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Nitrogen fertilizer replacement value of sewage sludge, composted household waste and farmyard manure

J. PETERSEN

Department of Agroecology, Research Centre Foulum, Danish Institute of Agricultural Sciences, P.O. Box 50, DK-8830 Tjele, Denmark

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SUMMARY

Field experiments were undertaken during 1998–2000 at Askov Experimental Station and Lundgård Experimental Site, Denmark, to investigate the fertilizer value of anaerobic and aerobic sewage sludges (SS1 and SS2), composted household waste (Compost) and farmyard manure (FYM). The organic residues were applied at two rates with or without supplementary mineral nitrogen (N). The effects of residue application on spring cereal dry matter (DM) yield and N-offtake were related to reference treatments with increasing rates of calcium ammonium nitrate (CAN). DM yields at the high application rates of aerobic sewage sludge (198 kg N/ha/year) and farmyard manure (300 kg N/ha/year) were comparable to the reference treatments receiving 90 kg mineral-N/ha/year. The comparable high application rate of anaerobic sewage sludge was 138 kg N/ha/year only and the DM yield was less. In contrast, the yield effect of Compost was very poor; even the high application rate of 321 kg N/ha/year yielded less than the low application rates of aerobic sewage sludge and farmyard manure, 66 and 100 kg N/ha/year, respectively.

The nitrogen fertilizer replacement value (NFRV) of the organic residues was estimated for all four combinations of the response variables DM-yield and N-offtake and the independent variables of total-N and ammonium-N applied. NFRV was in the range 49–68% for the sewage sludges and FYM based on the DM-yield: total-N relation, and slightly smaller for the N-offtake: total-N relation, 29–53%. The highest values were obtained for the aerobic sewage sludge, having a potential manurial value comparable to FYM. In contrast, the NFRV of Compost was low, about 10%.

The concentration of ammonium-N in organic residues and manures is often used for predicting the fertilizer value. Ammonium-N based NFRV of 160–210% for the sewage sludges indicate that water extractable ammonium-N underestimates the NFRV, probably due to the content of easily degradable organic matter in digested sewage sludge. The estimated NFRV for Compost was above 100% but connected with high uncertainty. In contrast, the ammonium-N based NFRV for FYM was only 70–87%, probably due to ammonia volatilization caused by incomplete incorporation of large quantities and the high ammonium-N: total-N ratio in this residue.

INTRODUCTION

The policy in the European Communities (EC) is to protect the aquatic environment from adverse effects of waste water discharges. Waste water treatment aims to remove more than 80% of the nitrogen (N) and phosphorus (P) from the urban waste water (CEC 1991*a*). The dewatered digested sewage sludge resulting from waste water treatment therefore contains nutrients potentially useful for plant production. Also, the agricultural use of sewage sludge is regulated, and the policy is to prevent harmful effects on soil, vegetation, animals and man, thereby encouraging the correct use of such sewage sludge (CEC 1986). In Sweden, Norway and Denmark, respectively 30, 48 and 67% of sewage sludge is applied on agricultural land and regarded as a fertilizer (Petersen & Petersen 1999).

Agricultural use of nutrients in organic household waste may come in focus due to the policy of reducing landfill disposal (CEC 1999). In the Scandinavian countries less than 10% of the organic household waste is collected separately for composting and only a minor fraction of this is composted and used as a nutrient source in agriculture (Petersen & Petersen 1999).

Email: Jens.Petersen@agrsci.dk

Table 1. *Soil characteristics (0–20 cm) of the two experimental sites*

Site	Askov	Lundgård
Soil type	Sandy loam	Loamy sand
Clay (<2 μm) (g/kg)	113	52
Silt (2–20 μm) (g/kg)	197	81
Fine sand (20–200 μm) (g/kg)	289	211
Coarse sand (200–2000 μm) (g/kg)	375	632
Carbon, total (g/kg)	16·2	14·3
Nitrogen, total (g/kg)	1·4	1·0
Phosphorus, total (mg/kg)	703	559
Phosphorus, 0·5 M $NaHCO_3$ extractable (mg/kg)	34	24
Potassium, 0·5 M NH_4-acetate extractable (mg/kg)	142	93
Copper, total (mg/kg)	10	5·8
Zinc, total (mg/kg)	30	19
pH (H_2O)	6·78	6·34
Cation Exchange Capacity (meq/100 g dry soil)	10	6·7

Based on an EC council directive (CEC 1986), the implementation of legislation on sewage sludge application to agricultural land can be adjusted to take account of national conditions and therefore differs between the EU countries. In Denmark, both sewage sludge and composted household waste have to be incorporated into the soil within 12 h after application (Anon. 2000). In addition, application of organic residue-P to agricultural land is restricted to 40 kg/ha/year in total. Stock farms usually apply animal manure at rates leaving no capacity for residue application within the P-threshold, and therefore organic residue application is restricted to arable farms only. Furthermore, organic residues may not be applied to growing crops for human consumption for reasons of hygiene (Anon. 2000), and the highest N-utilization is normally achieved with spring application (Petersen 1996*a*). In practice this reduces the potential crops receiving organic residues to spring sown cereals only.

