

# Interaction of soil pH and sulfonylurea herbicide degradation on legume growth

A. Farley<sup>1,2</sup>, J.R. Condon<sup>1,2,3</sup>, J.I. McCormick<sup>1,2</sup> and G. Doran<sup>1</sup>.

<sup>1</sup>School of Agricultural and Wine Sciences, Charles Sturt University; <sup>2</sup>Graham Centre of Agricultural Innovation (Charles Sturt University and NSW Department of Primary Industries); <sup>3</sup>NSW Department of Primary Industries, Wagga Wagga Agricultural Institute.

## Keywords

- triasulfuron, chlorsulfuron, lupin, nodulation.

## Take home messages

- Degradation of triasulfuron and chlorsulfuron decreases as soil pH approaches a neutral pH.
- Liming acidic soils can increase lupin growth and nodulation.
- Soils approaching a neutral soil pH due to 'overliming' can reduce lupin growth and nodulation.
- Sulfonylurea (SU) herbicides applied to soils with pH stratification may result in poor legume growth due to the impact of herbicide residue in the surface few centimetres and the soil acidity in subsurface layers.

## Background

Soil acidity is a major limitation to Australian grain production (Li et al. 2019). Liming is used to overcome acidity but in no-till systems adequate incorporation has not occurred. This confines the lime effect to the surface layers, creating pH stratification where the soil surface (0-5cm) exhibits higher pH than the soil immediately below (5-15cm) before the pH increases with depth (>15cm) (Burns and Norton 2018). The presence of this acidic subsurface layer (5-15cm) has been shown to adversely affect plant vigour, root growth and nodulation of legumes (Burns et al. 2017a, 2017b). Legumes are also susceptible to herbicide residues, with sulfonylurea (SU) herbicide (group B) residues being observed to reduce legume growth (Howie and Bell 2005). Soil pH plays an important role in the degradation of SU herbicides with rates of chemical hydrolysis being pH-dependent (Beyer et al. 1988) and microbial breakdown additionally adversely affected if optimal pH requirements are not met (Sarmah et al. 2000a).

Therefore, where pH stratification exists, damage to legumes may be caused by; acidic subsurface layers affecting growth and nodulation, or by a SU herbicide residue which remain due to the relatively high pH in the surface few centimetres, or a combination of these two factors. Experiments were conducted to determine the influence of pH on SU herbicide degradation over a range of soil pHs and to study the interaction of pH and SU herbicide residue on legume growth and nodulation.

## Methods

To quantify the influence of pH on the degradation of SUs, two laboratory incubation experiments were conducted using two SU herbicides and a Red Chromosol surface soil adjusted to a range of soil pHs. Secondly, a glasshouse pot trial was conducted to determine the effect on legume production and nodulation of soil pH and concentration of the same two SU herbicides.



## Soils

Soil from the 0-10cm layer of a sandy loam, Red Chromosol soil was collected from Big Springs (-35.414123°, 147.407347°), south of Wagga Wagga NSW. The site had a history of permanent pasture with periodic superphosphate application but no liming history. Collected soil was passed through a 10mm sieve and dried at 40°C. Soil was then divided into two groups: five quantities of approximately 80kg each for use in the pot trial, and five quantities of approximately 1kg, which was passed through a 2mm sieve, for use in the degradation study. Calcium hydroxide was then added and mixed through both groups of soil at rates of 0, 0.63, 1.42, 2.20 and 2.99 g Ca(OH)<sub>2</sub>/kg soil to create soils exhibiting a pH (CaCl<sub>2</sub>) range of 4.1, 4.8, 5.7, 6.5 and 7.2 respectively, after an initial four week period of incubation. Soil for use in pot trial was weighed (1200g air dried) into plastic bag lined pots (8cm diameter and 18cm length) prior to incubation, while soil for use in degradation study remained in 1kg quantities. During this time soil was watered by weight to 80% of field capacity and allowed to dry to approximately 50% of field capacity before rewetting following the method of Fergus and Stirk (1961).

