Controlled Release study of Urea Formaldehyde Microcapsules Containing Chlorpyriphos as an Insecticide

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Abstract

Chemical insecticides are more effective than other biological controlling the pests. In this communication we prepare a successful encapsulation of chlorpyriphos one of the most effective insecticide in urea formaldehyde microcapsules this was achieved by adopting an in-situ polymerisation process in oil in water emulsion. The synthesis consist of two parts, namely emulsification of oil and wall formation. The synthesized microcapsules were characterized by FTIR.

Keywords: Controlled release, in-situ polymerization, microencapsulation, Chlorpyriphos, Urea-formaldehyde (PF)

1. Introduction

Microencapsulation is a process in which tiny particles or droplets are surrounded by a coating to give small capsules, of many useful properties. In general, it is used to incorporate food ingredients, enzymes, cells or other materials on a micro metric scale. Microencapsulation can also be used to enclose solids, liquids, or gases inside a micrometric wall made of hard or soft soluble film, in order to reduce dosing frequency and prevent the degradation of pharmaceuticals. In a relatively simple form, Pesticides are important labor-saving agrochemicals useful for the improvement of the quality and quantity of agricultural products. However, extreme use and misuse of pesticides has resulted in environmental pollution. Adverse effects on the ecosystem, and loss of biodiversity, the protection of the active ingredient (a. i.) from early degradation, evaporation and leaching, safety to the applicator, and prolonging the effect of pesticides against different pests due to the controlled release of a. i.

In our research work, chlorpyriphos was used as the core material. Chlorpyrifos is a broad-spectrum insecticide which kills insects upon contact by affecting the normal function of the nervous system. ... When insects are exposed, chlorpyrifos binds to the active site of the cholinesterase (ChE) enzyme, which prevents breakdown of ACh in the synaptic cleft.

The purpose of encapsulation of chlorpyriphos was to prevent its rapid degradation in the environment and thus, sustained release and improved stability in the environment, which is
necessary to improve its effectiveness.

Encapsulation method applied in this research is in situ polymerization of urea-formaldehyde under low pH (acidic condition). There are 2 steps of process during microcapsule formation. The first step is emulsion of Chlorpyriphos in urea-formaldehyde resin solution, in which Chlorpyriphos is as a dispersed phase. High rotation speed of stirring is conducted to emulsify Chlorpyriphos this case, high rotation speed homogenizer is used as a stirrer. The second step is microcapsule shell formation. In this step, the pH of emulsion liquid is adjusted into acidic condition. This promotes the reaction of urea with formaldehyde in the interface of emulsion bubbles, producing a film of urea-formaldehyde polymer as a microcapsule shell.

Two reactions take place when urea reacts with formaldehyde, i.e., addition and condensation:

$$H_2N\text{-}CO\text{-}NH_2 + CH_2O \rightarrow H_2N\text{-}CO\text{-}NH\text{-}CH_2OH \quad (1)$$

(mono methylol urea)

$$H_2N\text{-}CO\text{-}NH\text{-}CH_2OH + H_2N\text{-}CO\text{-}NH\text{-}CH_2OH \rightarrow$$
$$H_2N\text{-}CO\text{-}NH\text{-}CH\text{-}HN\text{-}CO\text{-}NH\text{-}CH_2OH + H_2O \quad (2)$$

Reaction (1) is an addition reaction, where formaldehyde reacts with H of amine to form methylol. Condensation occurs between OH of methylol and H of amine, forming methylene link between amines of 2 urea molecules, as shown in Eq. 2. This condensation reaction results in urea-formaldehyde polymer chain. Addition and condensation reactions are catalyzed either by OH− or H+. The rate of addition reaction in basic condition is as high as the rate in acidic condition. However, the rate of condensation reaction is much faster in acidic condition, compared with that in basic condition. As urea has four H atoms in amines, it is most possible to produce crosslink or network polymer during condensation reaction. This network polymer is good for microcapsule shell due to its high strength, in which microcapsule is not easy to break up during its application.

2. Experimental

2.1 Materials

Urea and 37% formaldehyde were used as wall-forming materials, chlorpyriphos, as core material, polyacrylamide as the protective colloid, NH_4Cl and HCl were pH controllers, ferrous chloride was used as crosslinking agent. All chemicals were of synthetic grade and used as such for the reaction.

