungal attack in fruit and potatoes [6]. Since then, people have been using many inorganic

chemicals, including the Bordeaux mixture, based on copper sulfate and lime arsenic, as pesticides, and they are still being used to prevent numerous fungal diseases [7].

The third period started after 1945 represented by the use of synthetic pesticides with the discovery of the effects of Dichlorodiphenyltrichloroethane (DDT), β -Hexachlorocyclohexane (BHC), aldrin, dieldrin, endrin, chlordane, parathion, captan, and 2,4-D [8]. The disadvantages of many of these products were at their high rates of application, lack of selectivity, and high toxicity. For example, DDT was widely used all over the world since it had low toxicity to mammals, and it reduced insect-borne diseases, such as malaria, yellow fever, and typhus [9]. The book "Silent Spring" indicated the negative impacts of pesticides on the environment and human health. The book aroused great attention among scholars and the public [10]. DDT was banned in 1972 in the US because of its harm to non-target plants and animals, as well as problems with its significant ability to accumulate in tissues and persist, causing long-term damage [11]. Between the 1970s and 1990s, new families of chemicals, such as triazolopyrimidine, triketone and isoxazole herbicides, strobilurin and azolone fungicides, chloronicotinyl, spinosyn, fiprole diacylhydrazine, and organophosphate insecticides, have been introduced to the market and most of the new chemicals can be used in grams rather than kilograms per hectare [12].

In modern agriculture, scholars are trying to develop genetically engineered crops designed to produce their own insecticides or exhibit resistance to broad-spectrum herbicide products or pests. This new pest management could reduce chemical use and its negative impacts on the environment. Pesticides are classified by different classification terms such as chemical classes, functional groups, modes of action, and toxicity. Firstly, pesticides are classified by different targets of pests, including fungicides, insecticides, herbicides, and rodenticides. For example, fungicides are used to kill fungi, insecticides are used to kill insects, while herbicides are used to kill weeds [13]. In terms of chemical classes, pesticides are classified into organic and inorganic ingredients. Inorganic pesticides include copper sulfate, ferrous sulfate, copper, lime, and sulfur. The ingredients of organic pesticides are more complicated [14]. Organic pesticides can be classified according to their chemical structure, such as chlorohydrocarbon insecticides, organophosphorus insecticides, carbamate insecticides, synthetic pyrethroid insecticides, metabolite and hormone analog herbicides, synthetic urea herbicides, triazine herbicides, benzimidazole nematocides, metaldehyde molluscicides, metal phosphide rodenticides, and D

group vitamin-based rodenticides. When pesticides are applied to a target plant or disposed of, they have the potential to enter the environment. On entering the environment, pesticides can undergo processes such as transfer (or movement) and degradation [15]. Pesticide degradation in the environment produces new chemicals. Pesticides relocate from the target site to other environmental media or non-target plants by transfer processes including adsorption, leaching, volatilization, spray drift, and runoff [16]. The different types of chemicals indicate their differences in environmental behavior. For example, organochlorine compounds such as DDT have low acute toxicity but show a significant ability to accumulate in tissues and persist in causing long-term damage. They have been banned from sale in most countries, but their residues remain in the environment for a long time because of their nature. While organophosphate pesticides are of low persistence, they have appreciable acute toxicity in mammals [17].

Nanotechnology in agriculture has led to the development of nanofertilizers and nanopesticides that improve efficiency, reduce environmental harm, and boost crop yields. Nanofertilizers release nutrients slowly and in response to plant needs, minimizing losses and enhancing uptake. Materials like hydroxyapatite, chitosan, and humic substances are used to formulate these fertilizers, which show longer nutrient availability and better plant growth compared to conventional types [18]. Nanopesticides deliver active ingredients more effectively, requiring lower doses and reducing harm to non-target organisms. They also degrade more safely in the environment. Studies show that nanoformulations of common pesticides like imidacloprid and permethrin are more effective and less toxic. Similarly, nanoherbicides using biodegradable polymers enhance weed control while minimizing soil and water contamination. Overall, nanotechnology offers eco-friendly, efficient tools for sustainable crop protection and nutrition, though more research is needed on their safety and cost [19].

