

Original Research Article

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Evaluation of Diclosulam Residues in Soil at Harvest of Soybean

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HPLC method for the analysis for diclosulam herbicide was standardized using acetonitrile : 0.1% ortho-phosphoric acid (40 : 60) at a flow rate of 1 ml/min at 204 nm wavelength using PDA detector. Recovery of diclosulam from soil were above 70%. Diclosulam was applied at 20 and 26 g/ha as pre-emergence application. Soil samples were extracted and analysed for diclosulam residues by HPLC at zero day and harvest. The residues were found below the detectable limits.

Introduction

Diclosulam is a sulphonamide soil applied herbicide which controls broad-leaved weeds in peanuts, soybean and other crops. It is taken up by roots and foliage and inhibits the acetolactate synthesis. No information is available for its residual activity under Indian condition. Therefore a field experiment of soybean was taken with diclosulam to determine the residues of diclosulam in soil at the harvest of crop.

Materials and Methods

Preparation of standard solutions

10 mg of diclosulam was taken in 10 ml volumetric flask and solution was made with HPLC grade acetonitrile up to the mark to

give 1000 µg/ml solution. From this working standard of 100 µg/ml, 10 µg/ml and 1µg/ml concentration were prepared by serial dilution with acetonitrile.

High performance liquid chromatography (HPLC)

A reverse phase high performance liquid chromatography technique was used for quantitative analysis of diclosulam. A Hewlett Packard HPLC instrument (series 1100) connected with rheodyne injection system and a computer (model vectra) was used for analysis. The stationary phase consisted of lichrosphere on RP-18 packed stainless steel column (250mm × 4mm id). Chromatogram was recorded in a window 95 based HP Chemstation programme.

The chromatographic condition were

1-Mobile phase	Acetonitrile : 0.1% ortho-phosphoric acid (40 : 60)
2-Flow rate	1 ml min ⁻¹
3-Wavelength (λ max)	204 nm
4-Injection volume	20 μ l
5-Column and solvent	Ambient Temperature

Standardization of method of analysis

Suitability of technique for determining pesticide residues quantitatively in substrate require two basic things

- 1- Calibration of the technique.
- 2- Quantitative recovery of the pesticide

Calibration of diclosulam by HPLC

Determination of λ max

The diclosulam standard solution was scanned in different wavelength automatically by HPLC using photodiode array and the wavelength, in which the absorbance was

Substrate	level of fortification (μ g/g)	Replication
Soil	0.1	3
	1.0	3

Sieved and air dried control soil (50g) was taken in two sets each of four Erlenmeyer flask. 3ml of water was added to each flask and the water was mixed thoroughly with the soil by stirring with glass rod to bring the soil roughly to the field capacity.

After that, soil of the three flask from each set was fortified at the required level (different fortification 0.5, 1.0 μ g/g) with the standard solution of diclosulam, it was mixed thoroughly and care was taken so that the entire solution to be added fall on the soil and not on the glass surface of Erlenmeyer flask. One of the flasks from each set was not

maximum, was selected for further studies. The optimum λ max selected was 204 nm to analyze diclosulam.

Recovery experiment

It is most desired that the efficacy of extraction and clean up procedure should be standardized prior to the analysis of actual field or laboratory sample.

Fortification

The soils were fortified with standard solution of different concentration for analysis after the extraction and clean up in order to get reliable data.

fortified and kept as control. To the control flask solvent of equal amount was added.

Extraction

For extraction acidic acetonitrile was used. Extracting solvent was prepared by acidity 1 N HCl to acetonitrile in the ratio of 9 : 1 (CH₃CN : 1 N HCL). After, the 150 ml of solvent was added to each of the flask and shaken on the horizontal shaker for 30 minutes. The content of flask was allowed to settle and the supernatant phase was filtered through Buchner funnel using water pump. The filtration was done in such way that no or very little soil could move to the funnel.

The extraction was done twice more with the same solvent (70 + 50 ml) and filtered in the same way. The combined filtrate was then concentrated by evaporating the solvent on the rotary vacuum evaporator to dryness. The residue was dissolved in 10 ml acetonitrile and transferred to test tube for HPLC analysis.

HPLC Analysis

Sample were analysed by HPLC using acetonitrile : 0.1% ortho-phosphoric acid (40 : 60) at flow rate of 1 ml min^{-1} at λ max 204 nm.

Residue analysis of diclosulam in field soil

Sampling

Samples were collected at zero day and harvest of soybean crop, Soil sample from each replicated plot were taken with the help of auger. The soil from 0-15 cm depth was collected from 5-6 points in a plot and pooled together. The soil was air dried and sieved to separate the pebbles. A representative sample of 50 g soil was taken for residue analysis.

Extraction

Extraction of soil was carried out as described in section 2.3.2.2. The extraction was done in replicate along with control soil.

HPLC Analysis

The sample extract were analysed by HPLC as described in 2.3.2.3.

Results and Discussion

High performance liquid chromatography (HPLC)

Diclosulam when injected in to HPLC it could be resolved into a sharp peak at 10.68 min at

λ max 204 nm (Fig. 9). Mobile phase best suited for separation was acetonitrile : 0.1% ortho-phosphoric acid water in the ratio of (40 : 60) at flow rate of 1ml/min.

Standard curve for diclosulam in HPLC

Different concentration of diclosulam was injected into HPLC and calibration curve were drawn based on concentration versus peak area. The standard curve was linear for Diclosulam. Linearity was observed from 0.1 to 20 $\mu\text{g/ml}$ in case of diclosulam.

