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Quantifying the sulphur (S) supply from farm manures to winter wheat crops

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1. Abstract

The overall aim of this project was to quantify sulphur (S) supply from organic materials to winter wheat crops, in order to improve current recommendations on the use of farm manures and biosolids as sources of crop available S for arable crops.

An enhanced database of the S content of organic materials found good agreement with the 'typical' values given in Defra's Fertiliser Manual (RB209) for livestock manures (cattle farmyard manure - FYM, pig FYM, cattle slurry and pig slurry), digested liquid, lime stabilised and thermally dried biosolids. However, for digested cake and composted biosolids, the mean total SO₃ content was higher than the 'typical' values given in Defra's Fertiliser Manual (RB209) (Defra, 2010). The S content of organic materials (both total SO₃ and 'extractable' SO₃) was shown to be variable; hence, analysis of a representative organic material sample is advisable to ensure that crops grown on potentially deficient sites receive adequate S.

Field experiments were carried out at 3 sites cropped with winter wheat over 3 harvest years (2 harvest years at each site; 6 harvest years in total). At each site, there were 7 organic material treatments, including autumn applied cattle FYM, pig FYM, broiler litter and two biosolids products, and spring applied broiler litter and pig/cattle slurry. Three of the 6 sites responded to S and at these sites the organic material treatments were compared with inorganic (water soluble) fertiliser S response treatments (0, 12.5, 25, 50 and 75 kg/ha SO₃) to determine the fertiliser S replacement value and hence S availability from the applied organic materials.

For the spring applied organic materials, 'extractable' SO₃ (i.e. readily available SO₃) appeared to be a good indicator of S that was available to the crop. Analysis of the organic materials used in this project showed that 'extractable' SO₃ varied between around 15% of total SO₃ for cattle FYM, up to around 60% of total SO₃ for broiler litter. Results from this project showed that for spring applied organic materials, 'extractable' SO₃ was equivalent to inorganic fertiliser S i.e. the S use efficiency for spring applications is 15% of total SO₃ for cattle FYM, 25% for pig FYM, 60% for broiler litter, 35% for slurry and 20% for biosolids.

Readily available S from organic materials applied in the autumn may be lost via overwinter leaching with losses dependant on soil type and overwinter rainfall. Results from this project showed a lower S use efficiency from autumn compared with spring applied organic materials; typical autumn S use efficiencies in the range 5–10% of total SO₃ for livestock manures and 10– 20% of total SO₃ for biosolids.

This work has led to a better understanding of the crop available S supply from organic materials and produced guidance to farmers on the availability of S from applications of organic materials. This is likely to improve farm profitability by ensuring that crops receive adequate amounts of S from applied organic materials or, where necessary, that supplementary inorganic S fertiliser additions are made to meet crop needs.

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2. Introduction

Sulphur (S) has important effects on yield and quality of crops. Sulphur deficiency in susceptible arable crops has become more widespread over recent decades as levels of atmospheric S deposition have continued to decline (and are now estimated to be only 10% of levels deposited in the 1980's). Fertiliser S is now routinely advocated for susceptible crops in areas of high risk of S deficiency. Since the early 1990's, the tillage area receiving S fertiliser has increased from 3–6% of the cereal area and 8% of the oilseed rape area to 45–52% of the cereal area and 73% of the oilseed rape area to 45–52% of the cereal area and 73% of the oilseed rape area (in 2012), with average field application rates currently 54 kg/ha SO₃ for winter wheat and 86 kg/ha SO₃ for oilseed rape (BSFP, 2012). The cost of S fertiliser applied to the cereal (winter wheat, winter barley and spring barley) and oilseed rape area in 2012 is estimated at £18 million (based on £0.15/kg SO₃).

Organic materials contain useful quantities of S, as well as other plant nutrients and organic matter and are used on around 70% of farms in Britain (BSFP, 2012). However, prior to this project, there was very little information on the crop availability of S from organic materials, and the current 8th edition of Defra's Fertiliser Manual (RB209) does not provide advice on the fertiliser S replacement value of organic materials. There is a need to better understand the available S supply from organic materials so that farmers can reduce their manufactured fertiliser S use accordingly.

The overall aim of this project was to quantify the S supply from organic materials to winter wheat crops, specifically to:

- collate existing data on the S content of farm manures and biosolids
- quantify, and make better allowance for, the S supply from organic materials in nutrient management planning
- improve current recommendations on the use of organic materials as sources of crop available S for arable crops
- publicise the results of the project to farmers.

3. Review of organic material sulphur content

3.1. Introduction

The S content of organic materials may be very variable, depending on, for livestock manures, the S content of feed, bedding use (for solids manures) and dilution (for slurries) and, for biosolids, the source of waste water and biosolids treatment process. A review of published and unpublished information has collated the available UK data on the range and typical concentrations of S in livestock manures and biosolids.

3.2. Materials and methods

The review of livestock manure S content data included data from:

- i. Manure Analysis Database (MANDE) (Defra project NT2006, Chambers, 2003): this database includes the analysis of more than 800 individual samples of *livestock* manures, collected between 1992 and 2002, and is the basis for the 'typical' figures for the nutrient content of livestock manures in Defra's Fertiliser Manual (RB209).
- ii. LINK project on developing analysis of manure by NIRS (LK0988; HGCA Project Report 489). This project included the analysis (by wet chemistry) of 325 individual samples of livestock manures collected between 2007 and 2009 (cattle FYM, pig FYM, cattle slurry and pig slurry).

The review of livestock manure S content data included cattle FYM, pig FYM, cattle slurry and pig slurry; however, poultry manures and other livestock manure types were excluded in the absence of any new additional data for these other manure types.

The review of biosolids S content data included data from:

- iii. LINK project on developing manure analysis of by NIRS (LK0988; HGCA Project Report 489). This project included the analysis (by wet chemistry) of 93 individual samples of biosolids collected between 2007 and 2009. Eighty five of the analyses were from samples collected from 2 water companies; 8 further analyses were excluded as the method of biosolids treatment (i.e. digested/composted/limed) was unknown.
- iv. UKWIR research (Withers and Smith, 1996) which included analytical data for 61 samples of biosolids, collected in 1995 from treatment centres around the UK, for total and extractable S.
- Data supplied by water companies: additional data on biosolids total S content was provided by a third water company; 29 analyses from different treatment centres/biosolids types were provided – each value was the rolling median value for that treatment centre/biosolids type over the period 2007 to 2012.

All data sources reported analysis data for organic material *total* S content. Only Withers and Smith (1996) also included biosolids analysis for *extractable* SO_3 .

The S content (as kg/t or kg/m³ fresh weight SO₃) of livestock manures (cattle FYM, pig FYM, cattle slurry and pig slurry) and biosolids (liquid digested, digested cake, thermally dried, composted and lime treated) were summarised giving the mean, median, upper and lower 10%ile and range for each organic material type. For all organic materials, the majority of the S is in the dry matter. Data from Withers and Smith (1996) are expressed as % dry matter (with no data on dry matter content given); therefore biosolids data for S content data are summarised on both a fresh weight and dry matter basis.

Analysis of the 36 organic materials from the field experiments in this current project included analysis for total S and 'extractable' S ($0.016M \text{ KH}_2\text{PO}_4$ extraction and analysis via ICP-AES; Zhao, and McGrath, 1994). These data are compared with data on biosolids 'extractable' S content from Withers and Smith (1996) (measured using the same extraction and analysis method). No additional data on the extractable S content of livestock manures are available.

3.3. Results

3.3.1. Livestock manures

Cattle FYM

The mean, median, upper and lower 10%ile and ranges of cattle FYM dry matter and total SO₃ analysis data from the MANDE, NIRS and combined MANDE and NIRS databases are summarised in Table 1.

Variate	MANDE		NI	RS	MANDE + NIRS	
	DM (%)	Total SO ₃	DM (%)	Total SO ₃	DM (%)	Total SO ₃
	(70)	(Kg/l)	(70)	(Kg/l)	(/0)	(Kg/l)
Sample No.	224	224	101	101	325	325
Mean	22.7	2.4	26.4	2.8	23.8	2.5
Median	21.6	2.1	22.5	2.4	22.0	2.2
Min	10.5	0.8	14.5	0.9	10.5	0.8
Max	47.4	6.6	73.4	8.3	73.4	8.3
10 %ile	14.8	1.1	17.5	1.4	15.7	1.2
90 %ile	32.0	3.9	39.7	4.7	33.0	4.1

Table 1. Cattle FYM total SO₃ analysis data (fresh weight basis)

There was a good agreement between the mean cattle FYM total SO₃ content of the MANDE database (2.4 kg/t SO₃) and the NIRS database (2.8 kg/t SO₃); the slightly higher mean SO₃ content of the NIRS database reflects the inclusion of a small number of high dry matter cattle FYM samples in the NIRS database (Figure 1; 7 samples with \geq 50% DM), with consistently higher SO₃ contents. The MANDE and NIRS databases combined (325 samples) gave an overall mean of 2.5 kg/t SO₃, which is very close to the current 'typical' value of 2.4 kg/t SO₃ given in Defra's Fertiliser Manual (RB209). There was a significant relationship (*P*<0.001; R² = 46%) between cattle FYM total SO₃ content and dry matter content (Figure 1).



Figure 1. Relationship between cattle FYM total SO₃ and dry matter content.

Pig FYM

The mean, median, upper and lower 10% ile and ranges of pig FYM dry matter and total SO₃ analysis data from the MANDE, NIRS and combined MANDE and NIRS databases are summarised in Table 2.

Variate	MANDE		NI	RS	MANDE + NIRS	
	DM	Total SO ₃	DM	Total SO ₃	DM	Total SO ₃
	(%)	(kg/t)	(%)	(kg/t)	(%)	(kg/t)
Sample No.	35	35	70	70	105	105
Mean	24.9	3.4	23.9	3.9	24.2	3.7
Median	23.1	3.2	22.0	3.3	22.1	3.2
Min	14.9	<0.1	12.4	0.7	12.4	0.0
Max	54.0	7.3	50.4	11.9	54.0	11.9
10 %ile	17.6	1.6	17.1	1.7	17.3	1.6
90 %ile	31.8	5.3	35.7	6.8	35.5	6.4

Table 2. Pig FYM total SO₃ analysis data (fresh weight basis)

The mean pig FYM total SO₃ content of the NIRS database (3.9 kg/t SO₃) was similar to the MANDE database (3.4 kg/t SO₃). The overall mean SO₃ content of the combined MANDE and NIRS databases (105 samples) was 3.7 kg/t SO₃ and similar to the 3.4 kg/t SO₃ given in Defra's Fertiliser Manual (RB209). There was a significant, but weak relationship (P<0.001; R² = 11%) between pig FYM total SO₃ content and dry matter content (Figure 2).



Figure 2. Relationship between pig FYM total SO₃ and dry matter content

Cattle slurry

The mean, median, upper and lower 10% ile and ranges of pig FYM dry matter and total SO_3 analysis data from the MANDE, NIRS and combined MANDE and NIRS databases are summarised in Table 3.