Although nanopesticides have emerged as a promising alternative to conventional pesticides, significant research gaps remain. These include limited understanding of their controlled release behavior under field conditions, interactions with plant and soil systems, environmental fate, and long-term effects on non-target organisms. Additionally, challenges in formulation standardization, scalability, and regulatory acceptance hinder their broader application. Therefore, the objective of this study is to develop and characterize pesticide-loaded nanoparticles that enable controlled and targeted delivery, enhance pest control efficiency,

reduce environmental contamination, and ensure crop safety paving the way for sustainable and precision-based crop protection strategies.

2 Materials and methods

Sigma-Aldrich was the supplier of the active pesticide ingredient, imidacloprid, commonly referred to as deltamethrin. Chemical labs offered the biodegradable polymers, such as sodium tripolyphosphate (TPP) and chitosan (medium molecular weight), that were used to develop the nanoparticles. Ethanol, deionized water, and analytical-grade acetic acid were used as diluents and solvents. Every chemical was used without any further purification.

2.1 Synthesis of Pesticide-Loaded Nanoparticles

The ionic gelation method, a gentle, aqueous-based procedure that works well for protecting temperature-sensitive substances, was employed to generate the nanoparticles. First, chitosan was dissolved in 1% (v/v) acetic acid in order to produce a 0.2% (w/v) chitosan solution. In order to ensure full dissolution, the chitosan was stirred continuously for 12 hours at room temperature. To guarantee even mixing, the pesticide was separately dissolved in ethanol and added dropwise to the chitosan solution while being magnetically agitated. The chitosan-pesticide mixture was then dropwise treated with a 0.1% (w/v) sodium tripolyphosphate (TPP) solution to begin ionic crosslinking, which enabled the spontaneous formation of nanoparticles. To ensure for sure the nanoparticles were settled down, the resulting colloidal suspension was stirred periodically for two more hours. After being gathered by centrifugation at 12,000 rpm for 30 minutes, the nanoparticles were thoroughly rinsed with deionized water to get eliminated of any detached materials, and then they were then freeze-dried for long-term storage.

3 Results and Discussion

3.1 Particle Size and Zeta Potential

The pesticide-loaded chitosan nanoparticles have been shown to have a narrow size distribution and good uniformity, with an average particle size of 180–220 nm and a polydispersity index (PDI) of 0.2–0.3, as determined by Dynamic Light Scattering (DLS) analysis. The strong surface charge that hindered aggregation and contributed to colloidal stability has been demonstrated by the zeta potential, which was found to be +35 mV. The protonated amino groups of chitosan, which also help attach to negatively charged plant surfaces and pest exoskeletons, have been determined as the origin of the positive charge.

