

## Persistence of pendimethalin and oxyfluorfen at different temperature and moisture levels in an alfisol and vertisol

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### ABSTRACT

Persistence of pendimethalin and oxyfluorfen in an alfisol and vertisol was studied at three moisture levels (at saturation, Field Capacity and 50 % Field Capacity) and at two temperature levels ( $10 \pm 2^{\circ}\text{C}$  and  $27 \pm 2^{\circ}\text{C}$ ) for 137 days in the laboratory. Degradation of pendimethalin and oxyfluorfen was more rapid at high temperature and moisture levels. The disappearance curve / semi logarithmic plot followed first order kinetics with two distinct pathways, an initial faster rate followed by a slower and more gradual disappearance. Half life values for pendimethalin varied from 35.45 to 108.28 in alfisol and from 28.28 to 91.84 in vertisol ; for oxyfluorfen half life values varied from 97.60 to 192.50 in Alfisols and 90.05 to 147.44 in vertisols.

**Key word :** Half life, herbicide degradation rate, Residue of herbicides.

Persistence is an important characteristic of herbicide affecting efficacy and carryover to subsequent crops. The period during which the pesticide remained intact and biologically active has great practical importance in determining how well it performs its intended task in soil. It is also called as time of residence of chemical in soil and is measured in terms of half life. The persistence of herbicide depends on number factors like edaphic and climatic conditions as well as properties of chemical itself. The persistence of soil applied herbicide is of concern during the period in which weed control is required and also with regard to potential damage to succeeding susceptible crops. The effects of soil temperature and moisture on herbicide degradation are straight forward and the microbial and chemical degradation are enhanced with increased temperature and moisture. Degradation of many herbicides follows first order kinetics, meaning that the rate of degradation is proportional to the herbicide concentration (Brar *et al.* 2006). Pendimethalin and oxyfluorfen were widely used in vegetables as pre emergence for control of weeds. As there is information on persistence of these herbicides in soils of Andhra Pradesh was meager the experiment was conducted to study the persistence of pendimethalin and oxyfluorfen at different temperatures and moisture levels.

### MATERIALS AND METHODS

An Alfisol and Vertisol were selected for studying the persistence of pendimethalin and oxyfluorfen. For vertisol, the values of water holding capacity at saturation, field capacity and 50 % field capacity were 36 %, 20 % and 10% respectively. Whereas, for alfisols water holding capacity values at saturation, field capacity and 50% field

capacity were 28%, 16% and 8%, respectively as measured by using pressure plate apparatus.

A known quantity of 2 mm sieved soil samples approx. 350 gm. were spread on paper and treated with pendimethalin. Pendimethalin was diluted in methanol in order to get a concentration of 10 g/ mL. This was sprayed on the soil sample and then methanol was allowed to evaporate. Ten gram of herbicide treated soil samples were put in to polythene bags and the polythene bags were sealed after adding predetermined quantities of water to bring the moisture levels to saturation, field capacity and 50% field capacity. Duplicate soil blanks without the herbicide were maintained simultaneously.

The samples after moisture treatment were incubated at  $27 \pm 2^{\circ}\text{C}$  and  $10 \pm 2^{\circ}\text{C}$  in BOD and the soil samples were analyzed at 0, 3, 7, 15, 30, 45, 60, 90, 105 and 135 days intervals for the amount of herbicide retained in the soil solution. For this soil sample collected at each interval was extracted with methanol to measure the quantity of herbicide extracted. The individual soil samples at each interval was transferred to 60 mL capacity screw capped test tubes and shaken with two 20 mL portions of methanol for 30 minutes on mechanical shaker and filtered through Whatman No 1 filter paper. The final volume of extract was made up to 50 ml and the absorbance was measured at wavelength of 430 nm for pendimethalin. The concentration of pendimethalin and in methanol were determined with reference to the standard curves after correcting the soil blanks.

Similar procedure was followed for determining the persistence of oxyfluorfen . The absorbance was measured at a wave length of 213 nm for oxyfluorfen.