Recovery experiment from soil

Recovery of diclosulam was performed at two levels (0.5 and 1ppm) from soil. Extraction of diclosulam fortified soil samples with acetonitrile : 1 N HCL (9 : 1) gave more than 70% recovery at both the fortification levels.

Residue analysis of diclosulam in soil

The soil samples at zero day and harvest were analysed for diclosulam residues. At zero day the initial deposit of diclosulam was found to be 0.117 and 0.158 $\mu\text{g/g}$ at 20 and 26 g a.i./ha rate of application respectively. In harvested soil no residues of diclosulam could be detected at both rate of applications. Kewat *et al.* (2001) observed nearly 27-28% residues of pendimethalin remained in the soil after 45 days of its application and ascertained safety to succeeding wheat crop, as the pendimethalin residues reached non-detectable level after the harvest of soybean. Post-emergence application of chlorimuron in soybean reported no adverse effect on succeeding wheat and gram but affected Indian mustard. Clomazone has low to medium mobility in soil, but remain upto 34-40 days in water. In soil clomazone degrades slowly having half-life ranging from 90-276 days under aerobic and 60 days under anaerobic condition.

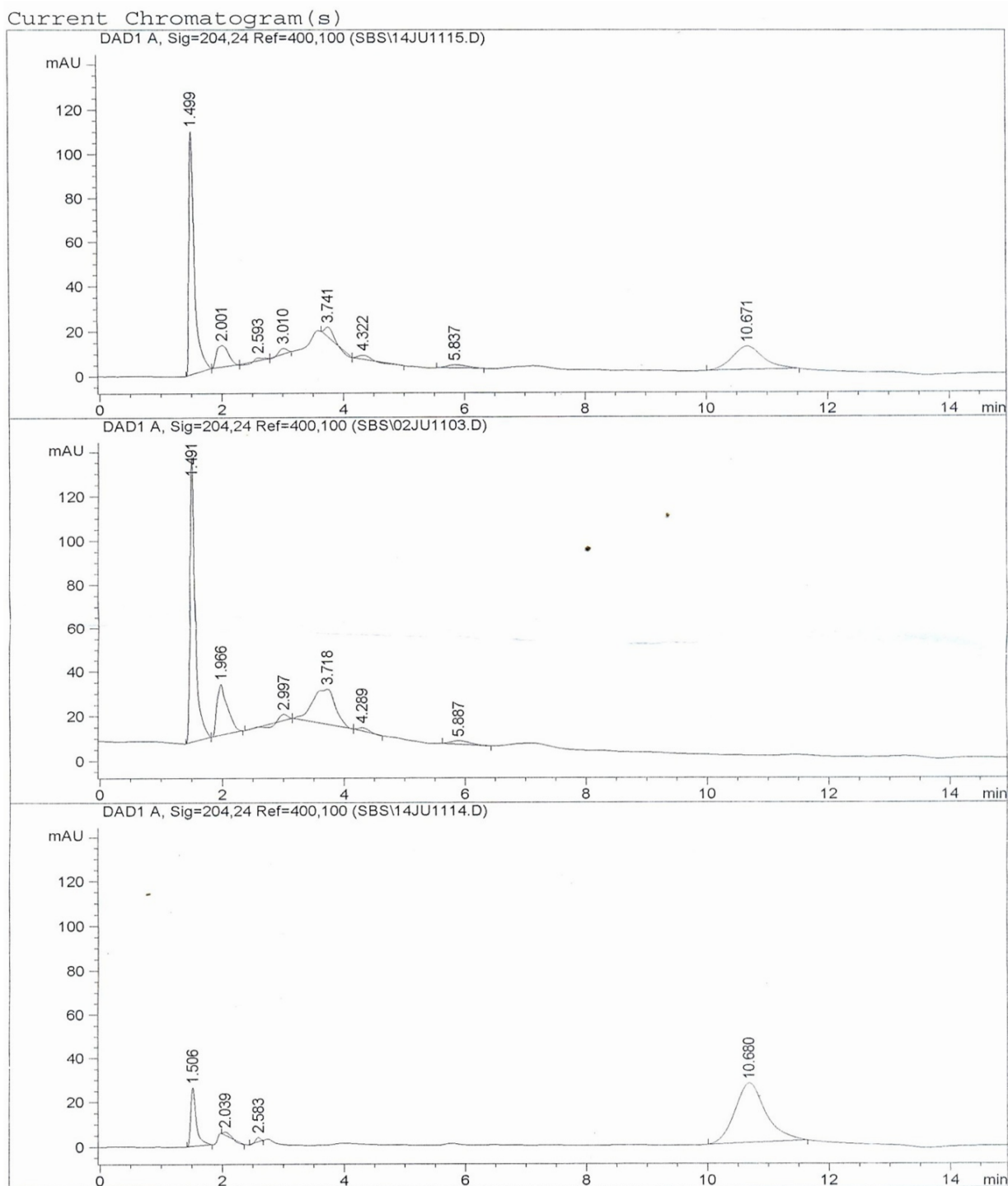


Fig.1 HPLC chromatogram of diclosulam herbicide

In aquatic field condition clomazone had a half-life of 5 days in paddy water and 38 days in paddy sediment (Anonymous, 2003).

In conclusion, field studies were conducted with soybean crop and diclosulam was applied at recommended and at higher rate of application. During the period of field experiment maximum temperature was 32.5 °C and relative humidity was 80.5%.

The residues of diclosulam were completely lost from harvest soil at both the rate of application as they were below detectable

limits. Limit of diclosulam of linearity was 0.1 µg/ml.

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