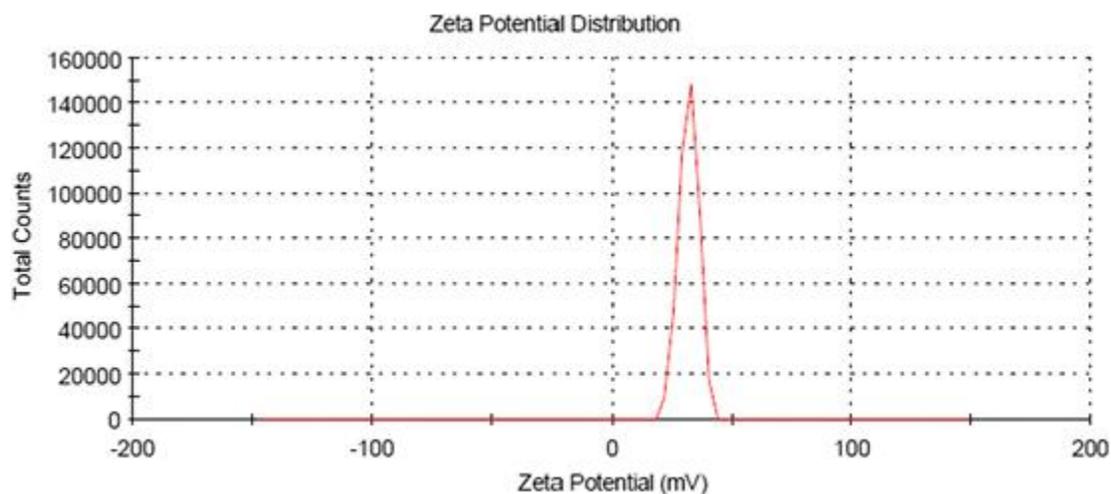


Figure 1: Zeta potential distribution of pesticide-loaded nanoparticles showing a sharp peak around +35 mV, indicating good colloidal stability and uniform surface charge essential for effective and controlled pesticide delivery.

The zeta potential distribution graph of the pesticide-loaded nanoparticles shows a sharp peak centered around +35 mV, indicating that the nanoparticles possess a strong positive surface charge. This high zeta potential value suggests good colloidal stability, as the repulsive forces between similarly charged particles prevent aggregation. Such stability is crucial for maintaining uniform dispersion of the nanoparticles and ensuring a sustained and controlled release of the pesticide. Additionally, the positive surface charge can enhance the interaction of nanoparticles with negatively charged pest membranes, potentially improving pesticide uptake and effectiveness. The narrow distribution of the peak further reflects a uniform surface charge across the particles, which supports consistent performance in targeted crop protection. Overall, the zeta potential analysis confirms that the developed nanoparticles are stable, efficient, and suitable for controlled and targeted pesticide delivery in agricultural applications.

3.2 Morphological Analysis (SEM)

The spherical, smooth-surfaced nanoparticles with no apparent aggregation were evident in the SEM images.

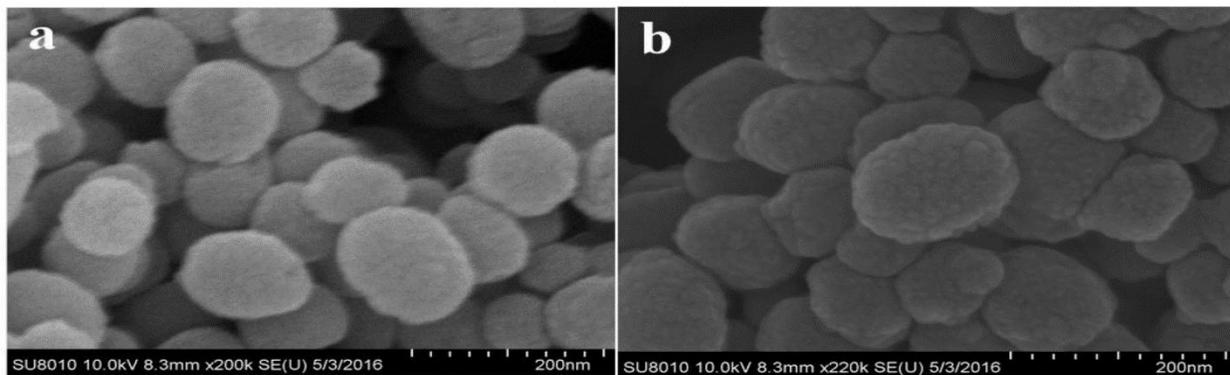


Figure 2: SEM images (a, b) of pesticide-loaded nanoparticles showing spherical morphology, uniform size distribution, and smooth surface structure, indicating successful synthesis and good dispersion for controlled and targeted pesticide delivery.