## RESULTS AND DISCUSSION

**Table 1. Physico-chemical properties of soils**

| Physico-chemical character | Alfisol    | Vertisol  |
|----------------------------|------------|-----------|
| pH                         | 7.71       | 7.82      |
| EC (dS/m)                  | 0.24       | 0.22      |
| Organic carbon (g/kg)      | 3.10       | 8.50      |
| Sand                       | 74.40      | 55.80     |
| Silt                       | 13.00      | 11.00     |
| Clay                       | 12.60      | 33.20     |
| Texture                    | Sandy loam | Clay loam |

The quantity of herbicide extracted was plotted against time for the different experimental conditions and these were depicted in Fig. 2 and 3 for pendimethalin and 4 and 5 for oxyfluorfen. The data was plotted as residual concentrations on a logerthemic scale, against time of incubation for each treatment. The disappearance curve consists of two distinct pathways, an initial faster rate followed by a slower and more gradual disappearance. The semi logerthemic plot of the data indicated that pendimethalin followed first order kinetics. The first order kinetic plot confirmed two distinct rates of disappearance. The faster mode of disappearance continued only till 45 to 60 days where as slower rate of disappearance continued till the end of study. A wide difference dissipation rate of both pendimethalin and oxyfluorfen was observed in both soils used in the study. The differences may be attributed to difference in herbicide availability in different soils.

A complete analysis of the persistence data was carried out for resolving the constants  $k$ ,  $k_1$ ,  $k_{-1}$  according to the method given by Hamaker and Goring (1976) and presented in Table. 2 and 3 for pendimethalin and oxyfluorfen, respectively.

In present investigation for pendimethalin and oxyfluorfen, the rate of re-entry into labile pool was considered negligible. The rate of degradation of pendimethalin and oxyfluorfen increased with increase in temperature from 10 °C to 27 °C and followed order  $k_{-1} < k_1 < k$ . The rate of entry into bound pool was higher for vertisol than for alfisol and was higher at higher temperature. It decreased with soil moisture in the order: saturation > field capacity > 50% field capacity. Behaviour of pendimethalin in Indian soils under flooded conditions has been reported by several workers (Arvind *et al.* 2000 Helling and Krivonak 1978, Kennedy and Talbert 1977). Their studies have shown that degradation of pendimethalin occurred more rapidly in anaerobic soils than in aerobic soils. They concluded that flooding of soils resulted in rapid dissipation of pendimethalin as compared to soils maintained at field capacity. The constant for rate of entry in to bound pool was higher for vertisol as compared to alfisol. This can be attributed to the fact that the clay content and organic matter content of vertisol was higher than alfisol and there by herbicides may be adsorbed more strongly on these sites in soil and can not be desorbed by organic solvents. From the rate constants, it can be seen that there was higher rate of degradation of herbicides at 27°C than at 10°C.

The half life values of pendimethalin at two temperature and three moisture levels are presented in Table 4. Half life values were higher at 50% field capacity than at saturation and at higher temperature. Arvind *et al.*(2000) reported that the half life values of pendimethalin under flooded condition were in the range of 23.6 41.8 days and under unflooded conditions (60% w/w WHC) the half life values ranged from 65.4 136 days. Singh *et al.* (1994) reported persistence of pendimethalin even up to 240 days in sandy loam soil. Pendimethalin has strong binding with soil colloids, because of which only little amount is lost to sub soil by leaching. Also strong adsorption on soil particles renders the chemical less

**Table 2. Specific reaction rates /day for different steps during dissipation of oxyfluorfen in alfisol (A<sub>4</sub>) and vertisol (V<sub>2</sub>) at different temperatures and moisture levels**

| Soil           | 27 ± 2°C   |        |        | 10 ± 2°C   |          |        |
|----------------|------------|--------|--------|------------|----------|--------|
|                | Saturation | FC     | 50% FC | Saturation | FC       | 50% FC |
| A <sub>4</sub> |            |        |        |            |          |        |
| k              | 0.0071     | 0.0063 | 0.0061 | 0.0046     | 0.0042   | 0.0036 |
| k <sub>1</sub> | 0.0043     | 0.0038 | 0.0036 | 0.0028     | 0.0024   | 0.0021 |
| k-1            | 0.0022     | 0.0013 | 0.0008 | 0.0018     | 0.0011   | 0.0006 |
| V <sub>2</sub> |            |        |        |            |          |        |
|                |            | 27±2°C |        |            | 10 ± 2°C |        |
| k              | 0.0077     | 0.0073 | 0.0065 | 0.0056     | 0.0049   | 0.0047 |
| k <sub>1</sub> | 0.0068     | 0.0064 | 0.0064 | 0.0051     | 0.0046   | 0.0041 |
| k-1            | 0.0056     | 0.0051 | 0.0046 | 0.0045     | 0.0038   | 0.0033 |