The present work was a subproject of the Danish *Centre for Sustainable Land Use and Management of Contaminants, Carbon and Nitrogen* (1997–2001) focusing on the environmental effects and turnover of organic contaminants in sewage sludge and compost applied to agricultural land. The results from this centre contribute to our knowledge of the risks associated with the recycling of nutrients in municipal residues (Petersen *et al*., in press). However, a prerequisite for the correct use of municipal residues in agriculture is information about the nitrogen fertilizer replacement value (NFRV), defined as the amount in kg N/ha of mineral nitrogen fertilizer needed to replace 100 kg total-N/ha in the residue to obtain the same response in yield or N-offtake. The aim of the present field experiment was to examine the NFRV of municipal sewage sludge and composted organic household waste applied at the low rates prescribed by legislation, including possible interaction effects of residue type, application rate and supplementary N applied as mineral fertilizer.

MATERIALS AND METHODS

A field experiment was undertaken during 1998–2000 at two sites in Denmark using a fixed layout. Four types of organic residue were applied at two rates with or without supplementary mineral nitrogen, giving a total of 16 residue treatments. Four rates of mineral N were included as reference treatments. Each of the 20 treatments was replicated three times in a randomized complete block design at both sites. The blocks were rows of 20 plots. To prevent cross-contamination from tillage-induced soil movement (Sibbesen *et al*. 2000), the blocks were 10 m apart and the full size of plots was 4 m × 14 m = 56 m^2 plus 1 m border between the full-plots. The net plot used for yield determination was 1·5 m × 10 m = 15 m^2.

Soil and climate

The two sites, Askov Experimental Station and Lundgård Experimental Site, represent two different soil types (Table 1). Prior to the start of the experiment a representative soil sample of the topsoil (0–20 cm) from each site was analysed for total carbon (C), total nitrogen (N), extractable phosphorus (P; 0·5 M $NaHCO_3$) and potassium (K; 0·5 M NH_4-acetate), total copper (Cu), total zinc (Zn) and cation exchange capacity. The Askov soil had a finer texture, which was reflected in the cation exchange capacity as well as the concentration of Cu and Zn (Table 1). Compared with Danish soils in general, the nutrient status of the experimental fields was medium with respect to P. The potassium status at Lundgård was medium and high at Askov.

The Lundgård Experimental Site is located less than 5 km east of the Askov Experimental Station

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Table 2. *Monthly air temperature and precipitation measured at Askov Experimental Station during the growing season of the 3 experimental years. The 1961–90 long-term average for Askov Experimental Station is also shown*

	Year	May	June	July	August
Air temperature, °C (mean)	1998	12·1	13·6	13·7	14·0
	1999	11·1	12·9	17·0	16·3
	2000	12·8	13·4	14·5	15·1
Long-term average 1961–90		10·8	14·3	15·5	15·5
Precipitation, mm (sum)	1998	24	77	153	53
	1999	42	123	65	40
	2000	67	51	50	53
Long-term average 1961–90		55	67	74	82

(55°28′ N, 9° 07′ E), and climatic conditions were assumed to be identical (Table 2). The number of accumulated degree days, using the soil temperature at a depth of 10 cm beneath short cut grass and 0 °C as a basis, was 488 (S.E. ± 13 °Cd) and 1934 (S.E. ± 42 °Cd) from date of residue application until ear emergence and harvest, respectively, as an average of the three growing seasons.

Crop and management

A spring cereal crop rotation was chosen for the 3-year experimental period (Table 3) based on the above-mentioned restrictions of organic residue application to agricultural land.

Studies on the turnover of organic contaminants may be affected by use of pesticides. Therefore, these were omitted from October 1996 to avoid interference by agrochemical compounds. During the experimental period, weeds were controlled mechanically by harrowing according to the strategy by Rasmussen & Rasmussen (1995). Irrigation was possible at Lundgård and carried out at 30 mm water deficit. In 1998 the experiment was irrigated with 15 and 30 mm on 26 May and 22 June, respectively, and in 2000 with 32 mm on 30 June.

The mature crops were harvested and dry matter (DM) determined in grain and straw after drying at 80 °C for 18 h. The concentration of nitrogen (N) was determined by the Dumas principle (Hansen 1989), and phosphorus (P) and potassium (K) were determined colorimetrically and photometrically, respectively, after dissolving incinerated material in acid (Stuffins 1967; CEC 1971).

Residues and application rates

In 1998–99 two types of sewage sludge were obtained from Lundtofte treatment works (Lundtofte Renseanlæg, DK-2800 Kgs. Lyngby). A sewage sludge with an elevated load of organic contaminants (SS1) was obtained from a pre-settling tank with chemical P removal. This sludge was anaerobically digested and dewatered prior to storage. A second sludge with a low level of organic contaminants (SS2) was obtained from an aeration tank and dewatered prior to use. Due to changes in processing at Lundtofte, SS1 and SS2 sludges in 2000 were obtained from Skanderborg (Skanderborg Centralrenseanlæg, DK-8660 Skanderborg) and Odder (Odder Spildevandscenter, DK-8300 Odder) treatment works, respectively. The compost (Compost) was obtained from a municipal composting works (Komposteringsanlæg Århus Nord, DK-8200 Århus N), where organic household waste was mixed with chopped straw (< 3 cm, 8 % by weight) to ensure adequate aeration during composting. The farmyard manure (FYM) was obtained from a local pig farm stocked with sows and piglets using straw for bedding. The piglets were weaned off after 4 weeks and grown until 30 kg before export from the farm. Calcium ammonium nitrate (CAN) was used as reference for calculation of the NFRV of applied organic residue.