Variate	MANDE		NI	RS	MANDE + NIRS	
	DM	Total SO ₃	DM	Total SO ₃	DM	Total SO ₃
	(%)	(kg/t)	(%)	(kg/t)	(%)	(kg/t)
Sample No.	96	96	74	74	170	170
Mean	9.1	1.0	6.3	0.8	7.9	0.9
Median	10.0	0.9	5.1	0.6	8.3	0.9
Min	0.8	0.0	0.2	0.0	0.2	0.0
Max	14.9	1.8	20.7	2.7	20.7	2.7
10 %ile	2.4	0.4	0.9	0.1	1.3	0.2
90 %ile	13.5	1.5	13.9	1.7	13.6	1.5

Table 3. Cattle slurry total SO₃ analysis data (fresh weight basis)

There was a strong and significant relationship (P<0.001; R² = 76%) between cattle slurry total SO₃ content and dry matter content (Figure 3). Based on this relationship between total SO₃ content and dry matter content, mean SO₃ content normalised to standard dry matter contents (2, 6 and 10% DM) were calculated (Table 4) and are the same as currently given in Defra's Fertiliser Manual (RB209).

Dry matter (%)	Total SO₃ (kg/m³) <i>Current RB209 valu</i> es	Total SO₃ (kg/m³) MANDE + NIRS database
2	0.3	0.3
6	0.7	0.7
10	1.2	1.2



Figure 3. Relationship between cattle slurry total SO₃ and dry matter content

Pig slurry

The mean, median, upper and lower 10%ile and ranges of pig FYM dry matter and total SO₃ analysis data from the MANDE, NIRS and combined MANDE and NIRS databases are summarised in Table 5. The NIRS database significantly increased the number of pig slurry SO₃ analyses (from 17 analyses in the MANDE database to 94 in the combined MANDE plus NIRS database).

Variate	MANDE		NI	RS	MANDE + NIRS	
	DM	Total SO ₃	DM	Total SO ₃	DM	Total SO ₃
	(%)	(kg/m ³)	(%)	(kg/m³)	(%)	(kg/m ³)
Sample No.	17	17	77	77	94	94
Mean	5.0	1.1	3.2	0.7	3.5	0.8
Median	2.8	1.3	1.9	0.6	2.0	0.6
Min	0.4	0.1	0.3	0.0	0.3	0.0
Max	15.1	2.1	18.6	3.3	18.6	3.3
10 %ile	1.0	0.2	0.8	0.1	0.8	0.1
90 %ile	12.7	2.0	8.2	1.6	9.2	1.8

Table 5. Pig slurry total SO₃ analysis data (fresh weight basis)

There was a strong and significant relationship (P<0.001; R² = 62%) between pig slurry total SO₃ content and dry matter content (Figure 4). Based on this relationship between total SO₃ content and dry matter content, mean SO₃ content normalised to standard dry matter contents (2, 4 and 6% DM) was calculated (Table 6), with results very similar to the values given in Defra's Fertiliser Manual (RB209).

Table 6. Pig slurry total SO₃ content normalised to standard dry matter contents

Dry matter (%)	Total SO₃ (kg/m³) <i>Current RB209 valu</i> es	Total SO₃ (kg/m ³) MANDE + NIRS database
2	0.7	0.6
4	1.0	0.9
6	1.2	1.2



Figure 4. Relationship between pig slurry total SO₃ and dry matter content

3.3.2. Biosolids

The review of biosolids S content divided biosolids into the 5 biosolids categories in Defra's Fertiliser Manual (RB209) – digested liquid, digested cake, thermally dried, lime stabilised and composted. Data that could not be categorised into one of these 5 types were excluded from the review; Withers and Smith (1996) included analyses of a number of crude (i.e. untreated) liquid and dewatered biosolids, which were excluded from this review because untreated biosolids can no longer be recycled to agricultural land.

Most of the variation in the Fertiliser Manual (RB209) standard figures for biosolids S content between the 5 biosolids types can be explained by variation in dry matter content. Where these standard figures are expressed on a %DM basis, values for digested liquid, digested cake and thermally dried are very similar (2.4-2.5% SO₃), the value for limed slightly lower (2.1% SO₃) and only the value for composted biosolids was markedly different (0.4% SO₃) (Table 7).

Category	Dry matter (%)	Total SO₃ (kg/t or m ³ FW)	Total SO ₃ (% DM)
Digested liquid	4	1.0	2.5
Digested cake	25	6.0	2.4
Thermally dried	95	23.0	2.4
Lime stabilised	40	8.5	2.1
Composted	60	2.6	0.4

Table 7. Sulphur content of biosolids (typical values from RB209)

Digested liquid

The mean, median, upper and lower 10% ile and ranges of biosolids digested liquid total SO₃ analysis data is given in Table 8. The data include 3 samples from the NIRS project ('Water company 1') and 24 samples from Withers and Smith (1996). The SO₃ analysis of the 3 digested liquid samples from the NIRS project were within the range of the values from Withers and Smith

(1996) and the mean SO₃ content of the 3 digested liquid samples from 'Water company 1' were the same as the mean of the Withers and Smith (1996) data (2.7% SO₃).

	N	later company	1	Withers & Smith	All data
Variate	DM (%)	Total SO₃ (kg/m ³ FW)	Total SO₃ (%DM)	Total SO₃ (%DM)	Total SO₃ (% DM)
Sample No.	3	3	3	24	27
Mean	2.9	0.8	2.7	2.7	2.7
Median	3.4	0.9	2.7	2.7	2.7
Min	2.0	0.5	2.4	1.9	1.9
Max	3.4	1.0	3.0	3.7	3.7
10 %ile	N/A	N/A	N/A	2.2	2.2
90 %ile	N/A	N/A	N/A	3.4	3.4

 Table 8. Digested liquid biosolids total SO3 analysis data

N/A – Not applicable

The overall mean SO₃ content of liquid digested biosolids was 2.7% SO₃, equivalent to 1.1 kg/m³ SO₃ (fresh weight) for 4% dry matter liquid digested biosolids, and is very close to the current 'typical' value of 1.0 kg/m³ SO₃ given in Defra's Fertiliser Manual (RB209) (Table 7).

Digested cake

The mean, median, upper and lower 10% ile and ranges of digested biosolids cake total SO_3 analysis data are given in Table 9. These data include 69 samples: 40 samples from the NIRS project ('Water company 1'), 18 samples from 'Water company 3' and 11 samples from Withers and Smith (1996).

	Water company 1			Wat	ter compar	าу 3	Withers & Smith	All data
Variate	DM (%)	Total SO ₃ (kg/t FW)	Total SO₃ (%DM)	DM (%)	Total SO ₃ (kg/t FW)	Total SO₃ (%DM)	Total SO₃ (%DM)	Total SO₃ (% DM)
Sample No.	40	40	40	18	18	18	11	69
Mean	23.8	8.2	3.5	22.9	8.8	3.8	2.4	3.4
Median	21.6	7.5	3.4	22.7	9.0	4.0	2.5	3.3
Min	16.9	5.9	2.0	17.6	2.1	1.0	1.7	1.0
Max	57.3	19.0	6.1	28.7	13.1	5.0	3.1	6.1
10 %ile	19.0	6.4	2.7	19.3	5.7	3.0	1.9	2.4
90 %ile	30.6	10.4	4.6	27.1	11.8	4.6	2.9	4.4

Table 9. Digested biosolids total SO₃ analysis data

The mean total SO₃ content of digested biosolids cake from 'Water company 1' and 'Water company 3' was similar at 3.5 and 3.8% SO₃, respectively, and *c*.50% greater than the mean value reported by Withers and Smith (2.4% SO₃). The overall mean (all data) total SO₃ content was 3.4% SO₃, equivalent to 8.5 kg/t SO₃ (fresh weight) for a 25% dry matter digested biosolids cake, and notably higher than the current 'typical' value of 6.0 kg/t given in Defra's Fertiliser Manual (RB209) (Table 7). The mean total SO₃ content at 3.4% SO₃ is also higher than the mean values for the other biosolids categories reported here (range 2.0% SO₃ for composted to 2.7% SO₃ for liquid digested).

Thermally dried

There were very limited data available on the S content of thermally dried biosolids; 4 samples from the NIRS project ('Water company 2') and 1 sample from Withers and Smith (1996). Total SO₃ in these 5 samples ranged from 2.3 to 3.0% SO₃, with a mean of 2.6% SO₃; equivalent to 25 kg/t SO₃ (fresh weight) for a 95% dry matter thermally dried biosolids, which is very close to the current 'typical' value of 23 kg/t given in Defra's Fertiliser Manual (RB209) (Table 7).

Lime stabilised

The mean, median, upper and lower 10% ile and ranges of lime stabilised biosolids total SO_3 analysis data is given in Table 10. The data include a total of 15 samples; 4 samples from the NIRS project ('Water company 1') and 11 samples from 'Water company 3'.

	Water company 1			Wa	All data		
Variate	DM %	Total SO ₃ (kg/t FW)	Total SO₃ (%DM)	DM (%)	Total SO ₃ (kg/t FW)	Total SO₃ (%DM)	Total SO₃ (% DM)
Sample No.	4	4	4	11	11	11	15
Mean	32.3	7.4	2.5	31.4	6.6	2.1	2.2
Median	34.1	7.3	2.2	31.3	6.5	2.0	2.1
Min	18.7	6.4	1.8	26.3	3.8	1.2	1.2
Max	42.4	8.7	3.9	36.2	9.9	3.1	3.9
10 %ile	N/A	N/A	N/A	26.7	4.4	1.4	1.6
90 %ile	N/A	N/A	N/A	34.9	9.4	2.9	3.0

Table 10. Lime stabilised biosolids total SO₃ analysis data

N/A – Not applicable

The mean total SO₃ content of lime stabilised biosolids from 'Water company 1' and 'Water company 3' were similar at 2.5 and 2.1% SO₃, respectively. The overall mean (all data) total SO₃ content was 2.2% SO₃, equivalent to 8.8 kg/t SO₃ (fresh weight) for a 40% dry matter lime stabilised biosolids, and was very close to the current 'typical' value of 8.5 kg/t SO₃ given in Defra's Fertiliser Manual (RB209) (Table 7).

Composted

The mean, median, upper and lower 10%ile and ranges of composted biosolids total SO₃ analysis data are given in Table 11. The data include 33 samples; 31 samples of 'phytoconditioned' biosolids from 'Water company 2' and 2 samples from Withers and Smith (1996). The phytoconditioning process used by 'Water company 2' involves the addition of green waste to digested dewatered biosolids and then a rye grass crop being grown on the material. Phytoconditioned biosolids may be compared to the 'composted' biosolids category within Defra's Fertiliser Manual (RB209), as both processes involved addition of a bulking agent (i.e. green waste). However, phytoconditioning is distinct from composting in that the material is not actively composted (i.e. turned) and a crop is grown on the material; therefore the nutrient content of phytoconditioned biosolids may vary from composted biosolids produced by other water companies.

	V	Vater company	Withers & Smith	All data	
Variate	DM (%)	Total SO₃ (kg/t FW)	Total SO₃ (%DM)	Total SO₃ (%DM)	Total SO₃ (%DM)
Sample No.	31	31	31	2	33
Mean	34	6.2	2.0	1.7	1.9
Median	31	6.4	1.9	N/A	1.9
Min	24	1.9	0.4	0.9	0.4
Max	63	11.3	4.1	2.5	4.1
10 %ile	27	4.0	1.2	N/A	0.9
90 %ile	47	9.3	2.9	N/A	2.9

Table 11. Composted biosolids total SO₃ analysis data

N/A – Not applicable

The total SO₃ content of the 2 samples from Withers and Smith (1996) was within the range of the data from 'Water company 2'. The overall mean total SO₃ content is 1.9% SO₃, equivalent to 11.7 kg/t SO₃ at 60% DM, which is considerably higher than the current 'typical' value of 2.6 kg/t (0.4%) SO₃ given in Defra's Fertiliser Manual (RB209) (Table 7), although still slightly lower than the mean values for other biosolids categories in this review (2.2 to 3.4% SO₃). Addition of a bulking material like green waste (during composting or phytoconditioning) is likely to reduce the SO₃ content of biosolids through dilution, if the added material has a lower SO₃ content than the biosolids.

