

Determination of Terminal Residue of Clodinafop propargyl in Soil, Wheat Grains and Straw

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Clodinafop-propargyl, a post-emergence herbicide, is being used largely to control weeds in wheat in India (Pool *et al.*, 1995; Brar *et al.*, 1999). Clodinafop-propargyl inhibits the acetyl co-enzyme A carboxylase (ACCase), which is essential for the production of lipids (fatty acids) needed for plant growth. Herbicide residue estimation in soil and edible plant parts is very useful to determine the duration of herbicide activity in soil and its effect on the following crops and to analyze the quality of the food and feed. The information on the extent of residues of this herbicide in wheat crop and soil is very much limited, hence, the present study was conducted.

The field experiment was conducted at the Research Farm of National Research Centre for Weed Science, Jabalpur, India during 2004-05 in **rabi** season in a randomized block design with three replications. Treatments consisted of three doses of clodinafop-propargyl (60, 120 and 240 g a. i. ha⁻¹) and a weed-free check. Cultivar GW-273 was sown in rows 22.5 cm apart on November 25, 2004. Treatments were applied 30 days after sowing (DAS). The herbicide was applied in 500 l water ha⁻¹ using flat fan nozzle. The crop was raised under irrigated condition with recommended package of practices.

Residue analysis was conducted in residue laboratory of National Research Centre for Weed Science, Jabalpur. The soil was clay loam in texture (clay 35.47%, silt 12.45% and sand 52.09%), low in available nitrogen (300 kg ha⁻¹), medium in available phosphorus (40 kg ha⁻¹) and high in available potassium (300 kg ha⁻¹) with organic carbon 0.80%, EC 0.35 mmhos cm⁻¹ and pH 7.2.

The soil samples from the clodinafop-propargyl treated plots and weed-free plots were collected at harvest (95 days after herbicide application) from a depth of 0-20 cm and used for residue studies. Five

soil cores were randomly taken from each treated and untreated plots avoiding the outer 20 cm fringes of the plots using a soil auger upto a depth of 20 cm. The cores (0-20 cm depth) were bulked together from each plot, air-dried, powdered and passed through a 2 mm sieve to achieve uniform mixing. Pebbles and other unwanted materials were removed manually.

Extraction of Clodinafop-propargyl

The herbicide was extracted as described by Singh *et al.* (2004). Twenty g representative soil sample was taken in 250 ml Erlenmeyer flask, ammonia (1-2 drops) was added, mixed properly and kept at room temperature. After 1 h, 100 ml of ethyl acetate was added and extracted twice by shaking on a horizontal shaker for 30 min and filtered through Buchner funnel using water pump. The combined extracts were concentrated on a rotary vacuum evaporator to about 2 ml. The residue was dissolved in 50 ml of 0.1 N KOH (aqueous) and the contents of the flasks were heated at 60°C on a water bath for half an hour. After cooling, the mixture was neutralized (pH 7) by addition of dilute (1N) HCl. The neutralized mixture was diluted with water (100 ml) and transferred to a 250 ml separatory funnel. The aqueous solution was partitioned with ethyl acetate (3 x 50 ml). The organic layer was dried on anhydrous Na₂SO₄ and the solvent evaporated to dryness on rotary evaporator. Final residue was dissolved in acetonitrile (1 ml) for HPLC analysis.

Grains and Straw

Representative 50 g samples of each grains and straw were taken for quantitative analysis and extracted with acetone using Soxhlet apparatus for 4 h. The concentrated extracts were processed similar

to soil samples. Final residue was dissolved in acetonitrile for HPLC analysis. All the samples were reconstituted in 2 ml acetonitrile for HPLC analysis.

Analytical Technique

Clodinafop-propargyl reference analytical standard of purity was obtained from ACCU standard, USA. All the other chemicals and solvents used in the study were analytical grade reagent and all the solvents were glass distilled prior to use. The extracted clodinafop-propargyl from soil, wheat grain and straw was analysed by a Shimadzu HPLC with Photo Diode Array Detector (PDA). AC-18 column (ODS) (250 mm x 4.6 mm i. d.) was used. The mobile phase used was acetonitrile : water (70 : 30) with flow rate 1 ml/min. 20 µl sample was injected. For the detection of clodinafop 215 nm wavelength was used. All the samples were filtered through 0.20 µm membrane filter 20-µl aliquots of sample extracts were injected in HPLC column alongwith standard solution. The retention time and peak area of the samples and standard were recorded and clodinafop in the samples was quantified. At these conditions, the retention time of clodinafop was found to be 2.15 min.

Clodinafop residues were not detected in the soil, grain and straw at doses 60 and 120 g ha⁻¹;

Table 1. Residues of clodinafop-propargyl in different matrices at harvest

Dose (g ha ⁻¹)	Residues (ppm)		
	Soil	Grains	Straw
60	ND	ND	ND
120	ND	ND	ND
240	ND	0.0089	ND

ND–Not detected.

however, 0.0089 ppm residues were detected in wheat grains at 240 g ha⁻¹ treatment (Table 1). Residue level, determined in wheat grains, did not exceed permissible level recommended by FAO/WHO (0.1 and 0.5 ppm for wheat grains and straw, respectively). This might be due to the fact that clodinafop at 60 and 120 g ha⁻¹ rates in wheat crop dissipated completely by the harvest. Singh *et al.* (2004) also found the similar results.

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