The residues were applied at two rates reflecting practice but using carbon (C) as a common basis. The 1 × target application rate of 300 kg C/ha/year for SS1 and SS2 corresponds to about 40 kg P/ha/year, which was the threshold value for sewage sludge application in Denmark during the experimental period (Anon. 2000). From July 2000 the P-threshold was reduced to 30 kg P/ha/year. The 3 × target application rate of 900 kg C/ha was based on the permission to apply 3 × 40 kg P/ha every 3 years. However, to obtain differences in application rate during the experimental period, the 3 × rate was applied each year in the present study. Preliminary investigations indicated that the input of nitrogen using an application rate of 300 kg C/ha of Compost and FYM would be very low. Hence, the target for the 1 × application rate of these residues were set at 900 kg C/ha/year, which, on carbon basis, is comparable to the 3 × rate of the sewage sludge. Consequently, the target application rate for Compost and FYM was 2700 kg C/ha/year for the 3 × treatment.

The residue applications were all combined either without (−N, 0 %) or with (+N, 80 %) supplementary mineral N applied as CAN. In the reference treatments, CAN was applied in rates of 0 %, 40 %, 80 % and 120 % of the legislative maximum N-rate for spring cereals (122 and 95 kg N/ha/year for barley and oat, respectively). PK-fertilizer (25 kg P/ha/year and 70 kg K/ha/year) was also applied in the reference treatments. The mineral nitrogen fertilizer was surface applied on 20 April 1998, but incorporated by harrowing just before sowing in 1999 and 2000.

On receipt of residues at Askov Experimental Station 6–10 subsamples were taken of each residue and dried at 100 °C for 18 h for determination of

Table 3. *Crop rotation and dates (Askov/Lundgård) of residue application, sowing, ear emergence and harvest*

			Date			
Year	Crop	Cultivar	Residue application	Sowing	Ear emergence	Harvest
1998	Spring barley	Lamba	4+5 May	11 May	6/4 July	3/1 Sep
1999	Oat	Corrado	27+28 April	29 April/3 May	7/6 July	1 Sep/25 Aug
2000	Spring barley	Punto	1+2 May	4 May	6/4 July	31/28 Aug

Table 4. *Characteristics of applied residues: anaerobic (SS1) and aerobic (SS2) sewage sludge, compost and farmyard manure (FYM). Mean and* S.E. *of 3 years*

	SS1		SS2		Compost		FYM	
	Mean	±S.E.	Mean	±S.E.	Mean	±S.E.	Mean	±S.E.
DM (g/kg)	227	24	162	12	718	36	237	8
g OM/kg DM	534	13	680	5	412	40	704	52
g C/kg DM	265	11	276	13	222	15	337	12
g inorganic-C/kg DM	6	0·6	1	0·6	17	6·3	4	0·7
g N/kg DM	32·2	1·2	57·8	8·8	19·1	1·4	31·2	2·5
g NH4-N/kg DM	5·5	1·1	11·2	2·9	0·6	0·2	10·6	3·9
g P/kg DM	31·5	1·0	33·0	1·6	3·7	0·3	23·7	2·8
g K/kg DM	1·8	0·1	4·9	0·6	10·7	0·5	17·9	0·6
mg Pb/kg DM*	106	26	68	13	28	—	—	—
mg Cd/kg DM*	2·2	0·03	1·4	0·2	0·4	—	—	—
mg Hg/kg DM*	2·6	0·7	0·9	0·03	0·2	—	—	—
mg Ni/kg DM*	22	0·7	18	2·0	8	—	—	—
mg Cr/kg DM*	30	1·7	18	3·3	10	—	—	—
mg Zn/kg DM	977	109	447	31	112	7	1024	132
mg Cu/kg DM	278	29	215	14	63	3	265	28

* Data obtained from the regular internal control at the treatment plants.
—, not determined.

DM. Subsequently the samples were incinerated at 550 °C for a minimum of 3 h for determination of organic matter content by loss on ignition. Assuming 40% C in the organic matter, the residue application rate was estimated for each target C rate. The residues were stored in covered heaps for protection against precipitation until application. The maximum storage time was 4 weeks.

The concentrations of organic contaminants, di(2-ethylhexyl)phthalate (DEHP), nonylphenol plus ethoxylates (NP+NPE), linear alkylbenzene sulfonates (LAS), and aromatic hydrocarbons (PAH), originating from plasticizers, detergents, soaps and incomplete combustion of fuel, respectively, in the residues increased in the order: FYM < Compost < SS2 < SS1 (Petersen *et al.*, in press). The residues were applied in this order to avoid cross-contamination of the organic contaminants by application tools. Application and spreading by fork was very demanding, and the four residues were therefore applied on two successive days in the spring (Table 3). The residues were incorporated to 10 cm depth using two passes of a rotary cultivator within 2 h of application to minimize N loss through ammonia volatilization.