### Controlled degradation study

Two randomised block incubation experiments with four replicates were conducted simultaneously. Each experiment tested a type of SU herbicide over a range of soil pHs. The soil pH used were those created by adjustment with calcium hydroxide as explained previously within this paper. Two SU herbicides, triasulfuron 750gai/kg (applied as Triasulfuron 750WG) and chlorsulfuron 750gai/kg (applied as Chlorsulfuron 750WG) were applied at equivalent label application rates of 35g/ha and 20g/ha, respectively. This was achieved by adding 0.39mL of either a 99ng triasulfuron /mL or 57ng chlorsulfuron/mL deionised water solution to 2g of air dried 2mm sieved soil at pH (CaCl<sub>2</sub>) 4.1, 4.8, 5.7, 6.5 and 7.2 weighed into 15ml centrifuge tubes and adjusting the soil moisture to 80% of field capacity. Calculations were based on a bulk density of 1.3 g/cm<sup>3</sup> and assuming an incorporation depth of 100mm for surface herbicide applications. Capped centrifuge tubes were incubated in the dark at 20°C. Herbicide concentration was determined at sampling intervals 0, 3, 7, 14, 28, 42, 56, 70 and 98 days, using high performance liquid chromatography, Agilent Technologies 6470 triple-quadrupole LC-MS/MS.

## Glasshouse pot experiment

Two randomised block glasshouse pot trial experiments, each with four replicates were conducted simultaneously. Each of these two tested a SU herbicide, either triasulfuron or chlorsulfuron. These experiments tested the influence of two factors; soil pH and herbicide rate representing a range of possible herbicide residual concentrations, on legume growth and nodulation. Plastic bag lined pots (8cm diameter and 18cm length) containing soil exhibiting pH (CaCl<sub>2</sub>) 4.1, 4.8, 5.7, 6.5 or 7.2 was amended with either triasulfuron 750gai/kg (applied as Triasulfuron 750WG) or chlorsulfuron 750gai/kg (applied as Chlorsulfuron 750WG) at rates of 0.2, 0.04, 0.008, 0.0016, 0.00032 and 0 times a base label rate of 33.0 or 18.9g/ha respectively. Application rates achieved for triasulfuron were 6.60, 1.32, 0.26, 0.05, 0.01 and 0g/ha, and chlorsulfuron 3.77, 0.76, 0.15, 0.03, 0.01 and 0g/ha. Herbicide calculations were based on a bulk density of 1.3 g/cm<sup>3</sup> and assuming an incorporation depth of 100mm for surface herbicide applications. To spike soil with each herbicide concentration, soil from each pot was individually spread onto a tray (resultant 2cm soil depth) and processed through spray cabinet, followed by mixing and repotting, providing a thorough distribution of product throughout the soil profile. Herbicides were applied using a twin nozzle boom with two Teejet® XR11001 nozzles at 250kpa and a height of 50cm applying 80.31L/ha at 5.32km/h. Soil moisture content was 80% of field capacity at herbicide application to reduce initial water additions post herbicide application. The SU herbicide and acid soil sensitive albus lupin variety Rosetta was sown three days post herbicide application on 15 August 2019. Seeds were inoculated with group G peak inoculant at a rate of 5g/kg seed, immediately prior to sowing. Phosphorous fertiliser was applied immediately post sowing at a rate of 10.4mg Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>/ml to achieve an application rate equivalent to 20kg P/ha. The pots were housed in a glasshouse and water content was maintained at 80% of field capacity for the duration of the experiment. Shoot dry matter yield and nodule abundance was determined 10 weeks after sowing. The nodule abundance score represented the total number of nodules per pot in a 10-nodule range bracket and is presented as a numerical score from 1 to 24 (for example a 0-10, 11-20, 21-30... 231-240 total number of nodules count is presented as a 1, 2, 3... 24 abundance score).