The data from this review suggests that the current RB209 SO_3 value for composted biosolids is an underestimate. However, as the results within this review were dominated by the products of a single water company, which used a treatment process slightly different to 'typical' composting (i.e. phytoconditioning), further analysis of composted biosolids (from other water companies) should be undertaken before any revision to the RB209 SO_3 value for composted biosolids.

3.3.3. Extractable SO₃

'Extractable' S gives total S in the extract and includes SO_4 -S and some dissolved organic S. It is likely that any dissolved organic S extracted represents the more labile pool of organic S in the organic material, which can become available to plants through mineralisation to SO_4 . Studies on grassland soils have shown that soil testing methods which include a fraction of organic S along with extractable SO_4 correlated best with the availability of S to herbage (Blair *et al.*, 1991), and the organic S extracted was directly related to the mineralisable organic S (Watkinson *et al.*, 1991).

The mean, median and range of extractable S in the 36 samples of organic materials used in the project is given on a fresh weight basis in Table 12 and as a proportion of organic material total S in Table 13. Table 14 summarises data on biosolids extractable S content, with mean, median and range of biosolids extractable S as a proportion of total S given for the 36 samples reported by Withers and Smith (1996).

The extractable SO₃ content of organic materials varied between a mean of 0.4 kg/t SO₃ for pig FYM up to 4.9 kg/t SO₃ for broiler litter. As a proportion of total SO₃, extractable SO₃ varied between c.15% of total SO₃ for cattle FYM up to c.60% of total SO₃ for broiler litter. Biosolids

extractable SO₃ measured in samples from the current project (mean 22% of total S) was higher than reported by Withers and Smith (mean 11-13% of total S).

Organic	Sample No.	Mean	Median	Minimum	Maximum		
material type		Extractable SO ₃ (kg/t FW)					
Cattle FYM	6	0.37	0.25	0.15	0.76		
Pig FYM	6	1.18	1.23	0.87	1.39		
Broiler litter	12	4.89	5.56	1.76	7.95		
Slurry	6	0.47	0.21	0.11	1.65		
Biosolids							
Digested	6	1.69	1.45	0.75	3.04		
Limed	6	1.98	2.10	0.93	2.57		
All biosolids	12	1.84	1.75	0.75	3.04		

Table 12. Organic material extractable SO₃ analysis data (kg/t FW)

Organic	Sample No.	Mean	Median	Minimum	Maximum	
material type		Extractable SO ₃ (% total SO ₃)				
Cattle FYM	6	13	14	10	17	
Pig FYM	6	25	24	19	38	
Broiler litter	12	58	59	38	70	
Slurry	6	36	25	18	61	
Biosolids						
Digested	6	19	16	12	34	
Limed	6	25	25	20	30	
All biosolids	12	22	21	12	34	

Table 14. Biosolids extractable SO₃ as a % total SO₃ applied (Withers & Smith, 1996)

Biosolids type	Sample No.	Mean	Median	Minimum	Maximum
		Extractable SO ₃ (% total SO ₃)			
Digested liquid	23	11	11	5	18
Digested cake	11	13	11	6	22
Composted	2	11	N/A	7	16

N/A – Not applicable

3.4. Discussion

- For livestock manures (cattle FYM, pig FYM, cattle slurry and pig slurry) and digested liquid, lime stabilised and thermally dried biosolids, there was good agreement between the mean total SO₃ content of the data considered in this review and the 'typical' values given in Defra's Fertiliser Manual (RB209).
- For digested cake and composted biosolids, the mean total SO₃ content of data considered in this review was notably higher than the 'typical' values given in Defra's Fertiliser Manual (RB209), indicating that the current 'typical' values may be an underestimate. However, for composted biosolids, as this review was focused on data from a single water company, which used a treatment process slightly different to 'typical' composting (i.e. phytoconditioning), further analysis of composted biosolids (from other water companies) should be undertaken before any revision to the RB209 SO₃ value for composted biosolids.

• Limited data from analysis of samples from the current project showed that the proportion of total S in the 'extractable' form can vary significantly between organic material types, from *c*.15% of total S for cattle FYM up to *c*.60% of total S for broiler litter.

4. Materials and methods – field experiments

4.1.1. Sites

Field experiments were carried out at 3 sites cropped with winter wheat over 3 harvest years (2 harvest years at each site; 6 harvest years in total). Field sites were located at;

Frostenden, Suffolk (sandy loam/loamy sand) in 2009/10 and 2011/12

Brockhampton, Herefordshire (sandy loam) in 2009/10 and 2010/11

Woburn, Bedfordshire (sandy loam) in 2010/11 and 2011/12

Field sites were selected that were likely to be responsive to S (i.e. light textured soil in an area of low S deposition and with no recent history of organic material application) and were located on commercial farms at Frostenden and Brockhampton, and on the experimental farm at Woburn run by Rothamsted. At each site the 2 years of field experiments were located on different fields within the same farm.

4.1.2. Experimental treatments and design

At each site, there were 7 organic material treatments:

Autumn applied – Cattle FYM Pig FYM Biosolids (2 treatments) Broiler litter Spring applied – Broiler litter Cattle/pig slurry

The autumn organic material treatments were applied to stubble in the autumn (prior to cultivation and drilling with winter wheat) and the spring organic material treatments were top-dressed to the growing crop. All organic material treatments were applied by hand at a target application rate equivalent to 50 kg/ha total SO₃. ¹

Organic material treatments were compared with inorganic fertiliser S response treatments (0, 12.5, 25, 50 and 75 kg/ha SO₃) to determine the fertiliser S replacement value and hence S availability of the applied organic materials. Fertiliser S was applied in a single dose in early spring as potassium sulphate. There were 4 replicates of each organic material and fertiliser S treatment arranged in a randomised block design.

¹ The target application rate was calculated based on actual organic material analysis, where this was available *before the start of the trial,* or on 'typical' RB209 figures where organic material analysis was not available. There was some variability in the actual organic material SO₃ application rate due to differences between 'typical' and actual organic material analysis. Samples of all organic materials were taken at application and analysed for total SO₃ to calculate actual application rates.

In order to ensure, as far as practically possible, that S was the only limiting nutrient, manufactured fertiliser N was applied at RB209 recommended rates (plus 20 kg/ha N), taking into account supply of crop available N from the organic materials (estimated using MANNER-*NPK*). Similarly, fertiliser P_2O_5 and K_2O were applied at recommended rates based on soil analysis.

4.2. Measurements

Topsoil samples (0-15 cm) were taken from each site at the start of the trial and analysed for pH, extractable P (Olsen's), extractable K and Mg, soil texture and soil organic carbon.

In addition, soil samples were taken in the spring for analysis of extractable SO₃. Soil profile (0–90 cm in 3 depths: 0–30 cm, 30–60 cm and 60–90 cm) samples were taken from the autumn applied organic material treatments and the zero S control treatment. Topsoil (0–15 cm) soil samples were taken from the 2 spring applied organic material treatments at least 1 month following application. Soil samples were anlaysed for extractable SO₃ by extraction with 0.016M KH₂PO₄ (Zhao and McGrath, 1994) and analysis via ICP-AES.

Samples of organic materials were taken at application and analysed for dry matter, total N (Kjeldahl digestion), available N (NH₄-N, NO₃-N and for broiler litter uric acid-N), total S, total P, total K, total Mg and other major/trace elements (aqua regia digest and analysis by ICP-AES) and extractable SO₃ (0.016M KH₂PO₄ extract and analysis via ICP-AES).

At mid flowering, leaf samples were taken from each plot and analysed for total S. Visual symptoms of S deficiency were recorded throughout the growing season at each site. Grain yields (fresh weight) were determined at harvest using a small plot combine. Grain samples were taken and analysed for dry matter, total N and total S. Grain yields (85% DM), grain N:S ratio and grain SO₃ offtake were calculated. Total S in plant tissue was determined by nitric/percholric digest and analysis by ICP-AES; total N in plant tissue was determined by the Dumas combustion method.

4.3. Field experiments data analysis

One-way statistical analysis of variance (ANOVA) was used to evaluate the effect of S fertiliser and organic material treatments on leaf S content, grain yields, grain SO₃ offtake, grain N content and grain N:S ratio. Where ANOVA showed statistically significant differences between treatments (P<0.05), Duncan's multiple comparison test was used to compare individual treatment means.

Where there was a response to S, a response curve was fitted to grain SO₃ offtake data. It was not possible to fit a response curve to yield data at any of the sites because the majority of the yield increase from applied fertiliser S occurred at the first S application rate. Fertiliser S replacement values of the organic material treatments were estimated by comparing S offtake from the organic material treatments with the fitted model to the fertiliser S response plots.

5. Results – field experiments

5.1. Frostenden, Suffolk (2009/10)

The 2009/10 experiment at Frostenden was located on a sandy loam soil, drilled with the winter wheat variety Viscount.

5.1.1. Organic material applications

The autumn organic material treatments were applied to stubble on 27-28/08/2009 and the spring organic material treatments were top-dressed on 26/03/2010. The organic material treatments applied between 46 and 84 kg/ha total SO₃, supplying between 7 and 36 kg/ha extractable SO₃ (Table 15).

Organic material	Application rate Total SO ₃ app ka/ka		Extractable SO ₃					
	t or m ² /na	kg/na	kg/na					
Autumn applied treatments (27-28/08/2009)								
Biosolids – digested cake	5.3	83	13					
Biosolids – lime stabilised	9.0	71	21					
Cattle FYM	27.8	62	7					
Pig FYM	14.7	84	20					
Broiler litter	6.3	64	35					
Spring applied treatments (26/03/2010)								
Broiler litter	6.3	66	36					
Cattle slurry	71.3	46	8					

Table 15. Organic material application rates - Frostenden, Suffolk 2009/10

5.1.2. Soil extractable sulphur

There was no effect (*P*>0.05) of autumn applications of organic materials on extractable SO_3 measured in the soil profile in the spring. Mean soil profile extractable SO_3 (0–90 cm) was 61 kg/ha SO_3 , and decreased with depth from 27 kg/ha SO_3 in 0–30 cm layer, 19 kg/ha SO_3 in the 30–60 cm layer and 15 kg/ha in the 60–90 cm layer (Figure 5).



Figure 5. Soil extractable SO₃ (0–90 cm) on autumn applied treatments – Frostenden, Suffolk 2009/10 (sampled 22/02/10)

In contrast, application of broiler litter and cattle slurry in the spring significantly (P<0.05) increased topsoil (0–15 cm) extractable SO₃ (samples taken on 28/04/2010, approximately 1 month following application) from 13 kg/ha SO₃ (0–15 cm) on the zero S control treatment to 22 and 16 kg/ha (0–15 cm) on the broiler litter and cattle slurry treatments, respectively. Topsoil (0–15 cm) extractable SO₃ from the zero S control treatment (equivalent to 6.7 mg/kg SO₃) was below the generally accepted level for S deficiency in soil of 25 mg/kg SO₃ (10 mg/kg S) (Carver, 2005), indicating a likely crop response to applied S².