**Table 3. Specific reaction rates/day for different steps during dissipation of pendimethalin in Alfisol (A<sub>4</sub>) and Vertisol (V<sub>2</sub>) at different temperatures and moisture levels**

| Soil            |            | 27 ± 2 <sup>o</sup> C |        |                       | 10 ± 2 <sup>o</sup> C |        |  |
|-----------------|------------|-----------------------|--------|-----------------------|-----------------------|--------|--|
| A <sub>4</sub>  | Saturation | FC                    | 50% FC | Saturation            | FC                    | 50% FC |  |
| k               | 0.0198     | 0.0105                | 0.0076 | 0.0140                | 0.0075                | 0.0064 |  |
| k <sub>1</sub>  | 0.0186     | 0.0098                | 0.0072 | 0.0134                | 0.0072                | 0.0061 |  |
| k <sub>-1</sub> | 0.0112     | 0.0086                | 0.0058 | 0.0112                | 0.0045                | 0.0038 |  |
| v <sub>2</sub>  |            | 27 ± 2 <sup>o</sup> C |        | 10 ± 2 <sup>o</sup> C |                       |        |  |
| k               | 0.0244     | 0.0129                | 0.0087 | 0.0159                | 0.0080                | 0.0076 |  |
| k <sub>1</sub>  | 0.0234     | 0.0119                | 0.0078 | 0.0148                | 0.0076                | 0.0071 |  |
| k-1             | 0.0166     | 0.0101                | 0.0038 | 0.0112                | 0.0058                | 0.0034 |  |

**Table 4. Half life values for pendimethalin oxyfluorfen in vertisol and alfisol at different temperatures and moisture levels**

| Soil           | Saturation | 27±2 <sup>o</sup> C<br>FC | 50% FC | Saturation | 10±2 <sup>o</sup> C<br>FC | 50% FC |
|----------------|------------|---------------------------|--------|------------|---------------------------|--------|
| Pendimethalin  |            |                           |        |            |                           |        |
| A <sub>4</sub> | 35.45      | 66.95                     | 90.97  | 49.55      | 92.40                     | 108.28 |
| V <sub>2</sub> | 28.40      | 53.72                     | 79.65  | 43.58      | 86.62                     | 91.84  |
| Soil           | Saturation | 27±2 <sup>o</sup> C<br>FC | 50% FC | Saturation | 10±2 <sup>o</sup> C<br>FC | 50% FC |
| Oxyfluorfen    |            |                           |        |            |                           |        |
| A <sub>4</sub> | 97.60      | 110.0                     | 113.60 | 150.62     | 165.00                    | 192.50 |
| V <sub>2</sub> | 90.05      | 94.93                     | 106.61 | 123.75     | 141.42                    | 147.44 |

prone to degradation by physical as well as biochemical actions (Kaplana *et al.* 1999). The half life of oxyfluorfen at temperature and moisture levels in the present investigation was observed to be in the range of 90.05 to 192.50 days, which was similar to the findings of Yen *et al.* (2003) and Scrano *et al.* (2004). Oxyfluorfen has a strong tendency to adsorb on to the soil particles and is nearly insoluble in water. The results indicated a decrease in half life values with increase in temperature. Brar *et al.* (2006) reported that the half life of sulfosulfuron decreased with increase in temperature. Similarly Punia *et al.* (1996) and Amarjeet *et al.* (2004) also reported a decreased half life of chlorosulfuron with increase in temperature.

Higher activation energy was obtained in alfisol as

compared to vertisol (Table 5). Similar trend was observed for atrazine by Shalali (1986), for metoxuron by Raman and Rao (1984) and atrazine and isoproturon by Nagamadhuri (2003). However, these values were lower than those reported by Walker and Zimdahl (1981). It may be pointed out here that they did not consider the existence of two rates of disappearance, but fitted the whole data to first order kinetics. This may be the reason for higher predicted levels of residues, in comparison with the field levels.

