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# Gross sulphur mineralisation–immobilisation turnover in soil amended with plant residues

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#### Abstract

The rates of sulphur (S) released to and removed from the soil inorganic pool were estimated using the isotopic dilution technique. In an initial study fresh soil was mixed with combinations of two inorganic S levels (0 and 10  $\mu$ g S g<sup>-1</sup> soil) and three plant residues (wheat straw, perennial ryegrass and oilseed rape) and followed over 32 days of incubation. As <sup>35</sup>S recovery was inadequate prior to day 2 and re-mineralisation of immobilised <sup>35</sup>S occurred after day 8 thereby invalidating the method, estimates of gross S transformation rates should be based on data sampled between days 2 and 8. In the main experiment 16 plant residues with ranges in S contents of 0.08–0.81%, C/S ratios of 50–604 and lignin content of 0.9–10.8 were mixed with soil and carrier-free <sup>35</sup>S label. Net turnover rates varied from 58% of S in Persian clover being immobilised to 76% of S in winter cress being mineralised within 5 days of incubation. Gross S mineralisation was strongly correlated to the C/S ratio of the plant material (*P*<0.001), whereas gross S mineralisation showed a weaker, but still significant, correlation with lignin content (*P*<0.05). The results indicate that immobilisation may predominantly have been a biological process in response to carbon addition while early mineralisation may have been dominated by the biochemical hydrolysis of organic sulphates in the residues. If attention is paid to the various constraints and limitations, isotopic pool dilution using <sup>35</sup>S offers a tool that may prove valuable in understanding and modelling soil S turnover.

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## 1. Introduction

Sulphur deficiency has been reported in parts of the world with previous S sufficiency. The main reasons are: (1) the environmental control of sulphur dioxide emissions in industrial areas, (2) the increasing use of high analysis, low S-containing fertilisers, and (3) the increase in yields obtained as a result of other technological advances (Scherer, 2001; Blair, 2002). In Europe, the controls of S emission have reduced the concentrations of sulphur dioxide in the atmosphere dramatically over the last 20–30 years, leading to lower inputs of S to agricultural land. Zhao et al. (2002) reviewed crop responses to S fertilisation in Europe and concluded that today S has

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become one of the most limiting nutrients for agricultural production in many European countries.

In agricultural systems, where S inputs from fertiliser and atmospheric deposition are low the release of S from organic forms is important for the supply of S to plants and a valuable parameter is the estimation of the potential contribution that the organic S pool makes to plant-available S, especially following the addition of fresh organic matter to the soil. Mineralisation and immobilisation of S occur concurrently (Maynard et al., 1983; Ghani et al., 1993; Eriksen, 1997b) and the release or incorporation of inorganic sulphate is thus the net result of several processes. Quantification of gross nutrient fluxes has been used successfully to understand the fundamentals of these processes for both nitrogen (Murphy et al., 2003) and phosphorus (Di et al., 1994) using isotopic dilution techniques. The principle of this methodology is to label the mineral nutrient pool with an isotopic tracer and measure the change with time. The rates of influx to and out flux from the labelled pool are then calculated using

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equations based on tracer kinetics. Although these equations were developed already in 1950s (Kirkham and Bartholomew, 1954) it was not until recently this technique has been widely used (Di et al., 2000; Murphy et al., 2003). The <sup>35</sup>S tracer has been used to study S transformations in soil without organic matter addition (e.g. Ghani et al., 1993; Eriksen, 1997a; Goh and Pamidi, 2003; Vong et al., 2003), following addition of urinary or faecal S (e.g. Blair et al., 1994; Nguyen and Goh, 1994; Williams and Haynes, 2000) and plant residues (Wu et al., 1993, 1995) but the use of the tracer dilution method for determination of gross S transformation rates have had little attention.

The objective of this work was to establish a suitable technique for quantitative determination of gross S transformation rates and to investigate its applicability on a range of plant materials. Sectin 3 will be interpreted in relation to traditional S release rates determined by plant uptake.

#### 2. Materials and methods

Lundgaard soil (for description see Eriksen et al., 1995) collected from the top 0–15 cm was sieved (<2 cm) to remove stones and plant residues. The soil contained 4.6% clay, 0.9% carbon and 150 µg organic S g<sup>-1</sup> soil.