5.1.3. Visual symptoms of sulphur deficiency

There were clear visual systems of S deficiency on the zero S control treatment (wheat was yellower and thinner) and clear differences between the treatments (Plate 1). The plots were visually scored for 'greenness' on 29/04/2010 and 04/06/2010; on both dates there was a general increase in 'greenness' with increasing inorganic S fertiliser rate. The autumn organic material treatments were similar in 'greenness' and comparable to between the 0 and 12.5 kg/ha inorganic S fertiliser treatments. The spring organic material treatments were greener than the autumn treatments; the broiler litter treatment was comparable in 'greenness' to the higher inorganic S application rates (25–75 kg/ha SO₃) and the cattle slurry was similar to the 12.5 kg/ha SO₃ inorganic S application rate.

² Soil extractable SO₃ results are converted from mg/kg to kg/ha assuming a soil bulk density of 1.33 g/cm³.



a. Photo taken 28/04/2010

b. Photo taken 28/04/2010





5.1.4. Leaf sulphur content

Leaf samples were taken on 18/06/2010 for analysis of total S content. There was no significant (P>0.05) effect of S fertiliser or organic material applications on leaf S content. There was an indication of increasing leaf S content with S fertiliser application rate (Figure 6) from a mean of c.2700 mg/kg S on the zero S control compared with c.3350 mg/kg S on the 75 kg/ha SO₃ fertiliser application, however, this increase was not statistically significant. Mean leaf S content on all treatments was above the generally accepted level for S deficiency of 2000 mg/kg S, indicating that the crop was not deficient in S.



Figure 6. Leaf S content - Frostenden, Suffolk 2009/10

5.1.5. Grain yields and sulphur offtake

Mean grain yield from the zero S control treatment was 4.0 t/ha (Table 16). Application of S fertiliser did result in greater yields, although this increase was not statistically significant (P>0.05). Where S fertiliser was applied (at application rates of 12 – 75 kg/ha SO₃) yields were between 4.5 and 4.8 t/ha, representing an increase of *c*.0.6 t/ha compared to the zero S control treatment (Table 16 and Figure 7). Yields from the autumn applications of organic materials varied between a mean of 4.0 and 4.4 t/ha and were similar to yields from the zero S control treatment. Yields from the spring applied broiler litter and cattle slurry were greater than from the autumn applications (P>0.05) at 5.6 and 4.6 t/ha from the spring applied broiler litter and cattle slurry treatments, respectively.

Grain S content and grain SO₃ offtake increased (P<0.05) with increasing fertiliser SO₃ application (Table 16). Grain SO₃ offtake increased from a mean of 8.6 kg/ha SO₃ on the zero S control treatment up to a maximum of *c*.12 kg/ha SO₃ on the 50 and 75 kg/ha SO₃ fertiliser treatments. Mean grain SO₃ offtakes on the autumn organic material treatments ranged between 8.4 and 9.1 kg/ha SO₃ and were similar (P>0.05) to SO₃ offtakes from the zero S control treatment. In contrast, application of broiler litter and cattle slurry in the spring did increase grain SO₃ offtake, compared to the zero S control treatment, to 13.5 and 10.0 kg/ha SO₃, respectively; this increase was significant (P<0.05) for the broiler litter, but not cattle slurry treatment.

Treatment	Yield t/ha 85% DM	SO ₃ offtake kg/ha SO ₃	Grain S mg/kg	Grain N %	Grain N:S ratio			
Fertiliser SO₃ respons	e treatments							
0 kg/ha SO₃	4.0	8.6 (ab)	978 (ab)	2.3	24 (cd)			
12 kg/ha SO₃	4.8	10.9 (abcd)	1052 (abcd)	2.1	20 (abc)			
25 kg/ha SO ₃	4.7	11.6 (abcd)	1174 (cde)	2.1	18 (ab)			
50 kg/ha SO₃	4.5	12.1 (cd)	1280 (cd)	2.1	17 (a)			
75 kg/ha SO₃	4.7	11.9 (bcd)	1206 (bcd)	2.1	17 (a)			
Autumn applied organic materials								
Biosolids – digested	4.0	8.8 (abc)	1032 (abc)	2.3	22 (bcd)			
Biosolids – limed	4.3	9.0 (abc)	1013 (abc)	2.3	23 (cd)			
Cattle FYM	4.2	8.4 (a)	926 (a)	2.3	25 (d)			
Pig FYM	4.0	8.6 (ab)	993 (ab)	2.3	24 (cd)			
Broiler litter	4.4	9.1 (abc)	949 (a)	2.1	23 (cd)			
Spring applied organic	c materials							
Broiler litter	5.6	13.5 (d)	1133 (bcde)	1.9	17 (a)			
Cattle slurry	4.6	10.0 (abc)	1001 (ab)	2.0	20 (abc)			
Statistics								
P-value	0.47	<0.01	<0.01	0.19	<0.01			

Note – values followed by different letters in brackets indicate significant differences (P<0.05).





The difference in S response (yield and grain SO_3 offtake) between the autumn and spring applied organic material treatments is likely to be due to the loss of available S from the autumn applied organic materials via overwinter leaching (sulphate like nitrate, is a mobile anion and hence is at risk of loss by leaching). For the spring applied organic materials, yields and SO_3 offtakes were

greater from the broiler litter than from cattle slurry treatment, reflecting the greater quantity of 'extractable' SO_3 supplied by the broiler litter (36 kg/ha 'extractable' SO_3) than cattle slurry (8 kg/ha 'extractable' SO_3) (Table 15).



Figure 8. Grain SO₃ offtake - Frostenden, Suffolk 2009/10

Mean grain N concentration was c.2.2% N, with no differences between treatments (*P*>0.05) (Table 16), indicating that N supply was not limiting on either the organic material or fertiliser treatments. The grain N:S ratio decreased (*P*<0.05) with increasing S fertiliser application rate from 24:1 on the zero S control to 17:1 at the higher fertiliser S application rates (50 and 75 kg/ha SO₃) (Table 16). The threshold value for indicating deficiency in cereals is 17:1 (Carver, 2005); based on this, the zero S control treatment (24:1) and autumn organic material treatments (22:1 - 25:1) would be classed as S deficient, whilst the higher fertiliser S application rates and spring applied broiler litter would be classes as 'normal' (17:1 for both the 50 and 75 kg/ha SO₃ treatment and spring applied broiler litter).

5.1.6. Recovery of sulphur from organic materials

Organic material grain SO₃ offtake (treatment minus control) for the spring applied broiler litter and cattle slurry was equivalent to 3 and 7% of total SO₃ applied, and 17 and 14% of 'extractable' SO₃ applied, respectively (Table 17).

Table 17.	Recovery in	grain of SO ₂	from applied	organic materials	- Frostenden.	Suffolk 2009/10
	INCOUVERY III	grain or oo3	nom applied	organic materials	- i i ostenuen,	Outroik 2003/10

Organic	Sulphur	applied	Grain recovery of SO ₃ from organic materials				
material	Total SO₃ kg/ha	Extractable SO₃ kg/ha	kg/ha	% total SO₃ applied	% extractable SO ₃ applied		
Spring applied organic materials							
Broiler litter	66	36	4.9	3	17		
Cattle slurry	46	8	1.4	7	14		

5.1.7. Fertiliser sulphur replacement value of applied organic materials

The spring applied cattle slurry had a SO₃ fertiliser replacement value of 7.5 kg/ha SO₃,

representing an efficiency of 16% of total SO₃ applied and 92% of 'extractable' SO₃ applied (Table 18). It was not possible to calculate a SO₃ replacement value for the spring applied broiler litter as the grain SO₃ offtake (13.5 kg/ha SO₃) was greater than the maximum SO₃ offtake on the fertiliser treatments.

	Sulphur	applied	Fertiliser SO ₃ replacement value				
Organic material	Total SO₃ kg/ha	Extractable SO₃ kg/ha	Extractable kg/ha SO ₃ kg/ha		Efficiency % extractable SO ₃ applied**		
Spring applied organic materials							
Broiler litter	66	36	>max	>max	>max		
Cattle slurry	46	8	7.5	16	92		

Table 18. Fertiliser SO₃ replacement value of applied organic materials - Frostenden, Suffolk 2009/10

* Fertiliser replacement value (kg/ha) as a percentage of total SO₃ applied in the organic material.

** Fertiliser replacement value (kg/ha) as a percentage of 'extractable' SO₃ applied in the organic material.

5.2. Frostenden, Suffolk (2011/12)

The 2011/12 experiment at Frostenden was located on a loamy sand soil, drilled with the winter wheat variety Viscount.

5.2.1. Organic material applications

The autumn organic material treatments were applied to stubble on 11/08/2011 and the spring organic material treatments were top-dressed on 28/03/2012. These treatments applied between 55 and 99 kg/ha total SO₃, supplying between 9 and 42 kg/ha extractable SO₃ (Table 19).

Organic material	Application rate t or m³/ha	Total SO₃ applied kg/ha	Extractable SO ₃ kg/ha			
Autumn applied treatments (11/08/2011)						
Biosolids – digested cake	7.6	55	9			
Biosolids – lime stabilised	8.0	69	20			
Cattle FYM	25.0	86	14			
Pig FYM	15.0	77	20			
Broiler litter	6.3	67	41			
Spring applied treatments (28/03/2012)						
Broiler litter	6.3	68	42			
Pig slurry	52.5	99	21			

Table 19. Organic material application rates - Frostenden, Suffolk 2011/12

5.2.2. Soil extractable sulphur

Soil profile (0–90 cm) extractable SO₃ was greater where organic material had been applied in the autumn (between 73 and 86 kg/ha SO₃) compared to the zero S control treatment (56 kg/ha SO₃), although this difference was not statistically significant (P>0.05). Soil profile extractable SO₃ was similar in the 0–30 cm layer across all treatments, but was greater in the 30–60 cm and 60–90 cm layers from the autumn organic material treatments compared to the zero S control (Figure 9), which may indicate some overwinter leaching of applied S down the soil profile, although these differences were not statistically significant (P>0.05).



Figure 9. Soil extractable SO₃ (0–90 cm) on autumn applied treatments – Frostenden, Suffolk 2011/12 (sampled $\frac{28}{02}$

The application of broiler litter and pig slurry in the spring significantly (P<0.05) increased topsoil (0–15 cm) extractable SO₃ (samples taken on 01/06/2012, approximately 2 months following

application) from 8 kg/ha SO₃ (0–15 cm) on the zero S control treatment, to 12 and 14 kg/ha (0-15 cm) on the broiler litter and cattle slurry treatments, respectively. Topsoil (0–15 cm) extractable SO₃ from the zero S control treatment (equivalent to 3.8 mg/kg SO₃) was below the generally accepted level for S deficiency in soil of 25 mg/kg SO₃ (10 mg/kg S) (Carver, 2005), indicating a likely crop response to applied S.

5.2.3. Visual symptoms of sulphur deficiency

The zero S control treatment showed no visual symptoms of S deficiency and there were no apparent visual differences in 'greenness' between the different S fertiliser treatments.

