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# Assembly of Nanoencapsulated Pendimethalin Herbicide using Solvent Evaporation Method for Season Long Weed Control under Irrigated Ecosystem

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**ABSTRACT**

*Laboratory experiment was carried at the Department of Nano Science and Technology, Tamil Nadu Agricultural University, Coimbatore during 2013-2014 to fabricate the slow release nanoencapsulated herbicide for season long weed free environment. In this experiment the pre-emergence herbicide pendimethalin was encapsulated using solvent evaporation method. Thus encapsulated pendimethalin particles were characterized using scanning electron microscope (SEM), X-ray diffraction (XRD) and Particle size analyzer. The solvent evaporation method effectively encapsulated the pendimethalin particles and released slowly throughout the study period of 40 days. Biosafety studies of encapsulated materials was carried out on earthworms and found to be non-toxic.*

***Key words*** *Bio safety, Moisture, Releasing efficiency, Solvent evaporation, Starch.*

## INTRODUCTION

Blackgram (*Vigna mungo* (L.) Hepper) occupies the pride of place among the pulse crops. In world, growing of legume crops is very important not only for nutritional values but also for improvement of soil fertility due to their capability of atmospheric nitrogen fixation[1]. The rapid growth of population has increased the demand of protein through pulses in cereal-based diets due to high costs of animal proteins and shortage of vegetable oil in the world. In light of increasing demand and potential of pulses, improved package of technology for growing of legume crop is pre-requisite for increasing crop production. Among the production constraints, weeds play major role in limiting the production potential of pulse crops. Among the cultivated crops legumes are highly susceptible to weed infestation. Due its slow growing nature in the initial stage as well as nitrogen released from the nodule in the later stage encourages the weed growth throughout its life cycle. Higher dose of herbicide for increased weed control duration inhibits the growth of pulse crop.

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The half life period for many soil applied herbicides remains very short period of time ranging from few hours to couple of weeks. Once the concentration of soil applied herbicide to reduce to 50 per cent of its original strength, correspondingly it loses its weed control efficiency[3]. An effective herbicide should control weeds with reasonable doses selectively non-toxic to crops, remain in the area where applied, persist throughout the growing season taking care of frequently germinating weeds and then leaving no residue at the end of the season permitting subsequent crop in the sequence.

Nanotechnology is a technology having the potential ability to study, design, create, synthesis, manipulation of functional materials, devices, and systems to fabricate structures with atomic precision by controlling the size of the matter at the scale 1–100 nanometers (one nanometer being equal to $1 \times 10^{-9}$ of a meter). It will provide the solution to a large number of problems faced by mankind today.

Hence, development of a new slow release nanoencapsulated herbicide formulation will help the agronomist to increase the productivity of blackgram by season long control of weeds.

## MATERIALS AND METHODS

### Materials

Pendimethalin, acetone, poly ethylene glycol, dichloromethane and starch were purchased from Alfa Aesar. All the chemicals were analytical grade and used as received without any further purification. The water used in all the experiment was double- distilled.

### Methods

***Preparation of organic phase with polymer PEG and pendimethalin***

Pendimethalin 1 mg was dispersed in 4 ml of acetone solvent and sonicated with energy output 25 W under continuous mode for 90 seconds. Poly ethylene glycol polymer of 20 mg was dissolved in 8 ml of dichloromethane solvent and sonicated with energy output 25 W under continuous mode for 90 seconds. Both of them were mixed well to form organic phase.

***Preparation of aqueous phase with emulsifier starch***

Took 4 ml of starch polymer with different concentration (4, 6 and 8 per cent) and stir it with magnetic stir for one hour for the preparation of aqueous phase.