2.2 Synthesis of Microcapsules:-

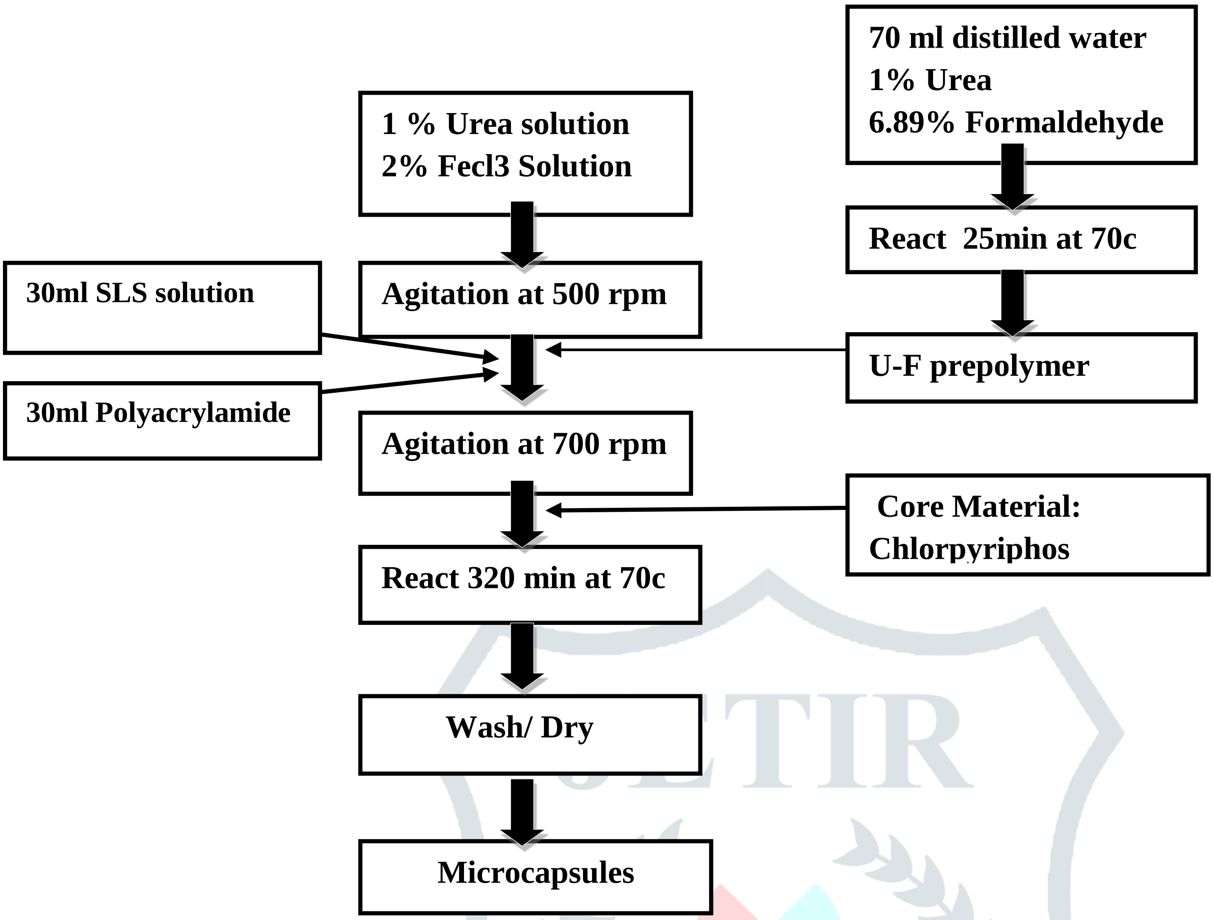
Microcapsules were prepared by adapting an in-situ polymerization technique using an oil-in-water interface. At room temperature, 0.15 gm of sodium lauryl sulphate (SLS) was dissolved in 30 ml of water in 50 ml beaker, and 0.25 gm of polyacrylamide is dissolved in 30 ml of water, this two compounds are aggitated together at 500 rpm for 35 minutes. Then 1 gm urea was added with 2%Fecl3. The whole mixture was fully aggitated at 500 rpm for 15 minutes then this mixture was kept aside.