5.2.4. Leaf sulphur content

Leaf samples were taken on 29/06/2012 for analysis of total S content. Although there were no visible differences in 'greenness' between the S fertiliser treatments, there was a significant (P<0.05) increase in leaf S content with application of S fertiliser, from 1570 mg/kg S on the zero S control (below the generally accepted level for S deficiency of 2000 mg/kg S), up to *c*.2800 mg/kg S on the 75 kg/ha SO₃ treatment (Figure 10). Leaf S content on the autumn applied organic material treatments varied between *c*.1700 and 2100 mg/kg and was not significantly (P>0.05) different from the zero S control. In contrast, application of broiler litter and pig slurry in the spring significantly (P<0.05) increased leaf S content to *c*.2800 and 3000 mg/kg S, respectively.



Figure 10. Leaf S content - Frostenden, Suffolk 2011/12

5.2.5. Grain yields and sulphur offtake

Mean grain yields in the zero S control treatment were 7.6 t/ha. Application of S fertiliser did result in a small (c.0.2 t/ha) increase in grain yield, although this increase was not statistically significant (P>0.05) (Table 20). There was no effect (P>0.05) of organic material applications on grain yield.

Grain S content and grain SO₃ offtake increased (P<0.05) with fertiliser SO₃ application (Table 20 and Figure 11). Grain SO₃ offtake increased from a mean of 14 kg/ha SO₃ on the zero S control treatment up to a maximum of *c*.19 kg/ha SO₃ on the 25 to 75 kg/ha SO₃ fertiliser treatments. Grain SO₃ offtake from the autumn applied cattle FYM, at 14 kg/ha SO₃, was the same as from the zero S control treatment. There was an increase in grain SO₃ offtake from the other autumn applied organic materials, to between 16 and 18 kg/ha SO₃, although this increase was only significant (P<0.05) for the limed biosolids treatment. The increase in grain SO₃ offtake from the autumn applied organic materials is in contrast to the results from the 2009/10 trial at Frostenden, where there was no increase in grain SO₃ offtake from any of the autumn applied treatments. The overwinter period in 2009/10 was much wetter (*c*.170 mm overwinter drainage) than the 2011/12 overwinter period (*c*.40 mm overwinter drainage), and it is likely that the greater overwinter drainage resulted in increased leaching of available S in 2009/10.

Treatment	Yield	SO ₃ offtake	Grain S	Grain N	Grain N:S		
	t/ha 85% DM	kg/ha SO₃	mg/kg	%	ratio		
Fertiliser SO ₃ response	e treatments						
0 kg/ha SO ₃	7.6	14 (a)	884 (a)	1.9	22 (e)		
12 kg/ha SO₃	7.8	17 (bc)	1044 (abc)	1.9	18 (abc)		
25 kg/ha SO₃	7.7	19 (bc)	1151 (bc)	2.0	17 (abc)		
50 kg/ha SO₃	7.9	19 (bc)	1122 (bc)	1.9	17 (ab)		
75 kg/ha SO $_3$	7.8	19 (bc)	1131 (bc)	1.9	17 (a)		
Autumn applied organ	Autumn applied organic materials						
Biosolids – digested	7.7	16 (ab)	997 (ab)	1.9	19 (bcd)		
Biosolids – limed	7.9	18 (bc)	1094 (bc)	2.0	18 (abc)		
Cattle FYM	7.4	14 (a)	909 (a)	2.0	22 (e)		
Pig FYM	7.7	16 (ab)	988 (ab)	2.0	21 (de)		
Broiler litter	7.6	17 (ab)	1025 (abc)	2.0	19 (cd)		
Spring applied organic materials							
Broiler litter	7.8	20 (c)	1202 (c)	2.1	18 (abc)		
Pig slurry	7.7	19 (bc)	1167 (bc)	2.0	17 (ab)		
Statistics							
<i>P</i> -value	0.14	<0.01	<0.01	0.93	<0.01		

Table 20. Grain yields, SO₃ offtake and grain S and N content - Frostenden, Suffolk 2011/12

Note – values followed by different letters in brackets indicate significant differences (P<0.05).

Application of broiler litter and pig slurry in the spring significantly (P<0.05) increased grain SO₃ offtake compared to the zero S control to 20 and 19 kg/ha SO₃ for the broiler litter and pig slurry treatments, respectively. Comparison of the autumn and spring applied broiler litter treatments showed higher SO₃ offtake from the spring (20 kg/ha SO₃), compared to autumn applied (17 kg/ha SO₃) broiler litter. Although this difference was not statistically significant (P>0.05), it is consistent

with the results from 2009/10 at Frostenden and is likely to be due to loss of available S from the autumn applied broiler litter via overwinter leaching.



Figure 11. Grain SO₃ offtake - Frostenden, Suffolk 2011/12

Grain N content was the same across all treatments at c.2.0% N (P>0.05) (Table 20), indicating that N supply was not limiting on either the organic material or fertiliser treatments. The grain N:S ratio decreased (P<0.05) with increasing S fertiliser application rate from 22:1 on the zero S control to 17:1 at the 25 to 75 kg/ha SO₃ fertiliser application rates (Table 20). The zero S control treatment and autumn pig and cattle FYM treatments were clearly S deficient (N:S ratio 21:1 – 22:1).

5.2.6. Recovery of sulphur from organic materials

Grain recovery of the additional SO₃ applied in the materials ranged between 0 and 8% of total SO₃ applied and between 0 and 23% of 'extractable' SO₃ applied (Table 21).

Organic	Sulphur applied		Grain recovery	Frain recovery of SO₃ from organ	
material	Total SO₃ kg/ha	Extractable SO₃ kg/ha	kg/ha	% total SO₃ applied	% extractable SO ₃ applied
Autumn applied organic materials					
Biosolids –					
digested	55	9	1.8	3	20
Biosolids –					
limed	69	20	4.1	6	20
Cattle FYM	86	14	0.0	0	0
Pig FYM	77	20	1.9	2	9
Broiler litter	67	41	2.3	3	6
Spring applied organic materials					
Broiler litter	68	42	5.7	8	14
Pig slurry	99	21	4.7	5	23

Table 21. Recovery in grain of SO₃ from applied organic materials - Frostenden, Suffolk 2011/12

5.2.7. Fertiliser sulphur replacement value of applied organic materials

The autumn applied organic materials (excluding cattle FYM), had fertiliser SO₃ replacement values of between 7 and 20 kg/ha SO₃, representing efficiencies relative to fertiliser SO₃ of up to 29% of total SO₃ applied and up to 99% of 'extractable' SO₃ applied (Table 22).

The spring applied pig slurry had a SO₃ fertiliser replacement value of 26 kg/ha SO₃, representing an efficiency of 27% of total SO₃ applied and >100% of 'extractable' SO₃ applied (Table 22). It was not possible to calculate a SO₃ replacement value for the spring applied broiler litter as the grain SO₃ offtake from the spring broiler litter treatment (20 kg/ha SO₃) was greater than the maximum SO₃ offtake from the fertiliser SO₃ response treatments.

Applications of organic materials in the spring were the most effective at supplying SO_3 to the crop, as the SO_3 was applied when the crop was growing and was not subject to overwinter leaching loss.

	Sulphur applied		Fertiliser SO ₃ replacement value				
Organic material	Total SO₃ kg/ha	Extractable SO₃ kg/ha	kg/ha	Efficiency % total SO₃ applied*	Efficiency % extractable SO ₃ applied**		
Autumn applie	Autumn applied organic materials						
Biosolids –							
digested	55	9	7.4	14	80		
Biosolids –							
limed	69	20	20.2	29	99		
Cattle FYM	86	14	0	0	0		
Pig FYM	77	20	7.5	10	38		
Broiler litter	67	41	9.3	14	23		
Spring applied organic materials							
Broiler litter	68	42	>max	>max	>max		
Pig slurry	99	21	26.3	27	125		

					• ··· ·· ···
Table 22 Fertiliser	r SO, renlacement	value of applied	organic materials	- Frostenden	Suffolk 2011/12
		value of applied	organio materiais	riostenacii,	

* Fertiliser replacement value (kg/ha) as a percentage of total SO₃ applied in the organic material.

** Fertiliser replacement value (kg/ha) as a percentage of 'extractable' SO₃ applied in the organic material.

5.3. Brockhampton, Herefordshire (2009/10)

The 2009/10 experiment at Brockhampton was located on a sandy loam soil, drilled with the winter wheat variety Alchemy.

5.3.1. Organic material applications

The autumn organic material treatments were applied to stubble on 15/09/2009 and the spring organic material treatments were top-dressed on 22-23/03/2010. The organic material treatments applied between 38 and 79 kg/ha total SO₃, supplying between 7 and 34 kg/ha extractable SO₃ (Table 23).

Organic material	Application rate t or m³/ha	Total SO₃ applied kg/ha	Extractable SO₃ kg/ha				
Autumn applied treatments (15	Autumn applied treatments (15/09/2009)						
Biosolids – digested cake							
(Barnhurst)	6.9	52	7				
Biosolids – lime stabilised							
(Rushmoor)	8.7	75	16				
Cattle FYM	27.8	60	6				
Pig FYM	14.7	79	17				
Broiler litter	6.3	47	24				
Spring applied treatments (22-23/03/2010)							
Broiler litter	6.3	59	34				
Pig slurry	72.7	38	9				

Table 23. Organic material	application rates -	Brockhampton,	Herefordshire	2009/10

5.3.2. Soil extractable sulphur

There was no effect (*P*>0.05) of autumn applications of organic materials on soil profile extractable SO_3 measured in the spring. Soil profile extractable SO_3 (0–90 cm) was a mean of 68 kg/ha SO_3 , and, in contrast to the Frostenden and Woburn sites, increased with depth from a mean of 19 kg/ha SO_3 in 0–30 cm layer, to 22 kg/ha SO_3 in the 30–60 cm layer and to 27 kg/ha in the 60–90 cm layer (Figure 12).



Figure 12. Soil extractable SO₃ (0-90 cm) on autumn applied treatments – Brockhampton, Herefordshire 2009/10 (sampled 09/03/2010)

Spring applications of broiler litter and pig slurry increased topsoil (0–15 cm) extractable SO₃ (samples taken on 28/04/2010, approximately 1 month following application) from 25 kg/ha SO₃ (0–15 cm) on the zero S control treatment, to 33 and 36 kg/ha (0–15 cm) on the broiler litter and pig slurry treatments, respectively; this increase was significant (P<0.05) for the pig slurry, but not broiler litter treatment. Topsoil (0–15 cm) extractable SO₃ from the zero S control treatment (equivalent to 12.4 mg/kg SO₃) was below the generally accepted level for S deficiency in soil of 25 mg/kg SO₃ (10 mg/kg S) (Carver, 2005), indicating a likely crop response to applied S.

5.3.3. Visual symptoms of sulphur deficiency

There were slight symptoms of S deficiency (yellower plants) apparent on the zero S control treatment, although these were not as clear as those at Frostenden in the same year and did not show clearly in photographs. Plots were scored for 'greenness' on 10/05/2010; all S fertiliser and organic material treatments were 'greener' than the zero S control treatment, although there was no apparent visual effect of increasing S fertiliser application rate or organic material treatment on 'greenness'.

5.3.4. Leaf sulphur content

There was no significant (P>0.05) effect of either SO₃ fertiliser or organic material treatments on leaf S content (Figure 13). Mean leaf S content on all treatments was above the generally accepted level for S deficiency of 2000 mg/kg S, indicating that the crop was not deficient in S and a yield response was unlikely.