During application 8–12 subsamples of each residue were taken making up a total sample of 15–20 litres in a closed bucket stored in shadow in the field. The total sample was cooled overnight, and after mixing 21 0·5-litre subsamples of each residue type were made and stored at −18 °C until analysis. Three randomly selected samples per residue type and year were analysed for DM (80 °C for 18 h), total concentration of C (carbon dioxide after combustion in pure oxygen), N (Kjeldahl), P (colorimetric), K, Cu and Zn (photometric), as well as inorganic-C (gas volumetric determination of carbon dioxide after acid addition) (Table 4). Mineral-N was extracted by shaking for 30 min using distilled water, filtered and determined colorimetrically. The remaining samples were used for analysis of organic contaminants (Petersen *et al.*, in press) or stored as spare samples for possible future analysis.

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Table 5. *Application rates at the 1 × residue rate: anaerobic (SS1) and aerobic (SS2) sewage sludge, compost and farmyard manure (FYM). Mean and* S.E. *of 3 years*

	SS1		SS2		Compost		FYM	
	Mean	±S.E.	Mean	±S.E.	Mean	±S.E.	Mean	±S.E.
Tonne matter/ha/y	6·4	0·52	7·0	0·49	7·9	0·42	13·7	1·05
kg DM/ha/y	1423	38	1130	25	5690	558	3235	244
kg OM/ha/y	759	10	768	22	2298	50	2252	6
kg C/ha/y	369	25	310	9	1141	34	1070	39
kg N/ha/y	46	0·9	66	11·1	107	5·2	100	0·8
kg NH4-N/ha/y	7·7	1·3	12·8	3·4	3·2	0·7	32·6	9·7
kg P/ha/y	45	0·8	37	1·9	21	1·3	78	13·8
kg K/ha/y	3	0·1	6	0·8	60	3·3	58	3·9
g Cu/ha/y	398	50	243	15	354	31	866	135
g Zn/ha/y	1396	181	506	44	636	77	3338	541

Due to the different origins, residue characteristics were very variable when compared with each other (Table 4). However, the in-year variation between parallel samples of each residue was minor, compared with the year-to-year variation shown in Table 4. The relative variation (CV) between years was not related to residue type, but to the variable analysed. Thus, CV below 10% was obtained for the variables inorganic C and ammonium-N, whereas CV for all other variables was below 3% (calculated from Table 4). The fraction of ammonium-N was 17–19% of total N for the sewage sludge, but only 3% for the Compost. FYM had the greatest fraction of ammonium-N at 33%. For all residues, the concentration of ammonium-N exceeded that of nitrate-N by more than 1000. The rates of applied dry matter and nutrients were calculated based on the figures in Table 4 (Table 5).

The assumed value of 40% C in organic matter was not valid. As a mean of all residue samples ($n = 36$), the carbon concentration was 52·2% (S.E. ± 1·8) in the organic matter. This is the main reason for an application rate of C about 20% higher than the target rate for SS1, Compost and FYM (Table 5).

The different origin of the residues was reflected in the applied rates of nutrients. The applied rate of P in sewage sludge was close to the legal threshold, whereas the amount of K was very low. Using FYM, both P and K were applied in excess of a cereal crop offtake. With Compost the K rate was also high, but the P rate was relatively low.

The applied amount of Cu in FYM was 2–3 times higher than in the other three residues, and Zn in FYM was 5–6 times higher than in SS2 and Compost due to application of supplementary Cu and Zn in piglets' diets (Table 5). The amount of Zn applied in SS1 was lower than in FYM, but three times higher than in SS2. Application of Cu and Zn in CAN used for supplementary N and the reference treatments were only 1·5 g/ha/year. In the PK-fertilizer used for the reference treatments, Cu and Zn application were 330 and 13 g/ha/year, respectively.

Figures for the concentrations of heavy metals were obtained from the regular internal control at the treatment works (Table 4). Based on these figures and the 1 × application rate, the mean application rate of heavy metals (g/ha/year ± S.E.) was as an average of SS1, SS2 and Compost: Pb (130 ± 19), Cd (2 ± 0·2), Hg (2 ± 0·3), Ni (33 ± 4) and Cr (40 ± 18).

Calculations and statistics

For each plot, site and year the DM yield of grain and straw were multiplied by their respective nutrient concentration, and then summarized to total nutrient offtake. Initially, general linear models (GLM) were applied to two balanced subsets of data, one for the 16 residue treatments and one for the four CAN reference treatments. The GLM for the residue treatments includes the class variables residue type (RT), residue rate (RR) and rate of supplementary nitrogen (SN), as well as all second-order interactions and the third-order interaction (RT × RR × SN). The CAN reference treatments were analysed using the nitrogen rate (NR) as class variable. In both cases the effects were tested using the respective Effect × Year × Site interactions, and the RT × RR × SN × Year × Site and NR × Year × Site interactions were used for calculation of the S.E. in Figs 1 and 2. The models were applied to the response variables DM yield, N-, P- and K-offtake for grain, straw and total crop.

The RT and RR effects interact with supplementary N and for further analysis of the RR × RT interaction the data were divided into two sections; section A: eight residue treatments without supplementary nitrogen plus the four reference treatments, 12 treatments in total. Section B: eight residue treatments with supplementary nitrogen.