Then 1 % of urea was dissolved in 100 ml conical flask then add 6.89% formaldehyde and stir it well. Then kept this mixture in oil bath for 25 minutes by using magnetic stirrer at 300 rpm. After 25 minutes the reaction mixture was removed out and kept for cooling at room temperature.

After cooling the mixture , Two mixtures were mixed together in two necked conical flask, this round bottom two necked flask is kept in oil bath at 70c at 700 rpm by using magnetic stirrer. Then one neck is closed by use of aluminium foil. other neck was opened for adjustment of the PH and for addition of the core material (Chlorpyriphos).After starting the reaction core material was slowly added in reaction mixture by use of Burrete.

After one hour the pH of the solution was adjusted to a value of 7 by adding an appropriate amount of NH4cl by using PH paper. The reaction was maintained at 70c and 700 rpm for 320 minutes. After two hours Hcl is added for maintaining the PH at 2 to 3. After 320 minutes, The reaction mixture was cooled an ambient temperature. Microcapsules form the suspension were recovered by filtration under vacuum. The synthesized microcapsules were rinsed with water and washed with xylene to remove the suspended oil and then dried under vacuum.

The flow chart of the entire process for making the PF microcapsules containing Chlorpyriphos is depicted in Scheme 1.



Scheme No.1. Procedure to synthesize the UF microcapsules containing Chlorpyriphos used in the study.

3.Result and discussion

In this work , chlorpyriphos -filled microcapsules surrounded by an UF shell material were synthesized and evaluated using a series of different manufacturing parameters including reaction temperature, amount of emulsifier, and amount and ratio of reactants.

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1. Compound Microscope

i) Study of Surface Morphology

The surface morphology of UF microcapsules containing Chlorpyriphos, prepared under the agitation of an overhead mechanical stirrer was observed using an optical microscope images for primary conformation of shell formation.

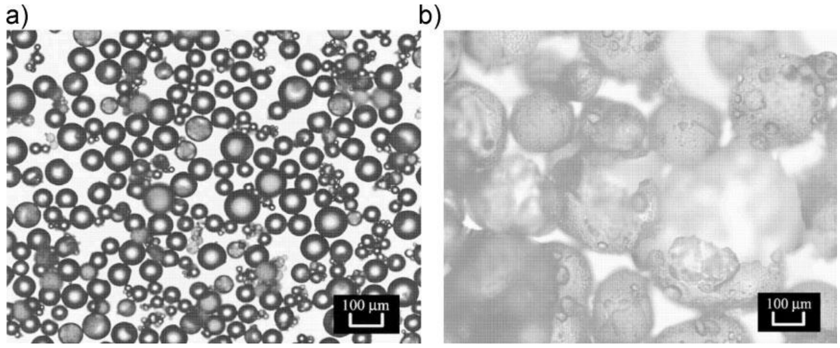
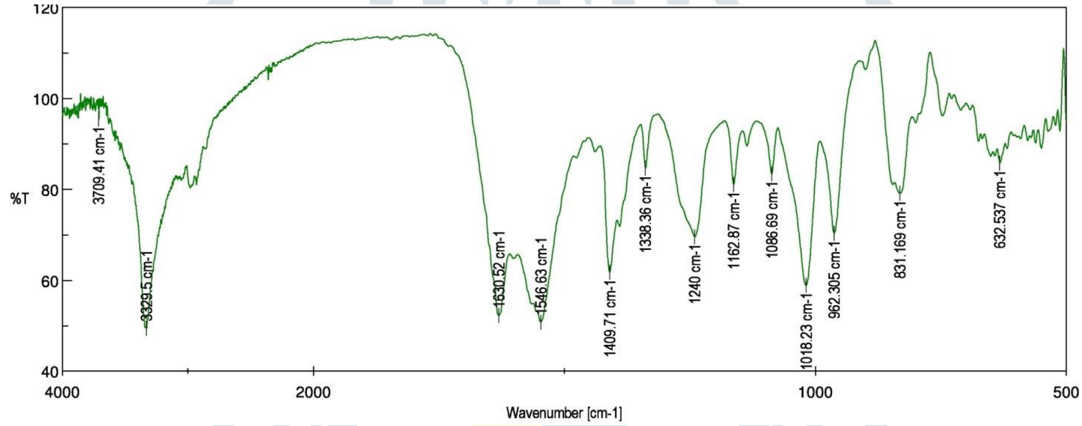


Fig. 1. Optical microscopic images (a and b) of the PF microcapsules containing Chlorpyriphos.