Figure 13. Leaf S content - Brockhampton, Hereford 2009/10

5.3.5. Grain yields and sulphur offtake

There was no effect of S fertiliser application or applications of organic materials on grain yields, grain S content or grain SO₃ offtake (P>0.05) (Table 24). Grain N content was in the range 2.2–2.3% N (Table 24), indicating that N supply was not limited on either the organic material or

Treatment	Yield t/ha 85% DM	SO₃ offtake kg/ha SO₃	Grain S mg/kg	Grain N %	Grain N:S ratio			
Fertiliser SO3 respons	Fertiliser SO3 response treatments							
0 kg/ha SO₃	8.2	23	1328	2.3 (ab)	17			
12 kg/ha SO₃	8.6	23	1289	2.2 (a)	17			
25 kg/ha SO ₃	8.8	24	1296	2.2 (ab)	17			
50 kg/ha SO₃	8.1	24	1400	2.3 (b)	16			
75 kg/ha SO₃	8.4	25	1396	2.3 (ab)	16			
Autumn applied organ	ic materials							
Biosolids – digested	8.1	21	1217	2.2 (a)	18			
Biosolids – limed	8.8	25	1324	2.2 (ab)	17			
Cattle FYM	8.8	23	1246	2.2 (a)	17			
Pig FYM	8.9	25	1338	2.2 (ab)	17			
Broiler litter	9.0	25	1329	2.2 (ab)	17			
Spring applied organic materials								
Broiler litter	8.4	24	1320	2.3 (b)	18			
Pig slurry	9.0	26	1358	2.3 (b)	17			
Statistics	Statistics							
P-value	0.46	0.44	0.09	0.02	0.41			

Table 04 Orain	violate CO effe	alea and anala C and	IN content Drockham	where I leveler debine 2000/	4 0
Table 24. Grain	vielas. SU ₂ om	ake and drain 5 and	1 N CONTENT - Brocknam	noton. Heretorasnire 2009/1	ιu
	J.C.C., CC3 C	and and grain e and			•••

Note – values followed by different letters in brackets indicate significant differences (P<0.05).

fertiliser treatments. The relatively high grain S content on the zero S control treatment (1328 mg/kg S) and N:S ratio of 17:1 indicate that this wheat crop was not deficient in S.

5.4. Brockhampton, Herefordshire (2010/11)

The 2010/11 experiment at Brockhampton was located on a sandy loam soil, drilled with the winter wheat variety Panorama.

5.4.1. Organic material applications

The autumn organic material treatments were applied to stubble on 23/09/2010 and the spring organic material treatments were top-dressed on 12/04/2011. The organic material treatments applied between 31 and 128 kg/ha total SO₃, supplying between 6 and 25 kg/ha extractable SO₃ (Table 25).

Organic material	Application rate t or m ³ /ha	Total SO₃ applied kg/ha	Extractable SO ₃ kg/ha			
Autumn applied treatments (23/09/2010)						
Biosolids – digested cake	7.6	48	6			
Biosolids – lime stabilised	8.0	73	21			
Cattle FYM	27.8	128	21			
Pig FYM	14.7	51	19			
Broiler litter	6.3	31	12			
Spring applied treatments (12/04/2011)						
Broiler litter	6.3	46	25			
Pig slurry	73.0	34	16			

Table 25. Organic material application rates - Brockhampton, Herefordshire 2010/11

5.4.2. Soil extractable sulphur

There was no effect (P>0.05) of autumn applications of organic materials on soil profile extractable SO₃ measured in the spring. Mean soil profile extractable SO₃ (0–90 cm) was 91 kg/ha SO₃, with 28 kg/ha SO₃ in 0–30 cm layer, 34 kg/ha SO₃ in the 30–60 cm layer and 29 kg/ha in the 60–90 cm layer (Figure 14).



Figure 14. Soil extractable SO₃ (0–90 cm) on autumn applied treatments – Brockhampton, Herefordshire 2010/11 (sampled 10/03/2011)

The application of broiler litter and pig slurry in the spring significantly (P<0.05) increased topsoil (0–15 cm) extractable SO₃ (samples taken on 11/05/2011, approximately 1 month following application) from 19 kg/ha SO₃ (0–15 cm) on the zero S control treatment, to 35 and 58 kg/ha (0–15 cm) on the broiler litter and pig slurry treatments, respectively. Topsoil (0–15 cm) extractable SO₃ from the zero S control treatment (equivalent to 9.5 mg/kg SO₃) was below the generally accepted level for S deficiency in soil of 25 mg/kg SO₃ (10 mg/kg S) (Carver, 2005), indicating a likely crop response to applied S.

5.4.3. Visual symptoms of sulphur deficiency

The zero S control treatment showed no visual symptoms of S deficiency and there were no apparent visual differences in 'greenness' between the different S fertiliser and organic material treatments.

5.4.4. Leaf sulphur content

Leaf samples were taken on 30/06/2011 for analysis of total S content. Although there were no visual differences between the different S fertiliser treatments, there was a significant (P<0.05) increase in leaf S content with fertiliser S application rate; from 2550 mg/kg S on the zero S control to *c*.3400 mg/kg S on the 50 and 75 kg/ha SO₃ fertiliser treatments (Figure 15). There was no effect (P>0.05) of any of the organic material treatments on leaf S content, and mean leaf S content on all treatments was above the generally accepted level for S deficiency of 2000 mg/kg S, indicating that the crop was not deficient in S and a yield response was unlikely.



Figure 15. Leaf S content - Brockhampton, Herefordshire 2010/11

5.4.5. Grain yields and sulphur offtake

There was no effect of S fertiliser application or applications of the organic materials on grain yields, grain S content or grain SO₃ offtake (P>0.05) (Table 26). There was a small, but significant (P<0.05) increase in grain S content with increasing S fertiliser rate, from 1225 mg/kg S on the zero S control up to *c*.1320 mg/kg S on the 50 and 75 kg/ha SO₃ fertiliser rates; this was reflected in a small, but not significant (P>0.05) increase in grain SO₃ offtake from 28 kg/ha SO₃ on the zero S control up to 31 kg/ha on the 50 and 75 kg/ha SO₃ fertiliser rates (Table 26).

Grain N content ranged between 2.1–2.3% N (Table 26), indicating that N supply was not limited on either the organic material or fertiliser treatments. The relatively high grain S content on the zero S control treatment (1225 mg/kg S) and N:S ratio of 18:1 indicate that the site was unlikely to be deficient in S.

Table 26. Grain yields, SO₃ offtake and grain S and N content - Brockhampton, Herefordshire 2010/11

Treatment	Yield	SO ₃ offtake	Grain S	Grain N	Grain N:S		
	t/ha 85% DM	kg/ha SO₃	mg/kg	%	ratio		
Fertiliser SO₃ respons	Fertiliser SO3 response treatments						
0 kg/ha SO ₃	10.8	28	1225 (a)	2.2	18		
12 kg/ha SO₃	10.8	28	1235 (a)	2.1	17		
25 kg/ha SO ₃	10.2	27	1259 (ab)	2.2	18		
50 kg/ha SO₃	11.0	31	1321 (b)	2.2	17		
75 kg/ha SO₃	10.9	31	1320 (b)	2.2	17		
Autumn applied organ	ic materials						
Biosolids – digested	10.5	28	1252 (ab)	2.2	17		
Biosolids – limed	11.4	30	1255 (ab)	2.2	17		
Cattle FYM	11.0	30	1281 (ab)	2.2	17		
Pig FYM	11.0	30	1286 (ab)	2.3	18		
Broiler litter	11.2	30	1235 (a)	2.2	18		
Spring applied organic materials							
Broiler litter	10.8	30	1323 (b)	2.3	18		
Pig slurry	11.2	30	1260 (ab)	2.2	17		
Statistics	Statistics						
P-value	0.27	0.11	0.05	0.08	0.32		

Note – values followed by different letters in brackets indicate significant differences (P<0.05).

5.5. Woburn, Bedfordshire (2010/11)

The 2010/11 experiment at Woburn was located on a sandy loam/loamy sand soil, drilled with the winter wheat variety Oakley.

5.5.1. Organic material applications

The autumn organic material treatments were applied to stubble on 07/10/2010 and the spring organic material treatments were top-dressed on 19/04/2011. The organic material treatments applied between 29 and 75 kg/ha total SO₃, supplying between 3 and 50 kg/ha extractable SO₃ (Table 27).

Organic material	Application rate t or m ³ /ha	Total SO₃ applied kg/ha	Extractable SO₃ kg/ha			
Autumn applied treatments (07/10/2010)						
Biosolids – digested cake	5.4	48	16			
Biosolids – lime stabilised	9.7	75	16			
Cattle FYM	20.0	29	3			
Pig FYM	13.2	60	12			
Broiler litter	6.3	59	38			
Spring applied treatments (19/04/2011)						
Broiler litter	6.3	73	50			
Cattle slurry	25.0	68	41			

Table 27	Organic material	application r	ates - Wohurn	Bodfordshire 2010/11	
I able Z1.	Organic material	application	ales - woburn,	Deutorustille 2010/11	

5.5.2. Soil extractable sulphur

Soil profile extractable SO₃ (0–90 cm) content was similar on the zero S control treatment (38 kg/ha SO₃) and autumn livestock manure treatments (34 kg/ha SO₃ on the cattle FYM and 40 kg/ha SO₃ on both the pig FYM and broiler litter treatments). Soil profile extractable SO₃ (0–90 cm) was higher on the autumn biosolids treatments (48 and 52 kg/ha SO₃ on the digested and limed biosolids treatments, respectively), although this increase was not statistically significant (*P*>0.05). Soil extractable SO₃ (0–90 cm) was 14 kg/ha SO₃ in 0–30 cm layer, 15 kg/ha SO₃ in the 30–60 cm layer and 13 kg/ha in the 60–90 cm layer (Figure 16).



Figure 16. Soil extractable SO₃ (0-90 cm) on autumn applied treatments – Woburn, Bedfordshire 2010/11 (sampled 15/03/2012)

The application of broiler litter and cattle slurry in the spring significantly (P<0.05) increased topsoil (0-15 cm) extractable SO₃ (samples taken on 24/05/2011, approximately 1 month following application) from 6 kg/ha SO₃ (0–15 cm) on the zero S control treatment, to 12–13 kg/ha (0–15 cm) on the broiler litter and cattle slurry treatments. Topsoil (0–15 cm) extractable SO₃ from the zero S control treatment (equivalent to 3.1 mg/kg SO₃) was below the generally accepted level for S deficiency in soil of 25 mg/kg SO₃ (10 mg/kg S) (Carver, 2005), indicating a likely crop response to applied S.

5.5.3. Visual symptoms of sulphur deficiency

There were clear visual systems of S deficiency on the zero S control treatment; the wheat was yellow, stunted and thin (Plate 2). In contrast, at the higher fertiliser S application rates (50 and 75 kg/ha SO_3), the wheat was notably greener and taller than on the zero S control. The plots were visually assessed for 'greenness' and symptoms of S deficiency on 24/05/11. Of the organic

material treatments, the autumn broiler litter, both autumn biosolids and spring cattle slurry treatments were greener and thicker than the autumn cattle and pig FYM and spring broiler litter treatments. The visual assessment was carried out approximately 1 month after the spring organic material applications and it was noted that the broiler litter was still visible on the soil surface; as the intervening period between application and visual assessments had been very dry (24 mm rain), it is possible that the applied S remained in the broiler litter largely at the soil surface.



a. Zero S control showing symptoms of S deficiency. b. Photo taken 24/05/11 Photo taken 24/05/11.