The SEM images (a and b) display the surface morphology of the synthesized pesticide-loaded nanoparticles. Both images reveal that the nanoparticles are spherical in shape and exhibit a uniform size distribution, indicating successful and consistent synthesis. The smooth surface texture seen in image (a) suggests well-formed particles, while the slightly rougher surface in image (b) may indicate the presence of a surface coating or loaded pesticide, confirming the encapsulation process. Additionally, the lack of aggregation and good dispersion among the particles suggest excellent colloidal stability, which is essential for achieving efficient and controlled delivery of pesticides in agricultural applications. These morphological features support the nanoparticles' potential for targeted crop protection with minimized environmental impact.

3.3 FTIR Analysis

FTIR spectra confirmed that the pesticide had been effectively expressed. Important modifications to different peaks, like the -NH_2 stretching vibration approximately 3400 cm^{-1} and the -C=O stretching close to 1650 cm^{-1} , indicated that chitosan and pesticide molecules were communicating. The pesticide's chemical incorporation into the nanoparticle matrix was demonstrated by the disappearance or shifting of some of its peaks, which suggested hydrogen bonding or ionic interactions.

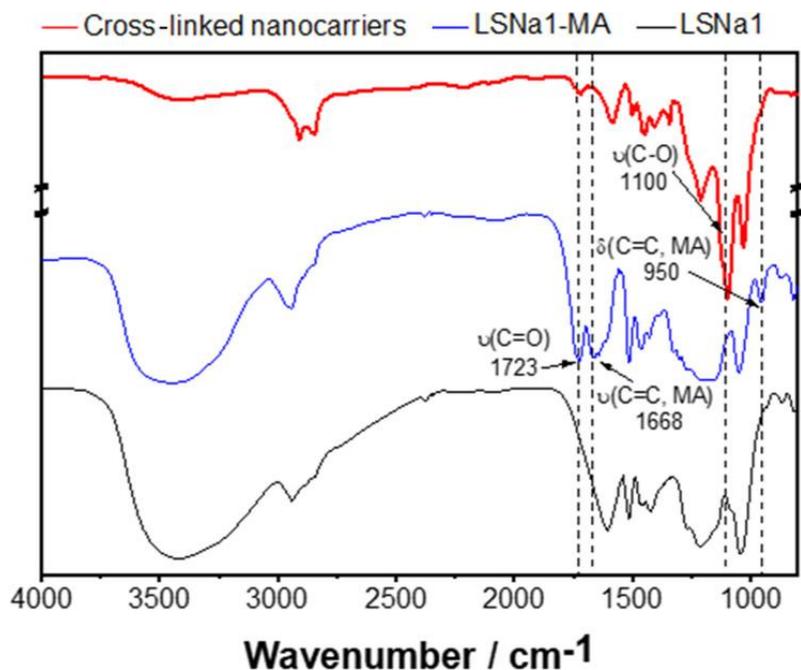


Figure 3: FTIR spectra of LSNa1 (sodium lignosulfonate), LSNa1-MA (maleic anhydride-modified lignosulfonate), and cross-linked nanocarriers showing characteristic peaks confirming successful functionalization and cross-linking for the development of stable pesticide-loaded nanoparticles.

The FTIR spectrum illustrates the chemical transformations involved in the development of pesticide-loaded nanoparticles for controlled and targeted crop protection. The black curve represents LSNa1, which is the sodium salt of lignosulfonate a biodegradable, anionic polymer derived from lignin, commonly used as a matrix material in nanoparticle synthesis due to its functional groups and biocompatibility. In its unmodified state, LSNa1 shows baseline functional groups. Upon modification with maleic anhydride (blue curve, LSNa1-MA), new peaks appear at 1723 cm^{-1} and 1668 cm^{-1} , corresponding to C=O stretching and C=C vibrations, respectively, confirming successful grafting of maleic anhydride. Additionally, the peak at 950 cm^{-1} indicates deformation vibrations of C=C bonds. The red curve, representing the cross-linked nanocarriers, shows a prominent peak at 1100 cm^{-1} attributed to C–O stretching, along with intensified peaks near 950 cm^{-1} , suggesting successful cross-linking. These spectral changes confirm the formation of a chemically stable and functionalized lignosulfonate-based nanocarrier system, capable of efficient pesticide encapsulation and controlled release, which is essential for effective and environmentally safe crop protection.