The temperature and moisture content of the soil had significant effect on persistence. An increase in temperature and moisture increased the loss of herbicide from soil. The order of disappearance of pendimethalin

**Table 5. Rate constants (/day) and activation energy (KJ/mole/day) at different temperature and moisture levels for pendimethalin and oxyfluorfen**

| Soil                       | Pendimethalin       |        |        |                     |        |        | Activation energy (KJ/mole/day) |       |        |
|----------------------------|---------------------|--------|--------|---------------------|--------|--------|---------------------------------|-------|--------|
|                            | 27±2 <sup>o</sup> C |        |        | 10±2 <sup>o</sup> C |        |        | Sat                             | FC    | 50% FC |
|                            | Sat                 | FC     | 50% FC | Sat                 | FC     | 50% FC |                                 |       |        |
| Alfisol (A <sub>4</sub> )  | 0.0198              | 0.0105 | 0.0076 | 0.0140              | 0.0075 | 0.0064 | 24.55                           | 27.39 | 7.75   |
| Vertisol (V <sub>2</sub> ) | 0.0244              | 0.0129 | 0.0087 | 0.0159              | 0.008  | 0.0076 | 19.80                           | 19.29 | 9.85   |
|                            |                     |        |        | Oxyfluorfen         |        |        |                                 |       |        |
| Alfisol (A <sub>4</sub> )  | 0.0071              | 0.0063 | 0.0061 | 0.0046              | 0.0042 | 0.0036 | 24.88                           | 23.24 | 30.23  |
| Vertisol (V <sub>2</sub> ) | 0.0077              | 0.0073 | 0.0065 | 0.0056              | 0.0049 | 0.0047 | 18.26                           | 22.85 | 18.59  |

and oxyfluorfen at three moisture levels was saturation > field capacity > 50% field capacity.

The rate of disappearance in two soils was compared and it indicated the rate of disappearance was inversely related to organic carbon content of the soil. Lower persistence of both pendimethalin and oxyfluorfen was observed in alfisol, having low organic matter and low clay content in comparison to vertisol, which showed maximum persistence indicating that the soil organic matter and clay content are the most important factors in determining the persistence of soil applied herbicides. These observations corroborate the findings of Arvind *et al.* (2000) and Eleftherohorinos (1989).

The results of this study are significant as they clearly brought qualitatively and quantitatively, the content of herbicides being partitioned into bound pool and labile pool. Although the concentration in labile pool has all long been considered more important in bioactivity of chemical, certain investigations have focused on the importance of bound pool as well. Barriuso *et al.* (1991) observed 15-40 per cent in bound form after 6 months of incubation in three French soils and showed that bound residues were clearly related to the organic fraction of the soil. Greater quantities of bound residues in soil with high organic matter could be expected as pesticide can be retained by adsorption and entrapped in the organic matter (Khan 1980). The amount of residue, in the form of atrazine or its metabolites increases with atrazine soil reaction time (Sorenson *et al.* 1993 and Topp *et al.* 1994).

In the end, it is relevant to point out that Walker's model for predicting the persistence of pesticides did not take into consideration, the possibility of the pesticide applied to soil being retained in the bound fraction. The model suggested by him assumed a straight forward one phase kinetics for disappearance of pesticides involving first order kinetics. This is certainly a lacuna and a correction for partitioning into bound pool is necessary, as there is overwhelming evidence now that this happens in a large number of cases. Thus, it is possible that the reasons for obtained higher values for pesticide residues through predictive modeling equations by Walker and his co-workers was actually due to this factor. Thus, a two phase kinetic pattern of disappearance of soil applied chemicals was observed by Katan *et al.* (1976) for the disappearance of <sup>14</sup>C-parathion, Liechtenstein *et al.* (1977) for <sup>14</sup>C-parathion, fonofos, and Raman (1987) for degradation of atrazine.