#### 2.1. Establishing methodology (Experiment I)

Prior to the determination of gross transformation rates on a wide range of plant residues, the method was tested to determine effects and interactions of initial sulphate level, residue composition and sampling time. In this experiment, organic matter and labelling were separated in time to distinguish effects of organic matter and label addition.

Eight 15-kg portions of fresh soil were mixed with combinations of two S treatments (0 and 10 mg S kg<sup>-1</sup> dry soil) and four plant residue treatments (control, wheat straw,

Table 1				
Composition	of	plant	residu	ies

perennial ryegrass and oilseed rape; milled to pass a 2 mm sieve; 10 g kg<sup>-1</sup> dry soil; Table 1) and incubated for 5 days at 20 °C. Carrier-free H<sub>2</sub><sup>35</sup>SO<sub>4</sub> was diluted with de-ionised water and added to eight 500-g portions of quartz sand, allowed to dry, ground and thoroughly mixed by shaking end-over-end for 3 h. The <sup>35</sup>S-containing sand was mixed with each of the eight treatment soils (0.5 kBq g soil<sup>-1</sup>) and 39 portions of 250 g soil were placed in 250-ml plastic bottles at 20 °C and covered by perforated film to allow for aeration. On 13 occasions (days 0, 1, 2, 3, 4, 6, 8, 10, 12, 14, 16, 18 and 32) three replicates were sampled from each of the eight treatments. During the experiment water content was adjusted every 2–3 days to the initial level of 11%.

# 2.2. Determination of gross mineralisation-immobilisation rates from plant residues (Experiment II)

Based on Experiment I results, a technique was chosen with labelling at the time of organic matter addition and last sampling at day 8. Seventeen 5-kg portions of fresh soil were incubated for 5 days at 20 °C. Except for a control treatment, plant material (milled to pass a 2 mm sieve; 10 g kg<sup>-1</sup> dry soil; Table 1) was added to each soil portion also receiving 100 g of sand containing carrier-free  $H_2^{35}SO_4$  (see above) and mixed. For each treatment 12 portions of 250 g soil were placed in 250-ml plastic bottles at 20 °C and covered by perforated film to allow for aeration. On days 1, 5 and 8 four replicates were sampled from each of the 17 treatments.

## 2.3. Plant and soil analyses

In plant residues, total-N was determined using Dumas combustion (Hansen, 1989) and total-S was determined by turbidimetry after wet ashing with magnesium nitrate and perchloric acid (Nes, 1979). The lignin content of catch crops was determined by the method of Van Soest (1963).

Common name	Scientific name	S (%)	C:N:S	Lignin (%)
Wheat straw <sup>a</sup>	Triticum aestivum L. ssp. vulgare	0.08	604:75:1	8.7
Kidney vetch 1	Anthyllis vulneraria L.	0.13	329:17:1	5.3
Persian clover	Trifolium resupinatum	0.13	324:17:1	5.0
Black medic	Medicago lupulina L.	0.16	278:14:1	4.5
Red clover	Trifolium pratense L.	0.16	276:15:1	9.3
White clover	Trifolium repens L.	0.17	250:14:1	6.8
Perennial lupine	Lupinus polyphyllos	0.17	246:15:1	4.5
Rye/vetch	Secale cereale L./Vicia sativa L.	0.17	222:11:1	4.4
Perennial ryegrass 1	Lolium perenne L.	0.19	226:24:1	6.4
Kidney vetch 2	Anthyllis vulneraria L.	0.20	221:13:1	3.7
Sorrel	Rumex Acetósa L.	0.23	188:16:1	10.8
Echium	Echium vulgare	0.26	146:14:1	5.6
Chicory	Cichorium intybus L.	0.28	146:22:1	6.2
Perennial ryegrass 2 <sup>a</sup>	Lolium perenne L.	0.38	116:11:1	1.5
Oilseed rape <sup>a</sup>	Brassica napus L.	0.70	59:8:1	0.9
Early wintercress	Barbarea verna	0.81	50:11:1	1.4

<sup>a</sup> Used in the methodological study (Experiment I).

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