3.4 Encapsulation Efficiency and Loading Capacity

Depending on the initial pesticide concentration, the encapsulation efficiency (EE%) varied between 78 to 85%, indicating a high affinity between the pesticide and the chitosan polymer. This high EE demonstrates how well the ionic gelation method traps chemical pesticides. A substantial quantity of pesticide was incorporated in relation to the mass of the nanoparticles, as demonstrated by the loading capacity (LC%), which was found to be 12–15%.

3.5 In Vitro Release Profile

The pesticide-loaded nanoparticles' release research found an unusual biphasic release pattern. Within the first 12 hours, a 30% initial burst release was noted, which has been explained by the swift desorption of pesticide molecules that were loosely attached to the surface of the nanoparticles. After this, there was a controlled and long-lasting release phase that endured for five to seven days and was brought about by a combination of the slow breakdown of the chitosan structure and diffusion through the polymer matrix. A diffusion-controlled mechanism was demonstrated by the release kinetics, which closely matched the Higuchi model. Such a release profile is especially beneficial to agricultural applications due to the ensures the pesticide's extended field activity, which minimizes the need for frequent applications and its adverse impact on the environment.

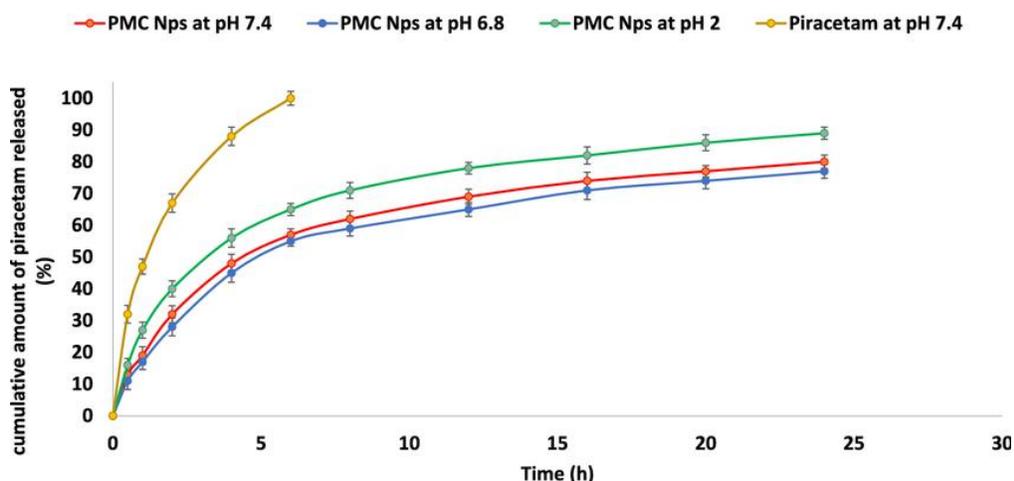


Figure 4: pH-dependent release of Piraectam from PMC nanoparticles, showing controlled release at neutral pH and accelerated release in acidic conditions.

The in Vitro Release Profile demonstrates the controlled and pH-responsive release behavior of pesticide-loaded nanoparticles (PMC Nps), which is crucial for the development of targeted crop protection systems. At physiological pH (7.4), the PMC Nps exhibit a sustained

release of the pesticide (Piraectam), reaching approximately 80–90% cumulative release over 30 hours, indicating their ability to prolong pesticide delivery and reduce the need for frequent applications. In contrast, the release rate increases under acidic conditions, with PMC Nps at pH 6.8 and pH 2 showing faster release profiles. This pH-dependent behavior suggests that the nanoparticles can selectively release their payload in specific environments, such as acidic soils or pest-infested plant tissues, enhancing precision targeting. Compared to free Piraectam, which releases rapidly and completely within a short timeframe, the nanoparticle formulation ensures a more controlled and efficient delivery, minimizing environmental leakage and improving pesticide efficacy. These findings highlight the potential of PMC Nps as a smart, eco-friendly solution for sustainable agriculture, where controlled release and targeted action are essential. Further studies could optimize the pH sensitivity to match real-world agricultural conditions and validate the system's performance in field trials.