Plate 2. Visual response to sulphur treatments – Woburn, Bedfordshire 2010/11

5.5.4. Leaf sulphur content

Leaf samples were taken on 23/06/2011 for analysis of total S content. There was a large and significant (P<0.05) increase in leaf S content with application of S fertiliser, from *c*.1250 mg/kg S on the zero S control (below the generally accepted level for S deficiency of 2000 mg/kg S), up to *c*.6000 mg/kg S on the 75 kg/ha SO₃ treatment (Figure 17). Leaf S content on the autumn applied organic material treatments varied between *c*.1400 and 2300 mg/kg and was not significantly (P>0.05) different from the zero S control. In contrast, application of broiler litter and cattle slurry in the spring significantly (P<0.05) increased leaf S content up to *c*.5000 mg/kg S.



Figure 17. Leaf S content - Woburn, Bedfordshire 2010/11

5.5.5. Grain yields and sulphur offtake

Mean grain yields from the zero S control treatment were 3.2 t/ha. Application of S fertiliser did result in greater yields (*P*=0.06). Where S fertiliser was applied, yields ranged between 4.2 and 4.7 t/ha, representing a mean increase of *c*.1.2 t/ha compared to the zero S control treatment (Table 28 and Figure 18). Grain yields from the autumn applied cattle FYM, at 3.3 t/ha, were comparable to yields from the zero S control treatment. However, application of pig FYM, broiler litter and 2 biosolids products in the autumn increased grain yields to between 4.0 and 4.5 t/ha, representing an increase of 0.8–1.3 t/ha compared to the zero S control. Yields from the spring applied broiler litter and cattle slurry were greater than from the autumn applications at 4.8 and 5.0 t/ha from the spring applied broiler litter and cattle slurry treatments, respectively, equivalent to a 1.6–1.8 t/ha yield increase compared to the zero S fertiliser control.

Treatment	Yield t/ha 85% DM	SO ₃ offtake kg/ha SO ₃	Grain S mg/kg	Grain N %	Grain N:S ratio		
Fertiliser SO3 respons	Fertiliser SO ₃ response treatments						
0 kg/ha SO₃	3.2	5 (a)	716 (a)	2.4 (bc)	34 (f)		
12 kg/ha SO₃	4.4	10 (cd)	1050 (c)	2.3 (ab)	22 (bc)		
25 kg/ha SO $_3$	4.7	11 (de)	1144 (d)	2.3 (abc)	21 (ab)		
50 kg/ha SO ₃	4.2	12 (de)	1310 (ef)	2.4 (bc)	19 (a)		
75 kg/ha SO₃	4.4	13 (e)	1377 (f)	2.5 (c)	18 (a)		
Autumn applied organic materials							
Biosolids – digested	4.5	9 (bc)	886 (b)	2.2 (a)	25 (d)		
Biosolids – limed	4.4	9 (cd)	985 (c)	2.3 (ab)	24 (cd)		
Cattle FYM	3.3	5 (a)	693 (a)	2.4 (abc)	34 (f)		
Pig FYM	4.0	7 (ab)	771 (a)	2.3 (ab)	30 (e)		
Broiler litter	4.2	8 (bc)	871 (b)	2.3 (ab)	26 (d)		
Spring applied organic materials							
Broiler litter	4.8	13 (e)	1252 (e)	2.3 (abc)	19 (a)		
Cattle slurry	5.0	14 (e)	1325 (ef)	2.4 (bc)	18 (a)		
Statistics							
<i>P</i> -value	0.06	<0.01	<0.01	0.04	<0.01		

Note – values followed by different letters in brackets indicate significant differences (P<0.05).





Grain S content and grain SO₃ offtake increased (P<0.05) with fertiliser SO₃ application (Table 28 and Figure 19). Grain S content almost doubled from 716 mg/kg S on the zero S control treatment to 1377 mg/kg S on the 75 kg/ha SO₃ fertiliser treatment and was reflected in greater grain SO₃ offtake (5 kg/ha SO₃ on the zero S control compared with 13 kg/ha SO₃ on the 75 kg/ha SO₃

fertiliser rate). Grain SO₃ offtake from the autumn applied cattle FYM, at 5 kg/ha SO₃, was the same as from the zero S control treatment. There was an increase in grain SO₃ offtake from the other autumn applied organic materials, to between 7 and 9 kg/ha SO₃; this increase was statistically significant (P<0.05) for the broiler litter and two biosolids treatments, but not for the pig FYM treatment. The lower yields and grain SO₃ offtake from the autumn applied cattle FYM reflects the lower proportion of total S in the 'extractable' form in the cattle FYM and hence lower application rate of 'extractable' SO₃ (3 kg/ha 'extractable' SO₃ applied) compared to the other autumn applied organic materials (12–38 kg/ha 'extractable' SO₃ applied) (Table 27).

Grain SO₃ offtake from the spring applied broiler litter and cattle slurry treatments was 13 and 14 kg/ha SO₃, respectively, and was comparable to the grain SO₃ offtake measured from the highest S fertiliser (75 kg/ha SO₃) rate. The greater yields and grain SO₃ offtakes from the spring compared to autumn applied organic materials (P<0.05), is consistent with the results from the Frostenden site and is likely to be due to some loss of available S from the autumn applied organic materials via overwinter leaching.





Grain N content was between 2.2 and 2.5 % N across all treatments (Table 28), indicating that N supply was not limited on either the organic material or fertiliser treatments. The grain N:S ratio decreased (P<0.05) with increasing S fertiliser application rate from 34:1 on the zero S control to 18:1 on the 75 kg/ha SO₃ fertiliser application rate (Table 28). The zero S control treatment, the lower S fertiliser application rates (12 and 25 kg/ha SO₃ fertiliser) and all autumn organic material treatments were clearly S deficient (N:S ratio > 21:1).

5.5.6. Recovery of sulphur from organic materials

For the autumn applied organic materials, grain recovery of SO₃ varied between 1 and 8% of total SO₃ applied and between 5 and 26% of 'extractable' SO₃ applied (Table 29). For the spring applied broiler litter and cattle slurry, grain recovery of SO₃ was 11 and 13% of total SO₃ applied, and 16 and 22% of 'extractable' SO₃ applied, respectively (Table 29).

As at Frostenden in 2011/12, the spring applications of organic materials were the most effective at supplying SO_3 to the crop, as the SO_3 was applied when the crop was growing and was not subject to overwinter leaching loss.

Organia	Sulphur applied		Grain recovery of SO ₃ from organic materials			
material	Total SO₃ kg/ha	Extractable SO₃ kg/ha	kg/ha	% total SO₃ applied	% extractable SO ₃ applied	
Autumn applie	ed organic mate	erials				
Biosolids –						
digested	48	16	3.9	8	24	
Biosolids –						
limed	75	16	4.1	5	26	
Cattle FYM	29	3	0.2	1	5	
Pig FYM	60	12	1.6	3	14	
Broiler litter	59	38	2.8	5	7	
Spring applied organic materials						
Broiler litter	73	50	7.8	11	16	
Cattle slurry	68	41	8.9	13	22	

Table 29. Recovery in grain of SO₃ from applied organic materials - Woburn, Bedfordshire 2010/11

5.5.7. Fertiliser sulphur replacement value of applied organic materials

The autumn applied organic materials had fertiliser SO_3 replacement values of between 0 and 10 kg/ha SO_3 , representing efficiencies relative to fertiliser SO_3 of up to 20% of total SO_3 applied and up to 63% of 'extractable' SO_3 applied (Table 30).

The spring applied pig slurry had a SO₃ fertiliser replacement value of 70 kg/ha SO₃, representing an efficiency of 96% of total SO₃ applied and >100% of 'extractable' SO₃ applied (Table 30). It was not possible to calculate a SO₃ replacement value for the spring applied cattle slurry as the grain SO₃ offtake from the spring cattle slurry treatment (14 kg/ha SO₃) was greater than the maximum SO₃ offtake from the fertiliser SO₃ response treatments. Table 30. Fertiliser SO₃ replacement value of applied organic materials - Woburn, Bedfordshire 2010/11

	Sulphur applied		Fertiliser SO ₃ replacement value			
Organic material	Total SO₃ kq/ha	Extractable SO₃ kq/ha	kg/ha	Efficiency % total SO ₃	Efficiency % extractable	
	9	° 0		applied*	SO ₃ applied**	
Autumn applie	ed organic mate	erials				
Biosolids –						
digested	48	16	9.6	20	58	
Biosolids –						
limed	75	16	10.0	13	63	
Cattle FYM	29	3	0.4	1	13	
Pig FYM	60	12	4.0	7	34	
Broiler litter	59	38	6.9	12	18	
Spring applied organic materials						
Broiler litter	73	50	69.7	96	140	
Cattle slurry	68	41	>max	>max	>max	

* Fertiliser replacement value (kg/ha) as a percentage of total SO₃ applied in the organic material.

** Fertiliser replacement value (kg/ha) as a percentage of 'extractable' SO₃ applied in the organic material.

5.6. Woburn, Bedfordshire (2011/12)

The 2011/12 experiment at Woburn was located on a sandy loam/loamy sand soil, drilled with the winter wheat variety Oakley.

5.6.1. Organic material applications

The autumn organic material treatments were applied to stubble on 22/09/2011 and the spring organic material treatments were top-dressed on 24/04/2012. The organic material treatments applied between 59 and 121 kg/ha total SO_3 , supplying between 16 and 47 kg/ha extractable SO_3 (Table 31).

Table 31. Organic material application rates	- Woburn, Bedfordshire 2011/12
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Organic material	Application rate t or m ³ /ha	Total SO₃ applied kɑ/ha	Extractable SO ₃ kg/ha				
Autumn applied treatments (22/09/2011)							
Biosolids – digested cake	4.4	85	19				
Biosolids – lime stabilised	6.8	77	16				
Cattle FYM	28.9	114	17				
Pig FYM	10.5	121	28				
Broiler litter	6.3	77	47				
Spring applied treatments (24/04/2012)							
Broiler litter	7.9	59	34				
Pig slurry	50.0	87	40				

5.6.2. Soil extractable sulphur

There was no effect (*P*>0.05) of autumn applications of organic materials on soil profile extractable SO_3 measured in the spring. Mean soil profile extractable SO_3 (0–90 cm) was 97 kg/ha SO_3 , and increased from the topsoil to subsoil, with 25 kg/ha SO_3 in 0–30 cm layer, 36 kg/ha SO_3 in the 30–60 cm layer and 36 kg/ha in the 60–90 cm layer (Figure 20).



Figure 20. Soil extractable SO₃ (0-90 cm) on autumn applied treatments - Woburn, Bedfordshire 2011/12 (sampled 15/03/2012)

There was no effect (*P*>0.05) of spring broiler litter or pig slurry applications on topsoil (0–15 cm) extractable SO₃ (samples taken on 29/05/2012, approximately 1 month following application). Topsoil (0–15 cm) extractable SO₃ was 12 kg/ha SO₃ on the zero S control treatment and 12 and 16 kg/ha SO₃ on the broiler litter and pig slurry treatments, respectively. Topsoil (0–15 cm) extractable SO₃ from the zero S control treatment (equivalent to 6.0 mg/kg SO₃) was below the generally accepted level for S deficiency in soil of 25 mg/kg SO₃ (10 mg/kg S) (Carver, 2005), indicating a likely crop response to applied S.