For section A, a uniform nitrogen response function was assumed. Thus, the same relation may

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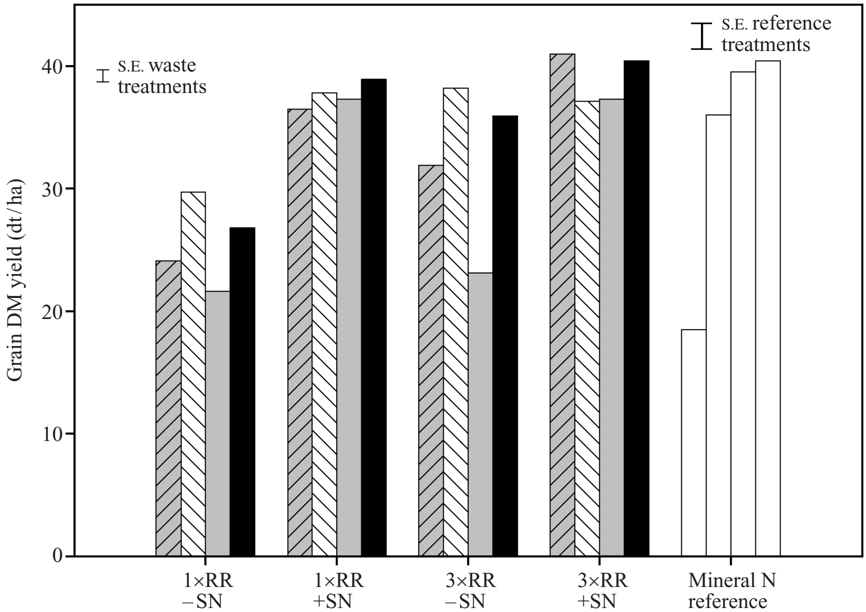

Fig. 1. Grain DM yields (dt DM/ha) for four residues applied in two rates (1 × and 3 ×) with or without supplementary N (±SN). Mineral N applied as reference at four rates. Means of three replicates on two sites in 3 years, in total 18 observations. The S.E. was calculated for the residues and reference treatments separately. Bars: Sewage sludge 1 (grey, crosshatched), Sewage sludge 2 (crosshatched), Compost (grey), FYM (black) and mineral reference (white).

describe the response to N applied in the residues using individual estimates of the parameters for each combination of residue, year and site. For this purpose, two quadratic equations using four combinations of independent-response variables were applied (Eqns 1 and 2).

First, a non-linear model (NLIN) including a quotient, q, expressing the effect of residue types in relation to mineral fertilizer (Eqn 1).

$$y = a\{k\} + q\{i\}(bx + cx^2) + \varepsilon \qquad (1)$$

where y is the response variable (DM yield or N-offtake), $a\{k\}$ is the intercept for each residue type, year and site, $k \in \{1 \ldots 30\}$, $q\{i\}$ is a quotient for the effect of residue type i in relation to the CAN reference treatment for which $q\{i\}$ equals 1, $i \in \{1 \ldots 5\}$, x is the independent variable (applied amount of total-N or ammonium-N), b and c are constants, and ε is an error term.

Thus, the estimate of q is the factor that multiplied with the CAN reference response curve brings the reference to overlay the response curve for the residue, using an individual intercept for each residue type, year and site. For convenience, the estimates of q are expressed in percentages giving the NFRV.

Second, a full general linear model with independent estimates for each residue including the Year × Site interaction was used for maximum explanation of the variance (i.e. minimum variance for the error term) using the independent variable as a covariant (Eqn 2). The reduced but interpretable model (Eqn 1) was compared with the full model (Eqn 2) using a F-test.

$$y = d\{j\}f\{i\} + g\{i\}x + h\{i\}x^2 + \varepsilon \qquad (2)$$

where y is the response variable (DM yield or N-offtake), $d\{j\}$ is the effect of the Year × Site interaction, $j \in \{1 \ldots 6\}$, $f\{i\}$, $g\{i\}$, $h\{i\}$ is the zero, first and second order effect of N source i, $i \in \{1 \ldots 5\}$ (four types of residue or the CAN reference), x is the independent variable (applied amount of total-N or ammonium-N) and ε is an error term.

For section B, the means of the 80% N reference treatment (by year and site) was subtracted from the residue treatments supplied with mineral nitrogen. These differences were treated by a GLM including the RT × RR-interaction effect. The effects were tested using the respective Effect × Year × Site interactions as residuals.

The apparent nitrogen recovery, ANR, was calculated for all 16 residue treatments as the difference in N-offtake between the residue treatment and the corresponding reference treatment divided by total-N applied in the residue treatment. A GLM was applied, including the class variables residue type (RT), residue rate (RR) and rate of supplementary nitrogen (SN), as well as all second-order interactions and the third-order interaction (RT × RR × SN). The effects were tested using the respective Effect × Year × Site