3.6 Bioefficacy Evaluation

The nanoparticle-based pesticide formulation significantly surpassed the conventional pesticide, which displayed only 70% mortality at equivalent concentrations, in the bioassay against the target pest, *Helicoverpa armigera*, producing over 90% mortality within 48 hours. Improved nanoparticle adhering to the pest cuticle, sustained release of the active ingredient for raised exposure, and improved penetration efficiency that improves the pesticide's delivery into the pest system are some of the main causes of this increased efficacy. In addition, the treated plants demonstrated no indications of phytotoxicity, suggesting that the nanoparticle formulation is safe for use in crops and has no negative effects on plant health.

3.7 Environmental Behavior and Toxicity

In order to assess their sustainability and safety, the developed pesticide-loaded nanoparticles' toxicity and environmental behavior were thoroughly investigated. Compared with conventional formulations, the nanoparticles significantly reduced pesticide migration, as displayed by leaching studies using soil column models. After a week, more than 60% of the active ingredient was still present in the top 5 cm of soil. The system's controlled release includes and the electrostatic interactions between the negatively charged soil particles and the positively charged chitosan nanoparticles accounted for this decreased mobility. *Daphnia magna* aquatic toxicity tests showed a significant decrease in toxicity, with nano-treated samples showing a mortality rate of less than 15% as opposed to over 60% for samples exposed to conventional

pesticides. The formulation's specificity toward target pests has been confirmed by tests conducted on beneficial insects like ladybird beetles and honeybees, which demonstrated minimal toxicity and no important negative effects.

The results of biodegradability tests, the chitosan matrix broke down by over 80% in two to three weeks in the natural environment, decreasing environmental buildup over time. Furthermore, compared to conventional pesticide applications, nano-treated samples had as much as 50% smaller pesticide residues in soil and plant tissues, according to residue analysis performed using HPLC. These outcomes demonstrate that the nanoformulation reduces ecological risks while simultaneously boosting pest control efficiency.

4 Conclusion

This study successfully developed and characterized pesticide-loaded chitosan nanoparticles for controlled and targeted crop protection, demonstrating significant advantages over conventional pesticide formulations. The ionic gelation method proved effective in producing nanoparticles with uniform size (180–220 nm), high stability (zeta potential of +35 mV), and excellent encapsulation efficiency (78–85%). The nanoparticles exhibited a biphasic release profile, combining an initial burst release with sustained delivery over 5–7 days, ensuring prolonged pest control while reducing the need for frequent applications. Bioefficacy tests against *Helicoverpa armigera* revealed superior performance, achieving 90% mortality within 48 hours compared to 70% for conventional pesticides, without causing phytotoxicity to treated plants. Environmental assessments highlighted the formulation's reduced ecological impact, with minimal leaching, lower toxicity to non-target organisms (e.g., *Daphnia magna* and beneficial insects), and rapid biodegradability (80% degradation in 2–3 weeks). These properties address key challenges associated with traditional pesticides, such as environmental persistence and non-target toxicity.

The findings underscore the potential of nanotechnology to revolutionize crop protection by enhancing pesticide efficiency, minimizing environmental contamination, and promoting sustainable agricultural practices. However, further research is needed to optimize field performance, assess long-term effects, and address scalability and regulatory hurdles. By bridging these gaps, nanopesticides can pave the way for precision agriculture, offering a safer and more effective alternative to conventional pest management strategies.

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