## Results

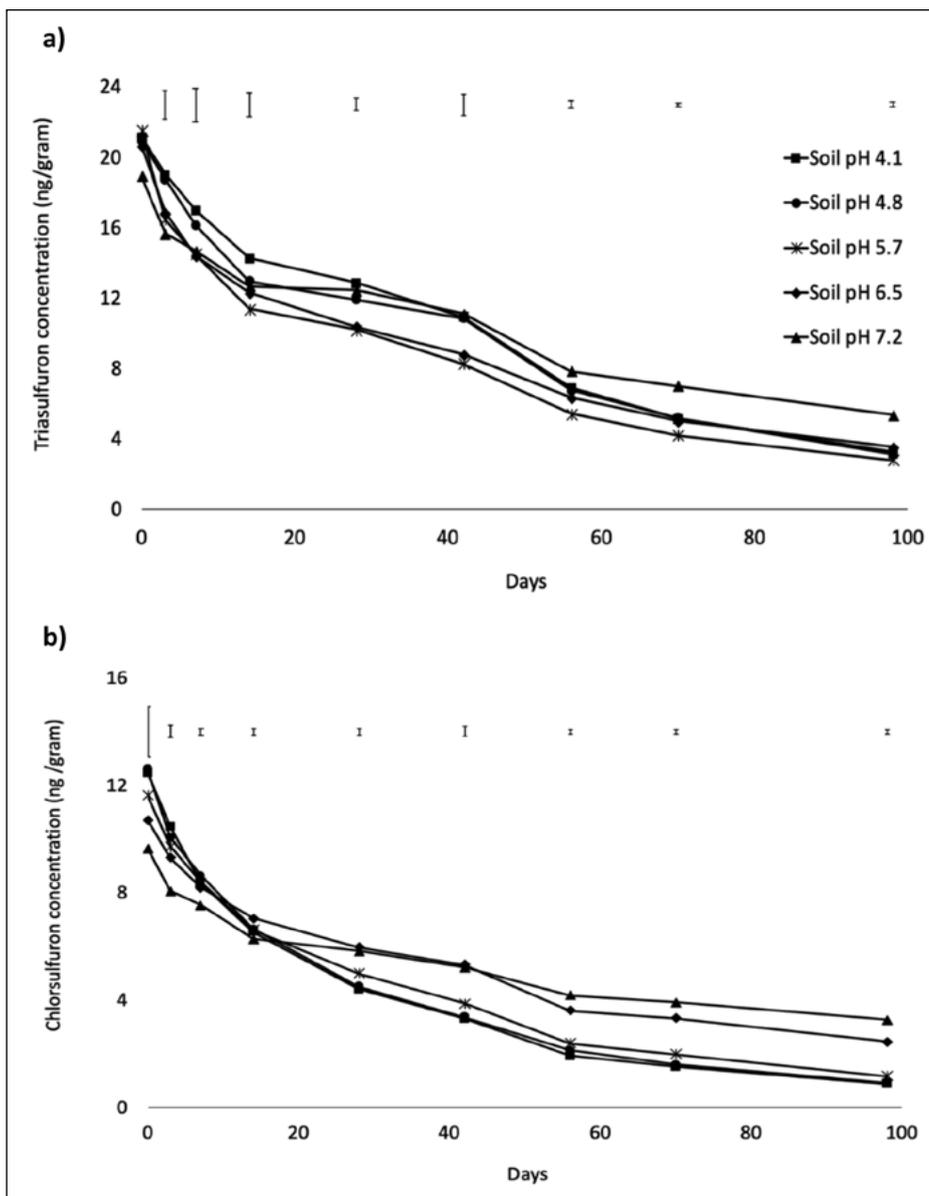
### Controlled degradation study

Soil pH had a significant effect on the rate of degradation of both triasulfuron (Figure 1a) and chlorsulfuron (Figure 1b). Triasulfuron persistence was significantly greater at pH 7.2 than all other soil pH onwards from 56 days post application. The degradation of chlorsulfuron was also sensitive to pH during the same period with increased persistence occurring as soil pH increased from 4.1 to 7.2 (Figure 1b).