***Preparation of oil-water emulsion leading to nanospheres***

Took the organic phase containing the polymer with herbicide and added drop wise to aqueous phase containing the different per cent of starch to form oil in water phase. The oil in water phase was sonicated with energy output of 25 W under continuous mode for 90s. Thus formed oil in water phase was stirred overnight in magnetic stirrer under room temperature. The nanoparticles produced were collected by centrifugation with 5000 rpm for 15 min under room temperature and dried in vacuum desiccators. The dried particles were collected and stored in vial. The treatment combinations were $T_1$ - Organic phase + 4% Starch Aqueous phase, $T_2$ - Organic phase + 6% Starch Aqueous phase and $T_3$ Organic phase + 8% Starch Aqueous phase. The materials synthesized and fabricated encapsulated pendimethalin were characterized using Scanning Electron Microscope (SEM), X-ray diffraction (XRD) and Particle Size Analyzer to ascertain the size, shape and its composition.

***Extraction of pendimethalin from nanoencapsulated particles***

Samples of fabricated nanoencapsulated pendimethalin were subjected to study the releasing pattern of herbicide. 10 ml of water was added to the petridish containing nanoencapsulated particles and the suspension was allowed to leach for overnight. The samples were transferred to a seperatory funnel by adding another 10 ml of water along with 40 ml of Methanol and shake it for one hour. Then 50 ml of aliquot was collected by filtering using Whatman 40 paper. Took 10 ml of oil phase filterate + 30 ml of 1 per cent NaCl + 50 ml of Hexane. Separated the oil phase and water phase using the seperatory funnel. Then 25 ml of Hexane was added to the water phase residue from above step, again

using the seperatory funnel and collected the upper layer of oil phase. Then it was dried using rotary evaporator at 60°C. Then the volume was made up to 10 ml by using 9 ml Hexane of HPLC grade. The extracted samples were analysed in GC.

**Bio safety evaluation**

***Studies on the safety of encapsulated pendimethalin on earthworms Eudrilus eugeniae (Kinberg)***

The effect of solvent evaporation encapsulated pendimethalin nano herbicide on earthworm *E. eugeniae* was tested by following the artificial soil test method proposed by Edwards and Bohlen[2]. The culture of *E. eugeniae* was obtained from a vermicompost unit at Central Farm of TNAU, Coimbatore.

Garden soil and FYM (mixed in the ratio (2:1)) were taken in the tubular plastic tubs (12 x 4 cm) and treated with different treatments along with control. Twenty earthworms washed cleanly with water were placed on the top of the substrate. After (every 120 hrs) 5 days, 50 g of FYM was mixed inside the container and the water lost by evaporation was replaced daily. The numbers of live earthworms were counted and the weight of the worms was recorded before release and after experimental period of 30 days. Earthworms were considered dead if they did not respond to a gentle mechanical stimulus.

## RESULTS AND DISCUSSION

The fabricate pendimethalin encapsulated nanoparticles were analyzed by using different instruments and the results are discussed here under.

***Scanning Electron Microscope (SEM) studies***

The surface morphology of nanoencapsulated with pendimethalin herbicide was examined using Scanning Electron Microscope (SEM).

Encapsulated pendimethalin particles prepared by solvent evaporation technique viewed under SEM shows perfect spherical shape (Fig.1). The uniform mono-dispersed nanoparticles obtained from this method were clearly accorded with the nano-sized crystals of loaded herbicide by Wang and Wuled Lenggoro[6]. The surface characteristics of the sphere formation embedded with polymer into nanoencapsules. At this point the solvent ratio and the reaction temperature aided in herbicides loading and its efficiency fluctuate correspondingly[4]. The same method (solvent evaporation) was employed for the preparation of hollow microspheres containing ranitidine hydrochloride using Eudragit RLPO dissolved in a mixture of dichloromethane and ethanol. The maximum yield and drug loading amount of hollow microspheres were 88.45% and 80 ± 4.0%, respectively[5].