#### **Comparison between field and lab persistence of herbicides**

As discussed earlier, the computer model of simulation of herbicide persistence of herbicide as

described by Walker (1974) and revised by Walker and Barnes (1981) has been shown to predict the persistence of herbicides in the field. Hence, the rate of degradation has been established in the same soil in the laboratory. However these studies were made in England, hence this model was evaluated in field conditions using the field persistence data for pendimethalin and oxyfluorfen. For this a field experiment was conducted in College Farm, Agricultural college, Rajendranagar during Rabi, 2006-07 and summer, 2007 and the soil samples were analyzed at fifteen days interval.

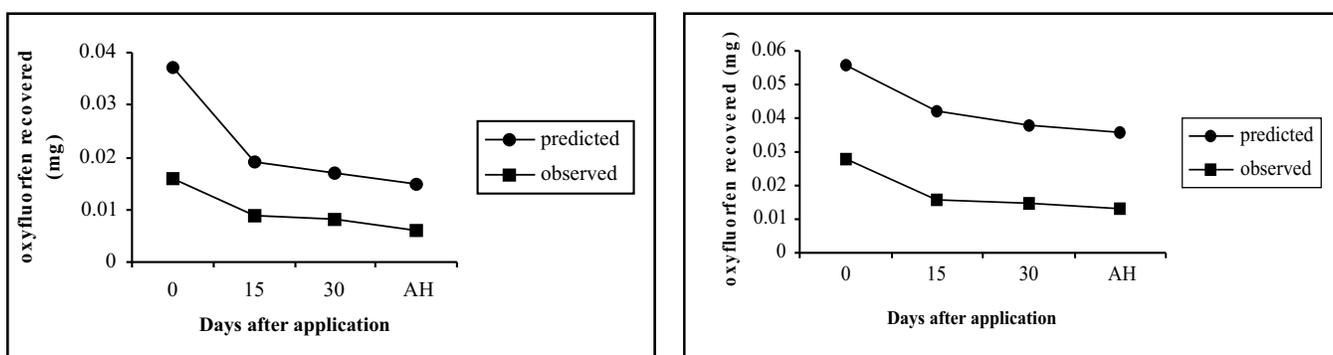
The values of persistence of pendimethalin in the laboratory varied from 0 to 45 days was 0.216 to 0.155 µg/g and 0.331 to 0.183 µg/g at 0.5 and 0.75 kg/ha and for oxyfluorfen the values ranged from 0.037 to 0.015 µg/g and 0.056 to 0.028 µg/g at 0.1 and 0.15 kg/ha at 0 to 45 days respectively. Persistence decreased progressively from 0 DAA to 45 DAA as indicated in Table 6. Under field conditions the values for pendimethalin ranged from 0.136 to 0.058 µg/g and 0.202 to 0.078 µg/g at 0.5 and 0.75 kg/ha and for oxyfluorfen the values ranged from 0.021 to 0.007 µg/g and 0.034 to 0.011 µg/g at 0.1 and 0.15 kg/ha at 0 to 45 days, respectively. The variation of pendimethalin and oxyfluorfen persistence was more than 30 per cent between the laboratory and field studies (Table 6; Fig. 6 and 7).

It has been suggested that if the model could predict the residues within 30 percent of the difference between the field and lab studies, This could be sufficiently accurate for the practical purpose of forecasting aspects under field conditions. (Walker and Bond 1978). Although in some circumstances the model may provide accurate prediction of residues in the field but in the present case, it underestimates rates of loss leading to over prediction of residues it could be possible that these over predictions occur because the loss processes such as photo-chemical degradation, volatilization and movement in soil layers below the sampling depths are not allowed for in this simulation model. Further the variable soil conditions such as alternate wetting and drying (as a result of irrigating the field) and their effect on temperature and moisture variations have stimulating effects on microbial activity that are not apparent under laboratory incubated conditions. These reasons suggest that this model may not accurately describe the rate of pendimethalin and oxyfluorfen dissipation from soil. However a large number of field experiments at different sites will have to be carried out on soils varying in soil properties to arrive at meaningful conclusions for predicting the fate of herbicides in soil. The observed values were much lower as compared to the predicted values. The other reasons could be that there could be losses during application