### Glasshouse pot experiment

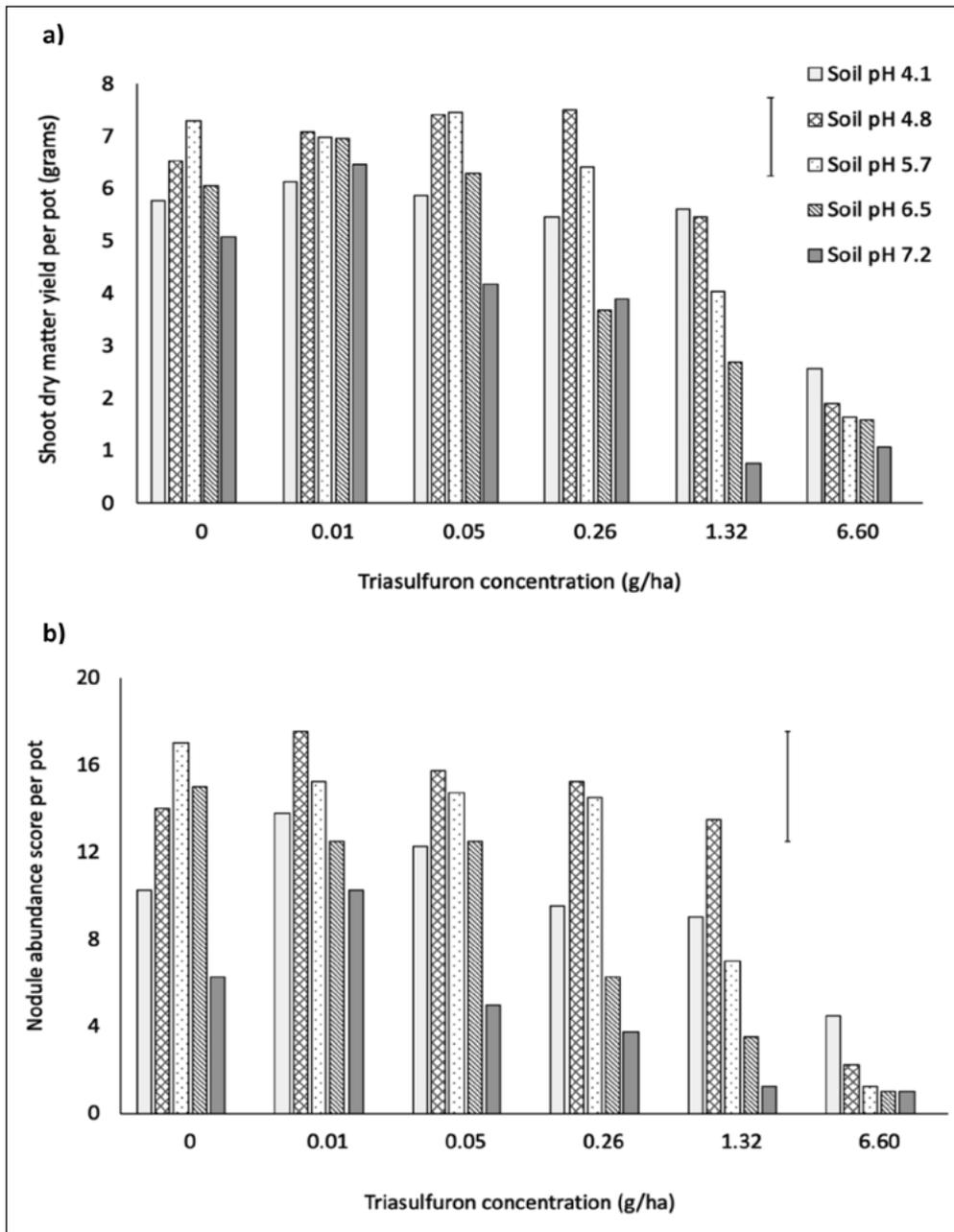
Lupin was susceptible to conditions of both soil pH and herbicide concentration. Significant interaction between soil pH and herbicide treatment occurred for lupin shoot dry matter yield (Figure 2a and Figure 3a) and nodule abundance (Figure 2b and Figure 3b).