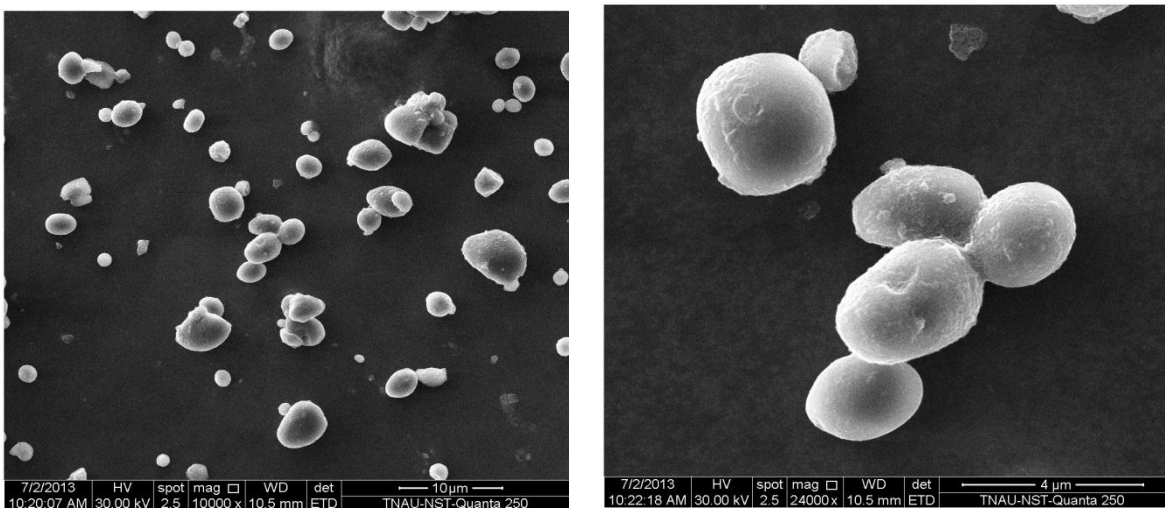


**Fig. 1: SEM images of nanoencapsulated herbicide by solvent evaporation method**

***X-ray diffraction (XRD) studies***

X-ray crystallography is a tool used for identifying the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. By measuring the angles and intensities of these diffracted beams, a crystallographer can produce a three-dimensional picture of the density of electrons within the crystal. From this electron density, the mean positions of the atoms in the crystal can be determined, as well as their chemical bonds, their ailment and various other information. The nanoencapsulated herbicide by solvent evaporation method X-ray diffractogram shows peaks at an angle of (2θ) 28°, 36° and 56° with d spacing values of 3.18, 2.49 and 1.64. The same angle was observed in JCPDS card no. 33-0664, 43-0679 and 05-0664 viz., pendimethalin, PEG and starch, respectively (Fig. 2).