2. FTIR analysis

FTIR spectra of the extracted core material, chlorpyriphos and Urea formaldehyde polymer as the wall-forming material are shown in Figure. The observed absorption bands at 1607 and 1459 cm_1 corresponded to the C¼C aromatic ring vibrations. The bands at 1239 and 1157 cm_
1 represented the characteristics of the C-C-O asymmetric stretching and C-H in plane deformations, respectively, while the 980 and 748 cm_1 bands belonged to the C-H out-of-plane vibrations.



3. **Release Rate Study by UV Spectroscopic Method**:-

UV Absorbance data for extracted core from microcapsule at time different interval.

Time(h)	Absorbance for UF Microcapsules 270-290(nm)
1	0.1760
2	0.4535
3	0.6304
4	0.7044
5	0.9876

Absorbance checked after hour intervals , it shows slow release of core material.

4. Physical Appearance

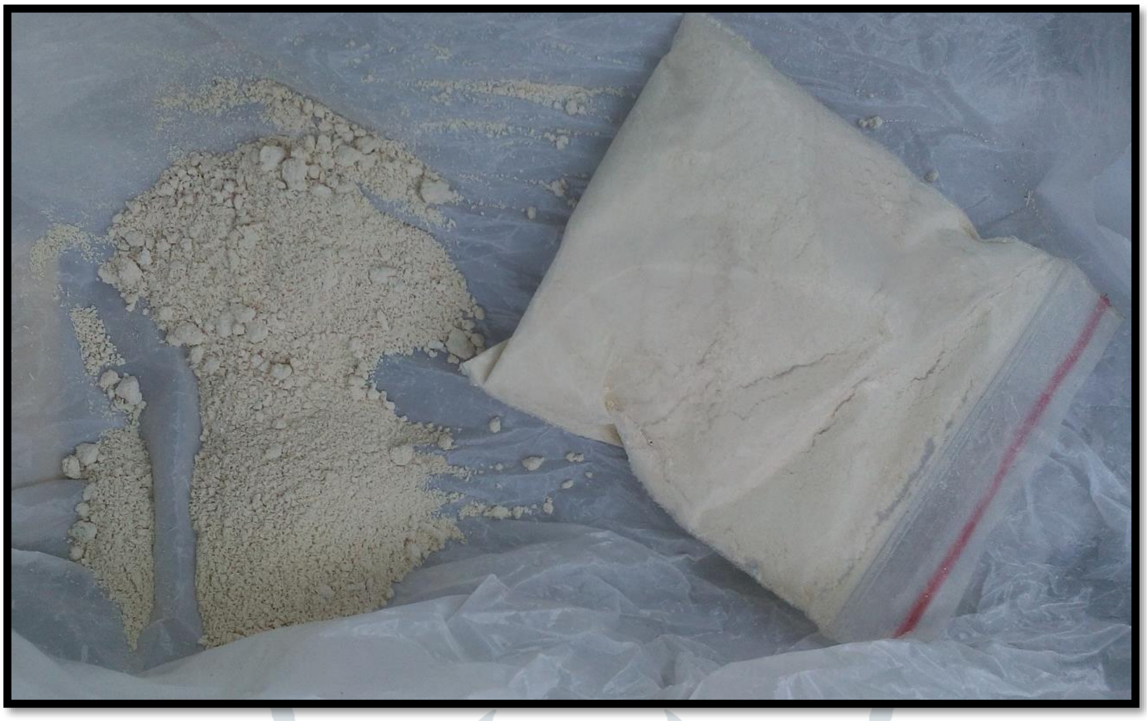


Fig.3:- Ready Product of UF Capsules

4. Conclusion

A process for the microencapsulation of chlorpyriphos as insecticide by in-situ polymerization of Urea formaldehyde(UF) in an oil-in-water emulsion has been successfully developed in the present investigation to fulfill the requirements for controlled release applications. The chemical constitution of synthesized microcapsules was confirmed by FTIR spectroscopy. The compound microscope study and physical appearence demonstrated the regular spherical shape.

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