In the absence of herbicide, shoot dry matter yield was significantly greater at pH 5.7 than pH 4.1 or pH 7.2 (Figure 2a and Figure 3a). Nodulation in the absence of herbicide also exhibits a pH-optima of approximately pH 4.8 and 5.7 (Figure 2b and Figure



**Figure 1.** Residual concentration through time of (a) triasulfuron 750gai/kg at 35g/ha and (b) chlorsulfuron 750gai/kg at 20g/ha applied to a sandy loam soil at pH 4.1, 4.8, 5.7, 6.5 and 7.2. Capped bars indicate the least significant difference (LSD) at  $P < 0.05$ .





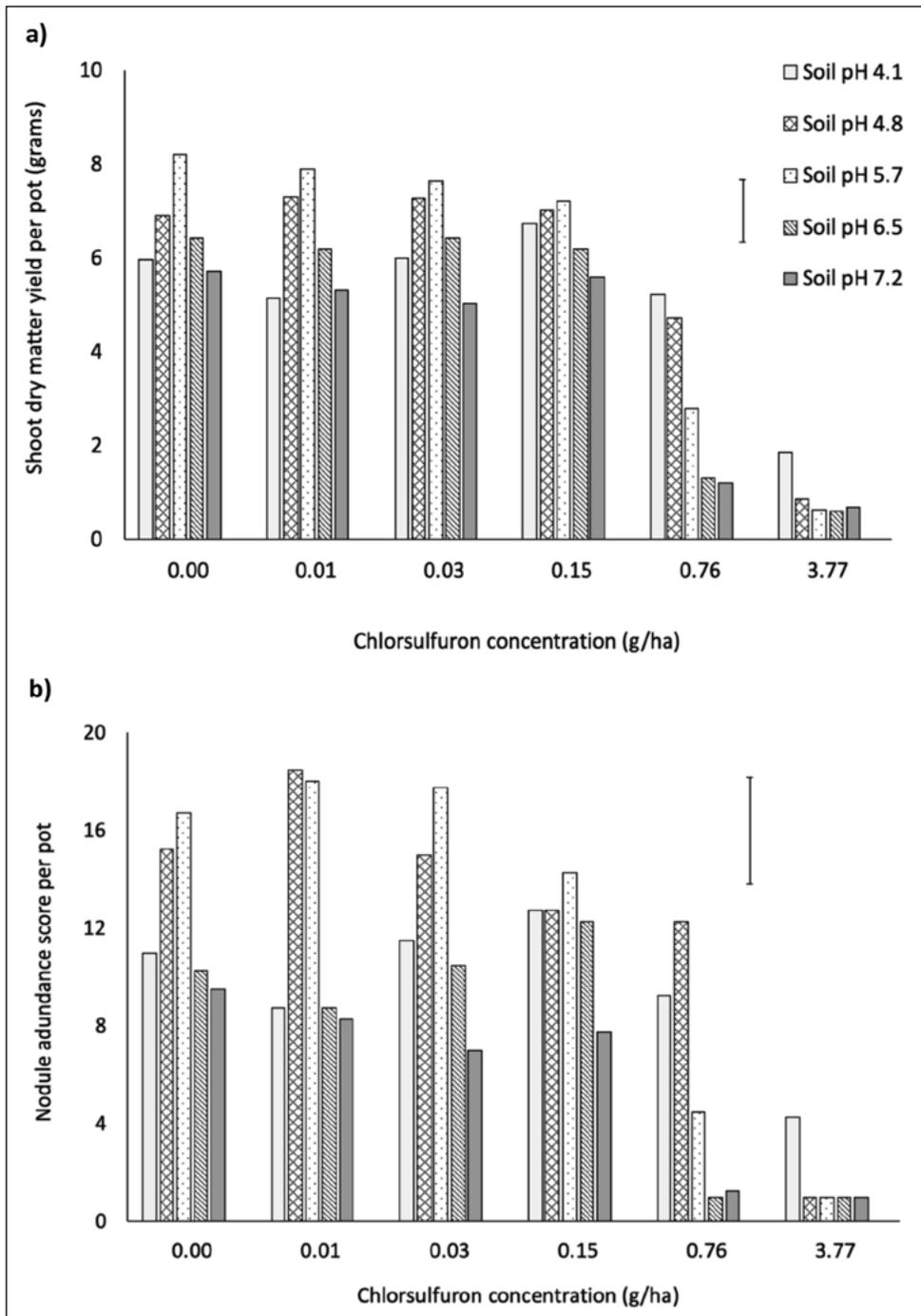
**Figure 2.** Effect of soil pH and varying triasulfuron 750gai/kg concentrations on (a) lupin shoot dry matter yield (g/pot) and (b) nodulation 10 weeks after sowing. Capped line bars indicate least significant difference (LSD) at  $P < 0.05$ .