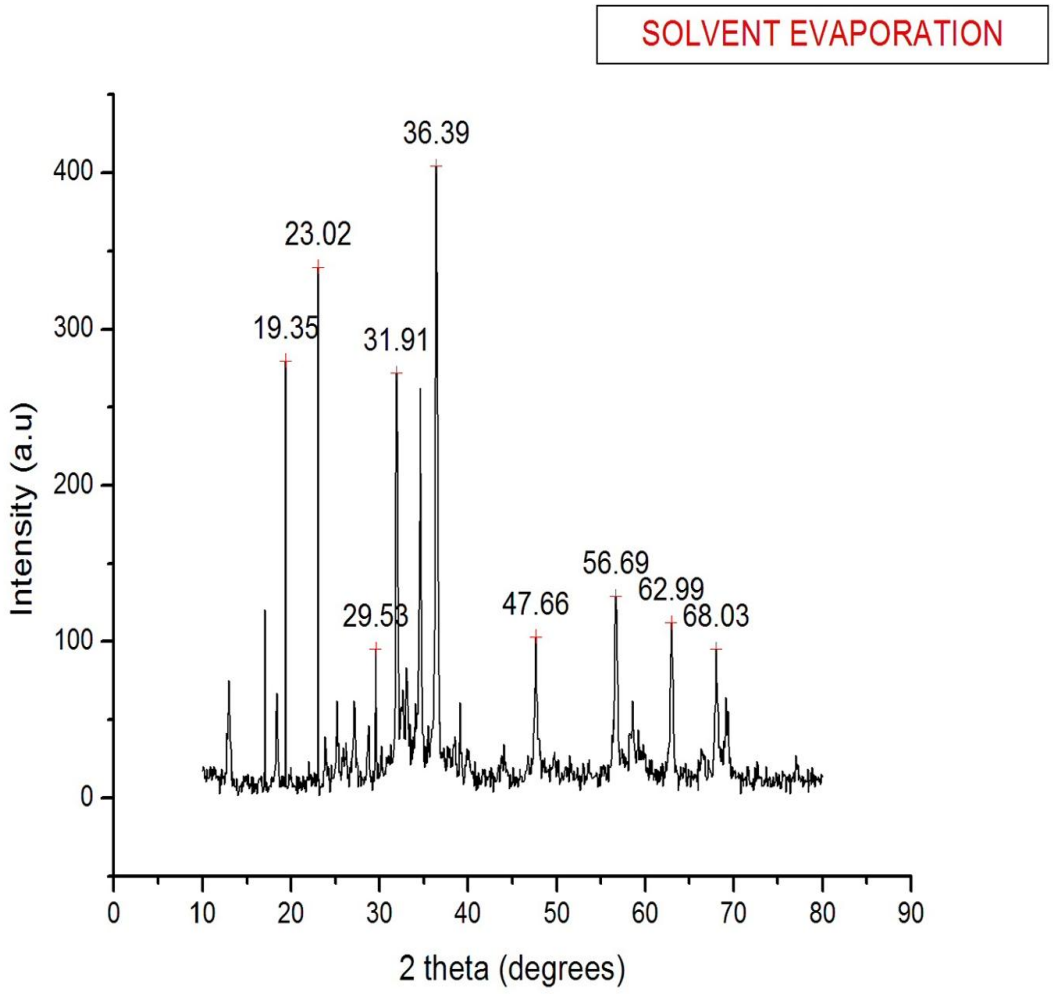


**Fig. 2: XRD- diffractrogram of nanoencapsulated herbicide by solvent evaporation method**

***Particle Size Analyzer studies***

The particle size distribution gives the average minimum and maximum diameter of various particles using laser scattering principle. The particles size varied at different intensity of distributions. The particle size distribution of solvent evaporation methods were 265.8 nm diameters with 100 per cent intensity (Fig. 3). Fabrication of uniform particles is very much important with respect to release of encapsulated active ingredient in a designated period of time.

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**Measurement Results**

| | |
|---|---|
| Date | : Thursday, December 26, 2013 1:02:39 PM |
| Measurement Type | : Particle Size |
| Sample Name | : Solvent evaporation |
| Scattering Angle | : 90 |
| Temperature of the holder | : 25.2 ℃ |
| T% before meas. | : 33963 |
| Viscosity of the dispersion medium | : 0.892 mPa·s |
| Form Of Distribution | : Standard |
| Representation of result | : Scattering Light Intensity |
| Count rate | : 66 kCPS |

**Calculation Results**

| Peak No. | S.P.Area Ratio | Mean | S. D. | Mode |
|---|---|---|---|---|
| 1 | 1.00 | 270.8 nm | 30.0 nm | 265.8 nm |
| 2 | --- | --- nm | --- nm | --- nm |
| 3 | --- | --- nm | --- nm | --- nm |
| Total | 1.00 | 270.8 nm | 30.0 nm | 265.8 nm |