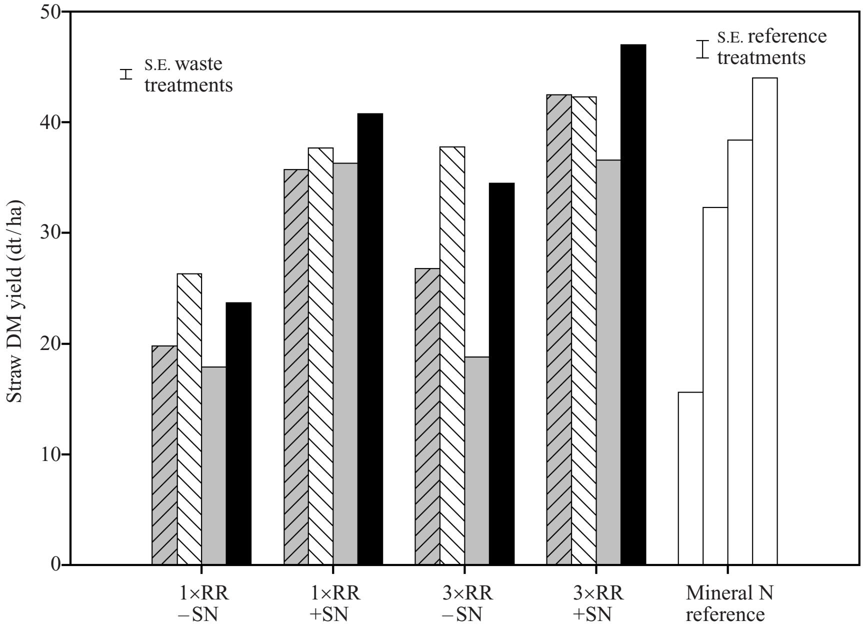

Fig. 2. Straw DM yields (dt DM/ha) for four residues applied in two rates (1 × and 3 ×) with or without supplementary N (±SN). Mineral N applied as reference at four rates. Means of three replicates on two sites in 3 years, in total 18 observations. The S.E. was calculated for the residues and reference treatments separately. Bars: Sewage sludge 1 (grey, crosshatched), Sewage sludge 2 (crosshatched), Compost (grey), FYM (black) and mineral reference (white).

interactions, and the RT × RR × SN × Year × Site interaction was used for calculation of the standard error.

The SAS procedures GLM and NLIN were used for the calculations (SAS Institute 1996).

RESULTS

The crops germinated and developed well. Weeds were normally well controlled by harrowing 1–2 times during the germination phase, but three harrowings were necessary at Lundgård in 1999. Aphids were not observed in the experiments. Mildew was only observed in 1998, covering 5–20% of the green leaf area on 10 July.

Lodging was not observed in the barley grown in 1998 and 2000, but did occur in oats in 1999 at both sites. An assessment performed a few days before harvest showed lodging was severe in some of the residue plots receiving supplementary mineral nitrogen as well as in some of the reference plots. Despite lodging, seed shedding was not observed.

Grain and straw DM yields

The initial GLM show interaction between RT, RR and SN for all measured response variables. For DM yields, Figs 1 and 2 illustrate that SN had a significant influence on the effect of RT and RR, as well as the RT × RR interaction.

Without supplementary N and for 1 × RR, the highest DM yields were obtained for SS2 and FYM, whereas the yields for SS1 and Compost were only slightly higher than for the unfertilized treatment. For Compost, the effect of increasing the residue rate from 1 × to 3 × was insignificant, but for the other residues the DM yield increased on average 32% (8·5 dt DM/ha) and 42% (9·8 dt DM/ha) for the grain and straw, respectively.

When applying supplementary N, the effect of increasing the Compost rate from 1 × to 3 × was also insignificant. For the other residues, the yield increase for the 3 × RR was less obvious and of smaller magnitude compared with the section without supplementary N. Thus, the 3 × RR increased grain yield for the SS1 treatment by 12% (4·5 dt DM/ha), and increased straw yield for the SS1, SS2 and FYM treatments by 15% (5·8 dt DM/ha) on average.

Residue type by residue rate interaction (RT × RR)

As the effect of RT and RR depends on the rate of supplementary mineral N, the second-order interaction effect of RT × RR may be treated separately for the two rates of supplementary N.

Without supplementary N

The R^2 values for the model (Eqn 1) were within the range 0·8–0·9, and only slightly less than for the GLM model (Eqn 2) used for maximal explanation of the

Table 6. *Estimates* (±s.e.) *of the nitrogen fertilizer replacement value* (*NFRV*) (%) (*total crop*)

Response variable Independent variable	DM-yield Total-N	N-offtake Total-N	DM-yield Ammonium-N	N-offtake Ammonium-N
SS1	48·5±6·05	32·0±4·29	169±23·1	161±24·8
SS2	67·6±6·20	53·1±4·29	158±16·5	206±19·5
Compost	12·5±4·51	8·2±2·25	113±47·3	190±55·0
FYM	54·6±6·47	29·0±3·51	87±8·4	70±6·2