3b). Therefore, it is likely that the presence of pH stratification, that exhibits acidic subsurface layers of pH less than pH 4.8, would be limiting above ground lupin growth and the nodulation of roots in the field.

The presence of triasulfuron did not influence above ground plant mass at the 0.01g/ha rate compared to the control treatment for any soil pH (Figure 2a). As the concentration of triasulfuron increased from 0.05g/ha to 6.60g/ha the resultant decrease in plant shoot dry matter was greatest at higher pH. At 0.05g/ha, only pH 7.2 produced

a significantly lower plant dry matter compared to all lower herbicide rates at all pHs. At 0.26g/ha the pH 6.5 soil decreased plant growth compared to lower rates of herbicide for that soil pH. At a concentration of 1.32g/ha the pH 4.8 and 5.7 soils exhibited significantly less plant dry matter than the same pH with lower herbicide rates. The pH 7.2 soil experienced further decreases in dry matter production compared to lower rates. At the highest triasulfuron rate (6.60g/ha) the dry matter of the pH 4.1 had also significantly decreased compared to all other rates for that soil pH (Figure 2a).





**Figure 3.** Effect of soil pH and varying chlorsulfuron 750gai/kg concentrations on (a) lupin shoot dry matter yield (g/pot) and (b) nodulation 10 weeks after sowing. Capped line bars indicate least significant difference (LSD) at  $P < 0.05$ .

The presence of pH-optima for nodulation was evident until the highest concentration of triasulfuron (Figure 2b). However, increasing concentration of triasulfuron did significantly decrease nodulation abundance at concentrations of 0.26g/ha for pH 6.5, 1.32g/ha for pH 5.7 and 6.6g/ha for pH 4.1 and 4.8.

The interaction of pH and triasulfuron is such that at the lowest soil pH (pH 4.1), the legume production is limited by pH until the highest triasulfuron rate (6.60g/ha). In general, as the pH of the soil

increased, the triasulfuron concentration required to reduce plant growth decreased. There was a similar trend for the effect of triasulfuron on nodulation.

Increasing the application rate of chlorsulfuron had no effect on plant dry matter production for a given soil pH until the concentration was 0.76g/ha and 3.77g/ha (Figure 3a). At the application rate of 0.76g/ha it is evident that the decrease in dry matter production was greatest at higher soil pH. At the highest concentration (3.77g/ha) all pH treatments



were affected equally. These effects in the above ground dry matter production were reflected in the nodulation abundance (Figure 3b). At pH 4.1 nodulation was reduced compared to other pH treatments. As herbicide concentration increased nodulation score for pH 4.1 did not change until the highest chlorsulfuron concentration.