**Cumulant Operations**

| | |
|---|---|
| Z-Average | : 6357.6 nm |
| PI | : 1.837 |

**Molecular weight measurement**

| | |
|---|---|
| Molecular weight | : $7.7 \times 10^{-17}$ kDa |
| Mark-Houwink-Sakurada parameters | : a = $1.000000 \times 10^{0}$ and K = $1.000000 \times 10^{0}$ |

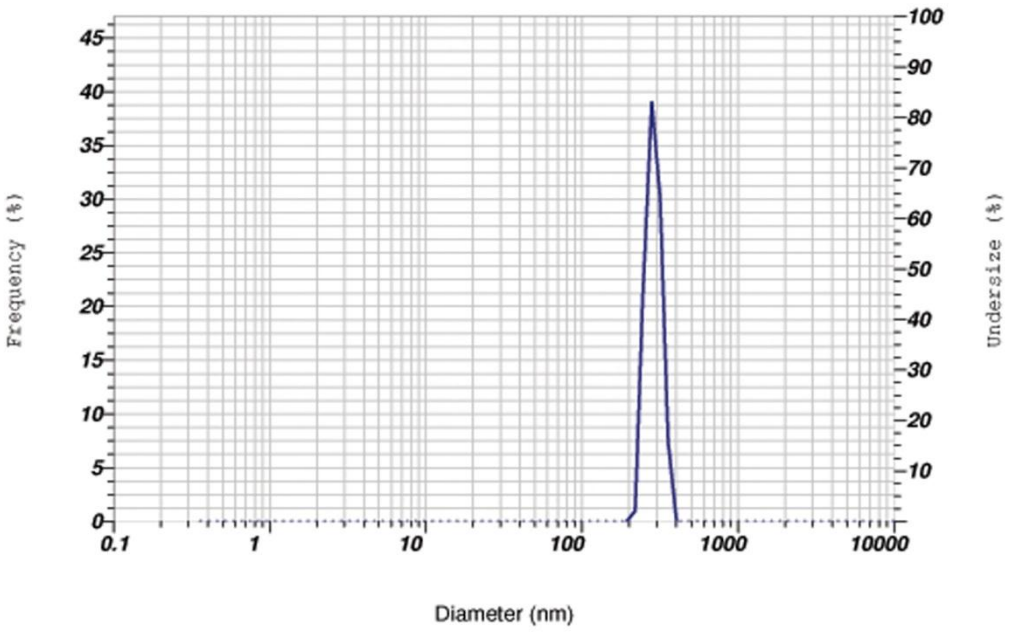


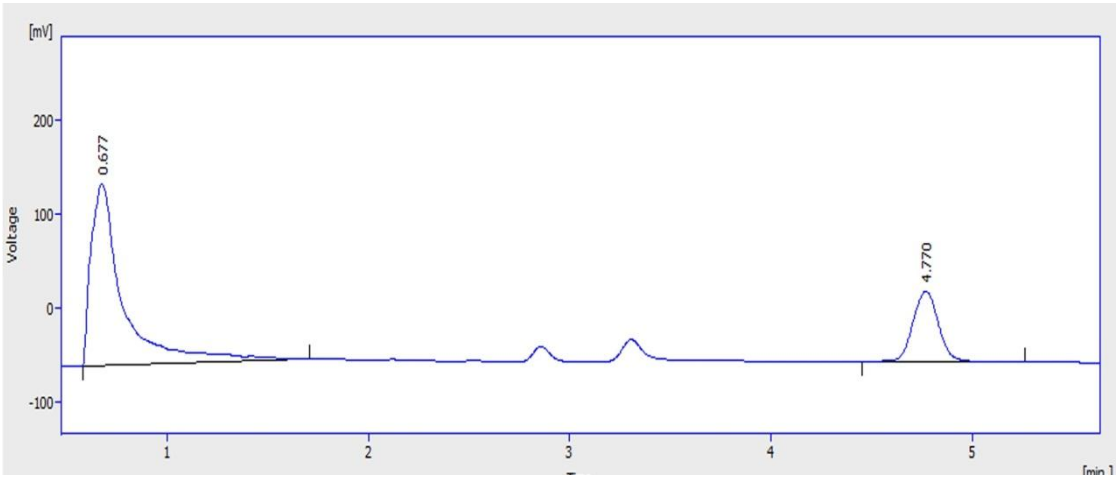

**Fig. 3: Particle size analysis image of nanoencapsulated herbicide by solvent evaporation**

**GC analysis – Encapsulation efficiency**

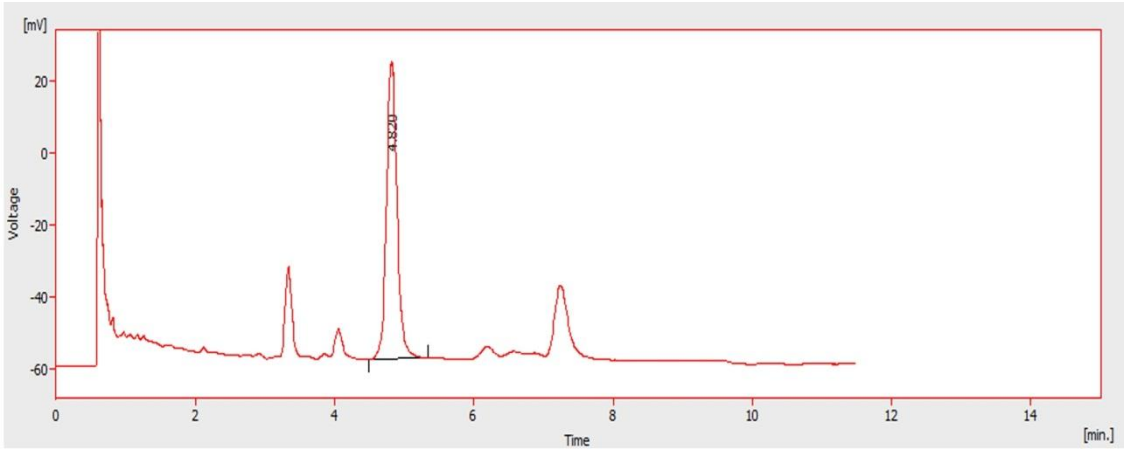
The releasing pattern or efficiency of premeditated nanoencapsulated herbicide from different methods was governed by the outer covering and porous nature of polymer layer. The release is instantaneous in nature, when irrigation water applied to the field the entrapped particles present inside the carrier is being slowly released. Left over herbicide particles retained inside the encapsulated structure will be released in the subsequent wetting sequences. This may be helpful for the checking the germination of weeds in the entire crop growth period. To understand the pattern of herbicide release from the polymer matrices prepared using from different methods was studied under weekly interval basis.

The solvent evaporation method the releasing efficiency was higher upto 10 days of the experiment in all the treatments. After 10 days the releasing efficiency was goes down constantly in 4 and 8 percent starch concentration encapsulated pendimethalin. However 6 per cent starch encapsulated pendimethalin releasing efficiency was persistently increasing in nature with the evidence of surface area (1026.20, 879.00, 1681.95, 2064.65 and 2455.30 mVs) and quantity (1.52, 1.30, 2.50, 3.06 and 3.64 ppm) at 3, 10, 20, 30 and 40 days, respectively. Based on the above results it can be concluded that the nanoencapsulated pendimethalin by solvent evaporation method (3.06 and 3.64 ppm) is one the best method for encapsulation studies (Fig 4).