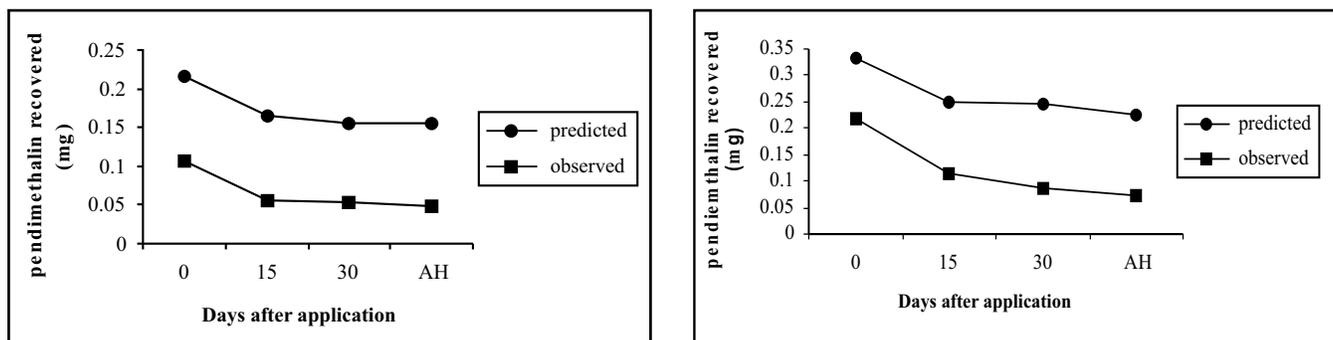
**Table 6. Persistence of pendimethalin and oxyfluorfen in field conditions and lab incubated samples**

| Treatments               | 0 DAA          |                    | 15 DAA         |                   | 30 DAA         |                   | 45 DAA         |                   |
|--------------------------|----------------|--------------------|----------------|-------------------|----------------|-------------------|----------------|-------------------|
|                          | Lab conditions | Field conditions   | Lab conditions | Field Conditions  | Lab conditions | Field conditions  | Lab conditions | Field conditions  |
| Pendimethalin 0.5 kg/ha  | 0.216          | 0.136<br>(37.03 %) | 0.195          | 0.079<br>(59.0 %) | 0.167          | 0.063<br>(62.2 %) | 0.155          | 0.058<br>(62.5 %) |
| Pendimethalin 0.75 kg/ha | 0.331          | 0.202<br>(43.24 %) | 0.278          | 0.112<br>(59.7 %) | 0.247          | 0.11<br>(65.4 %)  | 0.183          | 0.073<br>(60.0 %) |
| Oxyfluorfen 0.1 kg/ha    | 0.037          | 0.021<br>(43.0 %)  | 0.019          | 0.011<br>(42.1%)  | 0.017          | 0.009<br>(47.0%)  | 0.015          | 0.007<br>(53.3%)  |
| Oxyfluorfen 0.15 kg/ha   | 0.056          | 0.034<br>(39.28 %) | 0.048          | 0.022<br>(54.4)   | 0.036          | 0.015<br>(58.0 %) | 0.028          | 0.011<br>(60.7 %) |

Figures in parenthesis indicate percentage variation between Lab and Field persistence



**Fig. 3. (i& ii) : Oxyfluorfen persistence (observed and predicted) at 0.1 kg/ha and 0.15 kg/ha**



**Fig. 4. (i& ii): Pendimethalin persistence (observed and predicted) at 0.5 kg/ha and 0.75 kg/ha**

while spraying and the second loss immediately after application at the time when herbicide remain on the soil surface and the interaction between the herbicide and soil colloids. Hamaker (1975) observed that the losses during the spray drift and adsorption phases could be difficult to model quantitatively because of their complex and dynamic nature whereas the final degradation phase would be simpler to describe in quantitative terms.

The results from the present investigation are consistent with the suggestions of Hamaker (1975), Walker and Zimdahl (1981), Walker and Barnes (1981) and Nagamadhuri (2003), in that model over estimated the residue by large amounts and discrepancies between the predicted and organic matter data was apparent specially during the initial 45 days period suggesting that this model could be inaccurate during the period shortly after

application under field conditions, when the compound is likely to remain at the soil surface. Therefore the model could be inaccurate in predicting the herbicide residues in field conditions.

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