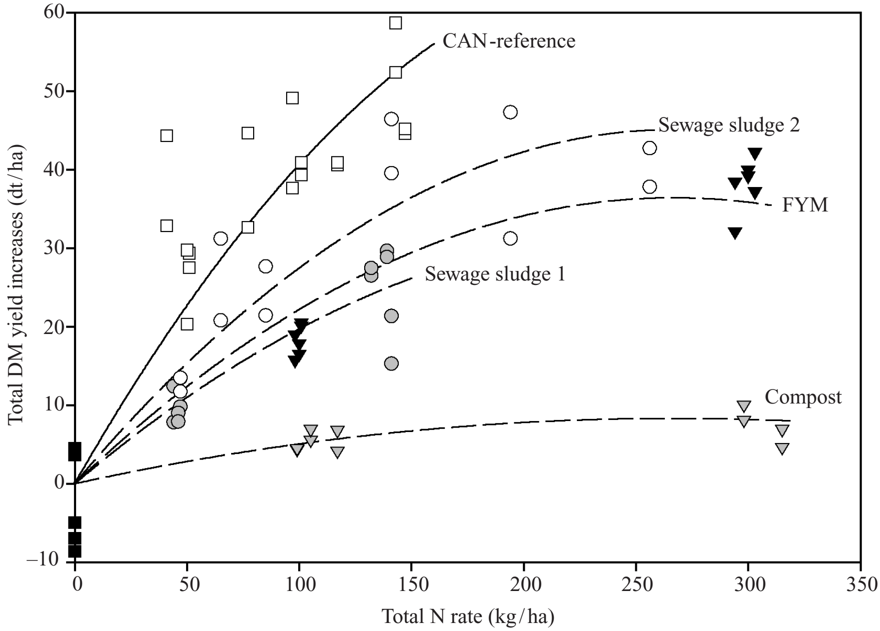

Fig. 3. Average yield (dt DM/ha) of three replicates measured for each combination of treatment, year and site as well as the estimated functions, using Eqn (1), exclusively the Treatment × Year × Site dependent intercept, a{*k*}.

variance. Using N-offtake as response variable, the two models are quite similar in description of the variation, but for the DM-yield response Eqn (1) leaves some variation unexplained (*F*-tests not shown). The increased residual variation was mainly caused by the RT × Year × Site interaction, but it was not possible to include this interaction in Eqn (1) without losing the interpretation of the nitrogen fertilizer replacement values, $q\{i\}$. Thus, the model in Eqn (1) was accepted despite the reduced explanation of the variation in DM-yield data. Furthermore, two different reductions of the model in Eqn (1) were tested and rejected. Neither exclusion of the quadratic term ($c=0$) nor use of a common intercept for each RT, instead of the RT × Year × Site interaction, were accepted (*F*-tests not shown).

The NFRV, $q\{i\}$ in Eqn (1), was estimated for both grain and straw, as well as for the total crop. The estimates for grain were on average slightly higher than for straw, but the effect of residue type showed the same pattern and magnitude. Therefore, only the estimates for the total crop are shown (Table 6). Using applied total-N as the independent variable, the NFRV estimates were all less than 100 for both response variables (Table 6), and the order was SS2 > FYM ≈ SS1 ≫ Compost (Figs 3 and 4). The estimates for sewage sludge and Compost increased to 100 or more using ammonium-N as the independent variable. Furthermore, the standard error of the estimates increased, especially the estimates for Compost which were connected with an uncertainty twice as high as the other estimates. The estimates for the sewage sludge were surprisingly high, 160–210. The estimates for FYM also increased using ammonium-N as the independent variable, but they did not exceed 100.

Supplementary N

Despite the rate of supplementary N applied for the residue plots being similar to the 80 %N reference treatment, application of 1 × RR resulted in negative

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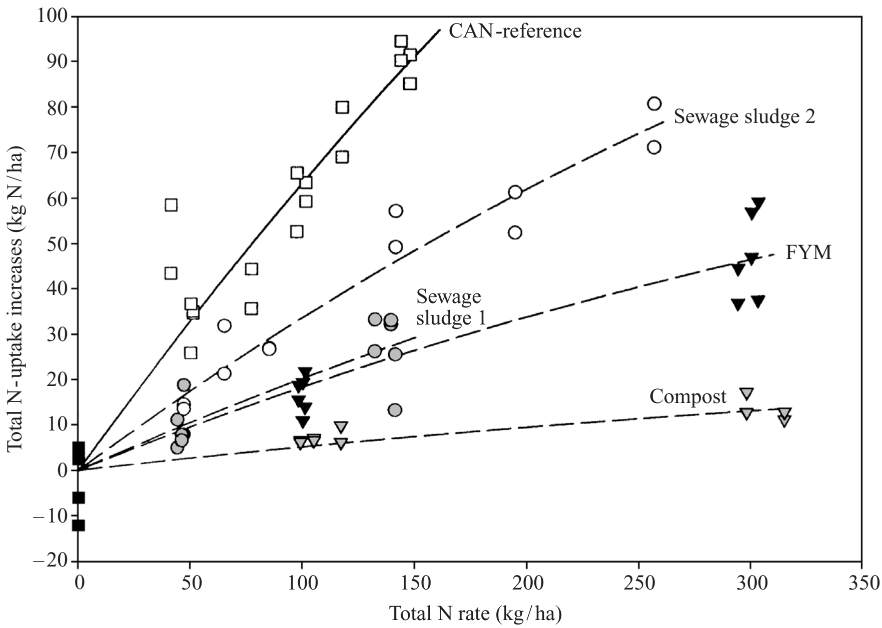

Fig. 4. Average N-offtake (kg N/ha) of three replicates measured for each combination of treatment, year and site as well as the estimated functions, using Eqn (1), exclusively the Treatment × Year × Site dependent intercept, a{*k*}.

or only low DM yield increases (Table 7). The DM yield was increased for 3 × RR of SS1 and FYM only. SS2 and FYM increased the N-offtake compared with the reference, even at 1 × RR. Also 3 × SS1 and 3 × Compost increased the N-offtake, but to a far lesser extent for the Compost (Table 7).