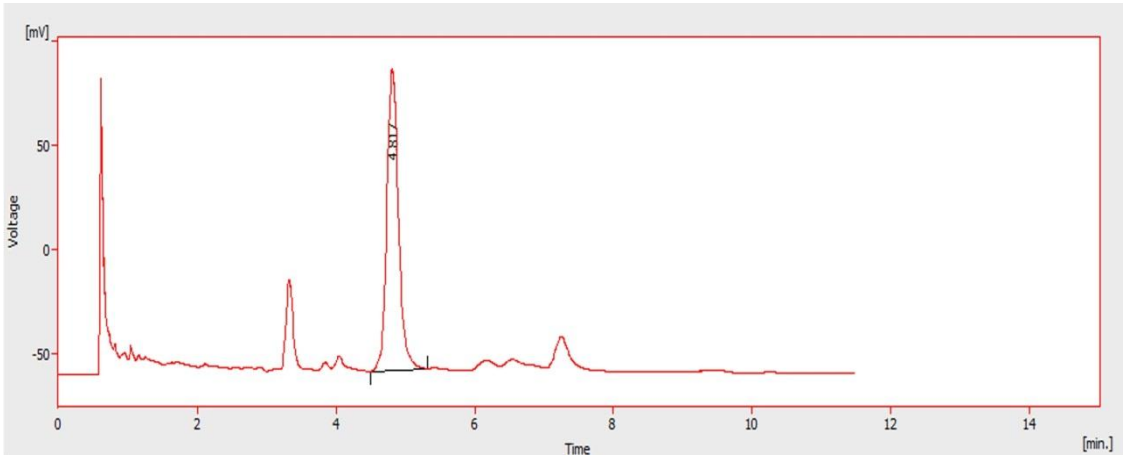
Sunil Kumar[4] reported that the nano encapsulated pendimethalin by solvent evaporation method found to have higher releasing efficiency compared to direct and indirect method of encapsulation.