## Discussion

The degradation study clearly demonstrated that higher soil pH slows the degradation of SU herbicides, reflecting the herbicide labels of these products. This interaction can be explained through increased stability of the SU molecule under neutral pH conditions, slowing the degradation caused via chemical hydrolysis (Hay 1990; Sarmah et al. 2000b). These characteristics have practical implications; restricting plant-back periods post herbicide application with recommended re-cropping intervals increasing as soil pH increases. For example, re-cropping interval for lupins post triasulfuron or chlorsulfuron application increases from 12 months to 22 months as the soil pH range increases from pH (water) 6.5 or less to 6.6 to 7.5. Label guidelines further restrict the use of chlorsulfuron in soils with pH greater than 8.6 due to potential for extended residual activity adversely affecting crop rotation.

Under controlled glasshouse conditions lupin growth and nodulation was decreased by both low soil pH (pH 4.1) and high soil pH (pH 7.2). Low pH effects on legume growth are well documented (Upjohn et al. 2005) and conditions of acidity are normally ameliorated by the application of lime, although lupins are susceptible to 'overliming'. Both acidic and alkaline soils influence the availability of essential macro- and micronutrients resulting in nutrient deficiencies and toxicity affecting crop production (Tang et al. 1995; Hackney et al. 2019).

The consequence of increasing pH beyond the pH optima of the legume, on its dry matter production and nodulation, is worsened by the presence of SU herbicide. The higher the pH, the lower the concentration of SU residue required to negatively affect plant production and nodulation. At low pH where lupin growth is reduced due to pH there appears to be little impact due to the herbicide except at the higher concentrations. This may be due to SU herbicides degrading faster in acid soils than in the higher pH soils or there could be a biological interaction within the plant between soil pH and herbicide residue. From this study it is not possible to determine the mechanism.

Specifically, triasulfuron reduced shoot dry matter by 35% as herbicide concentration increased from

0.01g/ha to 0.05g/ha at pH 7.2. Similarly, at pH 6.5 shoot dry matter decreased by 41% when the herbicide concentration increased from 0.05g/ha to 0.26g/ha. Similar trends were also observed for nodulation. When correlated with findings from the controlled degradation study these concentrations of 0.05g/ha and 0.26g/ha (equivalent to 0.04ng triasulfuron/g and 0.20ng triasulfuron/g, respectively) were 2.8ng/g and 2.6ng/g lower than the most rapid degradation observed at 98 days post application under non-limiting conditions. This highlights the potential for plant injury under environments where herbicide degradation is limited; such as reduced moisture. Lupins tended to be less sensitive to chlorsulfuron compared to triasulfuron with residues based on labelled rates, with growth and nodulation not negatively affected until the second highest concentration of 0.76g/ha, however this reduction was seen for all pH. Likewise, the herbicide concentration of 0.76g/ha (equivalent to 0.58ng/g) was 0.3ng/g lower than the greatest level of degradation observed in the controlled degradation study.

Most soils in southern NSW mixed farming zone do not have a 0-10cm soil pH greater than 6.5. But when those same soils have been tested for pH in smaller increments the presence of pH stratification can be observed (Burns et al. 2017b). Commonly we have been interested in the strongly acidic lower layers that reduce plant growth. However, pH stratification also results in the higher pH surface (0-5cm) soil due to return of alkaline plant material or poorly incorporated lime (Burns et al. 2017b). In pH stratified soil, with surface layers greater than pH 6.5, slowed degradation of triasulfuron and chlorsulfuron may increase the potential for herbicide carryover into a legume rotation phase. The carryover of these herbicides may not kill the crop, but it is likely that the top 0-5cm of the soil may restrict root growth from accessing nutrients from this layer. Soil sampling in intervals smaller than 0-10cm will aid identification of soil pH variations through the soil profile.

## Conclusion

Soil pH stratification within a soil could impact lupin growth by two contrasting processes. Low pH interactions with legume crops are well documented and could prevent root growth. The high pH layers in a stratified soil may also slow the degradation of SU herbicides resulting in reduced plant growth. Removing pH stratification and liming to appropriate levels will reduce the impact of both processes.



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## Contact details

Anne-Maree Farley  
Honours Student, Bachelor of Agricultural Science  
0429 607 820  
afarley@csu.edu.au; annemareefarley@gmail.com



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