5.6.3. Visual symptoms of sulphur deficiency

The wheat was assessed visually on 05/07/2012 and all fertiliser S treatments (0 to 75 kg/ha SO₃ fertiliser) showed yellowing of the leaves. In contrast and in general, the wheat on all the organic material treatments (autumn and spring applications) was greener and thicker than on the fertiliser S treatments.

5.6.4. Leaf sulphur content

Leaf samples were taken on 27/06/2012 for analysis of total S content. There was a large and significant (P<0.05) difference between leaf S content between the fertiliser S treatments and the organic material treatments (Figure 21), reflecting the visual differences seen between the fertiliser and organic material treatments. Leaf S content on the fertiliser S treatments was *c*.1900 mg/kg S, (close to the generally accepted level for S deficiency of 2000 mg/kg S), and significantly (P<0.05) less than on the organic material treatments (mean of *c*.2500 mg/kg S).





5.6.5. Grain yields and sulphur offtake

There was an effect of fertiliser S on grain yields, grain S content or grain SO₃ offtake. Grain yields from the fertiliser S treatments were a mean of 8.0 t/ha and grain S content was a mean of *c*.750 mg/kg S (Table 32). The low grain S content across the S fertiliser treatments is comparable to the grain S content from the zero S control treatment at Woburn in the previous year (716 mg/kg S; section 5.5.5) and would normally be indicative of S deficiency. However, the grain N content was also very low (1.1–1.2 %N) across all the fertiliser S treatments, indicating that N rather than S was the main yield limiting factor.

Grain yields were significantly higher in the treatments with organic materials (P<0.05), which we attribute to the extra N availability in those treatments, and this is backed up by the significantly higher grain N concentrations (Table 32). However, these concentrations were still lower than the

other site/seasons. Grain S concentrations were significantly higher in the treatments with organic materials (*P*<0.05).

The 2011/12 harvest year was a very unusual year; the 2011/12 overwinter period was much drier than normal resulting in difficult establishment conditions and a backward crop in early spring. This very dry period was followed by a very wet spring, which delayed the main N fertiliser dressings until early June, which was too late for the crop to utilise the applied N, resulting in N being the primary factor limiting yield.

Treatment	Yield	SO ₃ offtake	Grain S	Grain N	Grain N:S	
	t/ha 85% DM	kg/ha SO₃	mg/kg	%	ratio	
Fertiliser SO₃ response treatments						
0 kg/ha SO₃	8.2 (b)	13 (a)	762 (a)	1.2 (a)	16 (a)	
12 kg/ha SO₃	7.5 (a)	12 (a)	758 (a)	1.2 (a)	15 (a)	
25 kg/ha SO₃	8.6 (b)	14 (a)	748 (a)	1.2 (a)	16 (ab)	
50 kg/ha SO₃	8.3 (b)	13 (a)	762 (a)	1.1 (a)	15 (a)	
75 kg/ha SO $_3$	7.3 (a)	12 (a)	755 (a)	1.1 (a)	15 (a)	
Autumn applied organic materials						
Biosolids – digested	10.9 (d)	24 (c)	1035 (c)	1.9 (c)	18 (f)	
Biosolids – limed	10.2 (cd)	23 (bc)	1050 (c)	1.9 (c)	18 (ef)	
Cattle FYM	10.5 (cd)	23 (bc)	1044 (c)	1.9 (c)	18 (ef)	
Pig FYM	9.9 (c)	21 (b)	1017 (bc)	1.9 (c)	18 (f)	
Broiler litter	10.8 (d)	22 (bc)	964 (b)	1.6 (b)	17 (cd)	
Spring applied organic materials						
Broiler litter	10.7 (d)	23 (c)	1030 (c)	1.8 (c)	17 (de)	
Cattle slurry	10.9 (d)	24 (c)	1012 (bc)	1.7 (b)	16 (bc)	
Statistics						
P-value	<0.01	<0.01	<0.01	<0.01	<0.01	

Note – values followed by different letters in brackets indicate significant differences (P<0.05).

6. Discussion

At the 3 sites that responded to S, the yield increase from the application of S fertiliser was 0.6 and 0.2 t/ha at Frostenden in 2009/10 and 2011/12 (P>0.05), respectively, and 1.2 t/ha at Woburn 2010/11 (P=0.06). Although these yield increases were not statistically significant (based on analysis of variance – ANOVA), yields from all SO₃ application rates (12 to 75 kg/ha SO₃ fertiliser) were consistently higher than from the zero S control at these sites. The yield increase from applied inorganic S fertiliser occurred at the first S application rate (12 kg/ha SO₃ fertiliser), with no further trend for increasing yields with greater fertiliser S application rates (12 to 75 kg/ha SO₃). Application of SO₃ fertiliser resulted in a significant (P<0.05) increase in grain SO₃ offtake from the organic material treatments was compared to grain SO₃ offtake from the fertiliser S response treatments to assess differences between the organic material treatments.

For the spring organic material applications (broiler litter and slurry), there was a good relationship between the recovery of SO_3 in the grain (treatment minus control) and the amount of 'extractable' SO_3 applied in the organic materials (Figure 22). This suggests that, for spring applied organic materials, 'extractable' SO_3 is a good indicator of the SO_3 that is available to the crop.



Figure 22. Relationship between the grain recovery of SO_3 from the spring applied organic materials and quantity of 'extractable' SO_3 applied

At each responsive site, it was only possible to estimate the fertiliser SO_3 replacement value of one of the two spring applied treatments because at each site the grain SO_3 offtake from one of the spring treatments exceeded the maximum from the SO_3 response plots. Based on these limited data, for spring applications of organic materials, there appears to be a good and linear relationship between the fertiliser SO_3 replacement value and quantity of extractable SO_3 applied in the organic materials (Figure 23).



Figure 23. Relationship between fertiliser SO₃ replacement value and quantity of 'extractable' SO₃ applied - spring organic material applications

Following on from this, and the good relationship between the recovery of SO₃ in the grain and quantity of extractable SO₃ applied (Figure 22), it can be concluded that for spring applied organic materials 'extractable' SO₃ is equivalent to inorganic (water soluble) SO₃ fertiliser, and therefore the availability of S from spring applications of organic materials can conservatively be assumed to be equivalent to the proportion of total SO₃ in 'extractable' form. Based on the analysis of organic materials used in this project, 'extractable' SO₃ was between *c*.15% of total SO₃ for cattle FYM to *c*.60% of total SO₃ for broiler litter (Table 33).

Table 33. Organic material 'extractable'	SO_3 content and S use efficiency values for spring applied
organic materials	

Organic material	Extractable SO ₃ (% total SO ₃)
Cattle FYM	15%
Pig FYM	25%
Broiler litter	60%
Cattle/pig slurry	35%
Biosolids	20%

'Extractable' SO₃ from autumn applications of organic materials may be lost via overwinter leaching. The quantity of SO₃ lost via leaching will depend on the amount of 'extractable' SO₃ applied, soil type and overwinter rainfall. For the autumn applications, there was no relationship between grain SO₃ recovery (or fertiliser replacement value) and the amount of 'extractable' SO₃; this was most likely a reflection of overwinter SO₃ leaching losses. At the Frostenden (2011/12) and Woburn (2010/11) sites, where we were able to calculate fertiliser SO₃ replacement values for the autumn applications of organic materials, SO₃ use efficiency was lower than the spring applied organic materials; ranging from 0 to 13% of total SO₃ for livestock manures (mean 5% total SO₃) and from 5 to 29% for biosolids (mean 14% total SO₃). Based on the data from this project, for autumn applied organic materials we suggest efficiency figures of 5-10% of total SO₃ for livestock manures and 10-20% of total SO₃ for biosolids.

Deficiency of S can affect not only crop yield, but also protein quality through its effects on the synthesis of the S-amino acids cysteine, cysteine and methionine. Increased grain S concentrations are a quality improvement that is particularly important for animal feed and the breadmaking quality of wheat (McCaskill and Blair, 1988; Zhao *et al.*, 1999). In the case of breadmaking wheat, increased grain S increases the relative proportion of low-molecular-weight subunits in glutenin, which is important for dough elasticity and extensibility and therefore breadmaking quality (Zhao *et al.*, 1999). We have shown that organic materials increase the S concentrations in wheat grain above those in untreated controls, even though in some cases this did not increase yield. Because both protein content and quality increase with increased grain sulphur, this has a positive quality outcome for farmers who are contracted to produce grain for feed/breadmaking markets.

This work has lead to a better understanding of the available S supply from organic materials and produced guidance to farmers on the availability of S from applications of organic materials. This is likely to improve farm profitability by reducing S applications to cereal crops receiving applications of organic materials.

There is a need for additional work to quantify the S supply from organic materials to other crop types, notably oilseed rape and grassland, to build on the results from the current project. Both oilseed rape and grass cut for silage have a higher S requirement than winter wheat, and therefore organic material applications that supply the S requirement of a winter wheat crop may not supply the full S requirement of an oilseed rape or grassland crop. Furthermore, as the current project has found the 'extractable' SO₃ content of organic materials to be a good indicator of crop S availability, there is now a need for additional laboratory analysis of a range of organic materials to provide robust 'typical/standard' figures for 'extractable' SO₃.

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7. References

- Blair, G.J., Chinoim, N., Lefroy, R.B., Anderson, C.G. and Crocker, G.J. (1991). A soil sulphur test for pasture and crops. Australian Journal of Soil Research, 29, 619-626.
- BSFP (2012) British Survey of Fertiliser Practice 2011.
- Carver, M.F.F. (2005). Monitoring winter barley, wheat, oilseed rape and spring barley for sulphur in England and Wales to predict fertiliser need. HGCA Project Report 374.
- Chambers, B.J. (2003). Manure ANalysis DatabasE (MANDE). Final Project Report to Defra, Project NT2006.
- Defra (2010) Defra's Fertiliser Manual (RB209). The Stationary Office
- McCaskill, M.R and Blair G.J. (1988) Development of a simulation model of sulfur cycling in grazed pastures. Biogeochemistry, 1, 165-181.Watkinson, J.H., Perrott, K.W. and Thorrold, B.S. (1991). Relationship between the MAF pasture development index of soil and extractable sulphur. In: Soil and Plant Testing for Nutrient Deficiencies and Toxicities, Eds.
- Watkinson, J.H., Perrott, K.W. and Thorrold, B.S. (1991). Relationship between the MAF pasture development index of soil and extractable sulphur. In: Soil and Plant Testing for Nutrient Deficiencies and Toxicities, Eds. R.E. White and L.D. Currie. pp 66-71. Fertilizer and Lime Research Centre, Massey University Palmerston North, New Zealand.
- White, R.E. and Currie, L.D. pp 66-71. Fertilizer and Lime Research Centre, Massey University, Palmerston North, New Zealand.
- Withers, P.J.A and Smith, S.R. (1996). The content and fertilizer value of sulphur and magnesium in sewage sludge. UK Water Industry Research Ltd.
- Zhao, F.J. and McGrath, S.P. (1994). Extractable sulphate and organic sulphur in soils and their availability to plants. Plant and Soil 164, 243-250

Zhao, F. J., Salmon S. E., Withers, P. J. A., Evans, E. J., Monaghan, J. M., Shewry, P. R. and McGrath, S. P. (1999). Responses of breadmaking quality to sulphur in three wheat varieties. Journal of the Science of Food and Agriculture 79(13): 1865-1874.