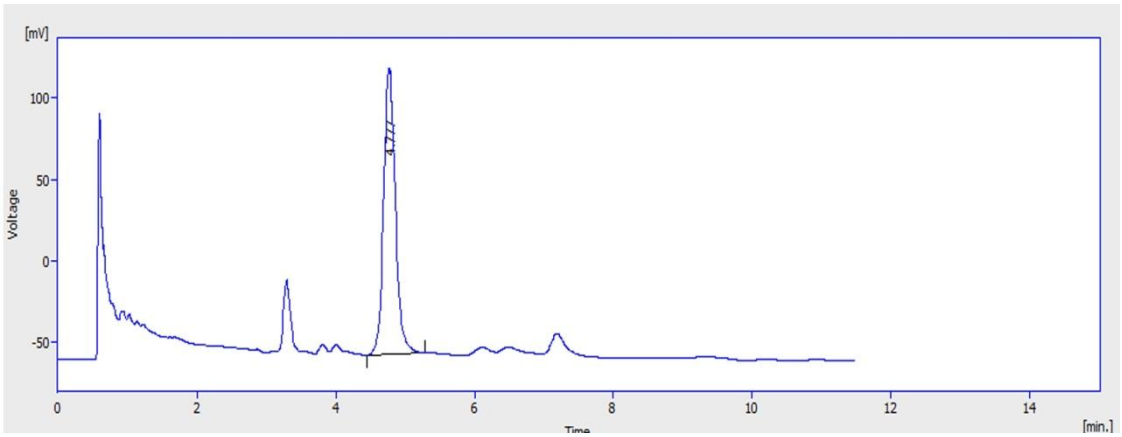

a) 3rd sample



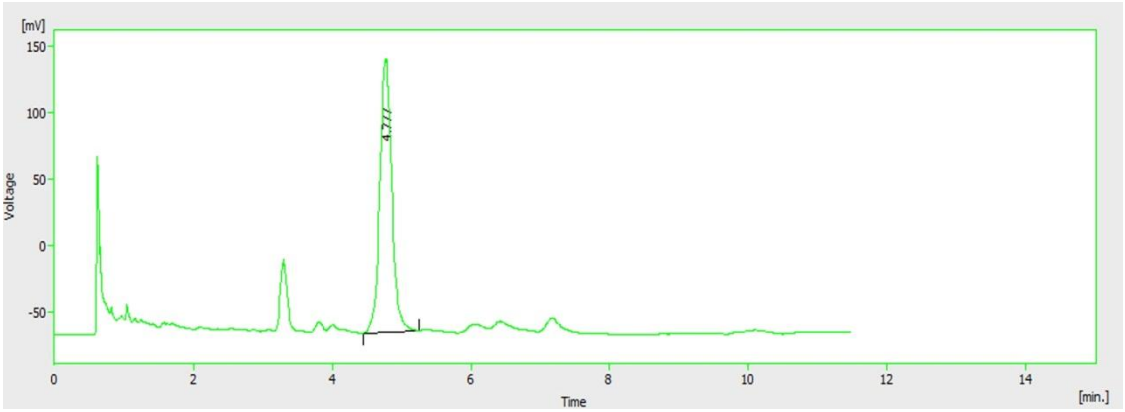

b) 10th day sample




c) 20th day sample




d) 30th day sample




e) 40th day sample

**Fig. 4: GC analysis- Releasing efficiency of solvent evaporation method samples**

***Biosafety studies***

Earthworms in the environment are the clear indicators of environmental pollution. The change or fluctuation in the population after the introduction of any foreign chemical is an indication of toxicity of that compound. Hence, tests were carried out to study the toxicity of nanoencapsulated pendimethalin against natural organisms.

Application nano encapsulated pendimethalin of solvent evaporation methods had no significant effect on the survival rate of earthworm. The present investigation clearly indicates the safety of nanoencapsulated pendimethalin to earthworms as progressive degradation in herbicidal activity themselves to the new environment and started building up. (Plate 1).

**(a). Release of earthworms in to the experimental tubs**


**(b). Toxicity study of nanoencapsulation by solvent evaporation on earthworms**

**(C). Earthworms after 30 days of study**

**Plate 1. Toxicity of nanoencapsulation by solvent evaporation of pendimethalin on earthworms *Eudrilus eugeniae* (Kinberg)**

## CONCLUSIONS

The nano encapsulation was achieved by solvent evaporation technique wherein the organic solvent was utilized for fabrication. In latter stage the organic solvent was evaporated and nanoencapsulated herbicide particles were obtained. The nano structures fabricated using solvent evaporation was found to be longer in releasing the encapsulated herbicide molecules consistently upto the study period of 40 days under controlled environmental condition. This confirms that the herbicide entrapped inside the polymer was well protected from the environmental factors and released in slow manner based on the moisture availability. Biosafety studies of encapsulated materials carried out on earthworms revealed that nanoencapsulated pendimethalin were non-toxic.

## REFERENCES

1. Asthana, A.N., Pulse crops research in India. *Indian J. Agric. Sci*., **68(8):** 448-452 (1998).
2. Edwards, C.A. and Bohlen, P.J., The effects of toxic chemicals on earthworms. In: Review of environmental contamination and toxicity. Ware. G. W (Eds.). Springer- Verlag, New York. pp. 23-99 (1992).
3. Khan, Rahmat Ullah, Rashid, Abdul Khan, and Muhammad Sohail., Impact of various rates of pendimethalin herbicide on weed control, seed yield and economic returns in mungbean under rainfed conditions. *Journal of Agric. Res*., **49(4):** 491-498 (2011).
4. Sunil Kumar, N., Designing nano structures for the controlled delivery of pre-emergence herbicides in rainfed agriculture. M.Sc. (Ag.) Thesis, Tamil Nadu Agric. Univ., Coimbatore (2011).
5. Vandana Singh and Chaudhary, A.K., Preparation of eudragit E-100 microspheres by modified solvent evaporation method. *Acta Poloniae Pharmaceutica-Drug Research*, **68(6):** 975-980 (2011).
6. Wang, W. and Wuled Lenggoro, I., Direct synthesis of barium titanate nanoparticles. *J. Mater. Res*., **20:** 10 (2005).