The DM yield and N-offtake of 3 × FYM and 3 × SS1 were comparable to the 120 %N reference, which increased by 6·3 dt DM/ha (S.E. ± 2·6) and 31·6 kg N/ha (S.E. ± 3·6), respectively, compared with the 80 %N reference treatment. In addition, the N-offtake for 3 × SS2 exceeded the 120 %N reference treatment, but this was not reflected in the yield. The apparent nitrogen recovery was less variable than increases in DM yield and N-offtake. Significant differences in ANR between residues were obtained, whereas residue rate and the RT × RR interaction were of minor importance (Table 8). Generally, ANR decreased when supplementary N was applied.

P- and K-offtake

Total P-offtake at harvest was in the range 9–27 kg P/ha as means of years and sites, and 73 % (S.E. ± 9 %) of the crop P-offtake was found in the grain. The difference in P-offtake was related to DM-yield, and not caused by differences in the grain P concentration, since the average concentration was 3·8 mg/g DM with a maximum difference between treatments of 0·17 mg/g DM.

The total K-offtake at harvest was 32–101 kg K/ha, and 69 % (S.E. ± 11 %) of the crop K-offtake was located in the straw. The straw K concentration was 12·6 mg/g DM for the two sewage sludge treatments, but increased to 16·0 mg K/g DM for both Compost and FYM at 3 × RR, even though the applied amount of K in these residues was 10 times greater than for the sludge (Table 5). Despite differences in application rate and K concentration in the crop, DM yield was the dominating factor with respect to K-offtake in the straw.

Determination of P- and K-uptake in the crop at ear emergence at Lundgård showed that more than 90 % of the P-offtake at harvest was taken up at this time (data not shown). On the other hand, the K-uptake at ear emergence corresponded to 1·7 times the offtake at harvest (data not shown), indicating a substantial K loss during the grain-filling period.

DISCUSSION

General conditions for the experiment

Most of the 230 modern waste water treatment works in Denmark operate with one production line only, but at the time this experiment began Lundtofte treatment works operated with two lines producing sewage sludge with different concentrations of heavy metals and organic contaminants, and provided the advantage that waste water for the two sludges was

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Table 7. *Total crop increases in DM yields and N-offtake of the residue treatments with supplementary nitrogen compared with the 80% N reference treatment*

	DM yield (dt DM/ha)		N-off take (kg N/ha)	
	1 × residue rate	3 × residue rate	1 × residue rate	3 × residue rate
SS1	−5·9	5·4	−1·5	25·4
SS2	−2·6	1·2	12·2	40·5
Compost	−4·6	−4·2	−1·3	7·8
FYM	1·6	9·3	9·0	36·4
S.E.	1·3		2·3	

Table 8. *Apparent nitrogen recovery, ANR (%), for the four residues at two application rates without (−N) and with (+N) supplementary N using the 0%N and 80%N treatments as reference, respectively.* S.E. = *3·0*

	1 × residue rate		3 × residue rate	
	−N	+N	−N	+N
SS1	17	—	19	19
SS2	31	19	31	22
Compost	6	—	4	3
FYM	14	10	15	12

—, negative values, not significantly different from nil.

obtained from the same catchment area. These two lines were used to represent 'high' and 'low' charged sewage sludge. Unfortunately, the owner (Lyngby Council) fused the lines in autumn 1999, which precluded further delivery of two sludge qualities from this treatment works. Treatment works at Odder and Skanderborg were chosen as new suppliers for the 2000 growing season. Thus, the experiment was continued with the desired difference in sludge quality. FYM originating from monogastric animals is comparable to sewage sludge and compost with respect to origin and structure, and this commonly occurring carbon source, with very low concentrations of DEHP, NP+NPE, LAS and PAH, was included as a reference for turnover studies of organic contaminants (Petersen *et al*., in press).

Incorporation is an important factor in reducing ammonia volatilization from animal manure (Sommer 1992) and organic residues. Therefore, all residues in this experiment were incorporated within 1–2 h of application. Incorporation by ploughing, although an effective method of reducing ammonia volatilization and a common practice, turns the soil around leaving the applied residue in bands. This uneven distribution was not acceptable in relation to representative sampling of the soil for determination of very small amounts of organic contaminants (Petersen *et al*., in press). Therefore, a more even distribution and incorporation was achieved with a rotary harrow. In this way a more effective residue-soil contact was achieved resulting in a faster turnover of the applied residue (Petersen *et al*., in press), but a minor fraction of the residues were left at the soil surface exposed for ammonia volatilization.

Sowing was done relatively late in spring in all three experimental seasons. This was due to the demand for two convenient successive days for residue application, which could not be obtained earlier in the spring. The delay was about 14 days, and the shorter growing period may have reduced the grain yield slightly. This was accepted, because uninterrupted residue application and successful establishment of the crop had high priority. Despite the occurrence of mildew in 1998 and lodging in 1999, the growing conditions were acceptable, making the interpretation of the results unrestricted.

The treatment effects were apparent already from the tillering stage and throughout the growing season. Measurement of spectral reflectance for calculation of a vegetation index related to light interception and DM production (Christensen 1992) was performed weekly during the elongation phase (data not shown). The differences recorded in May at the start of the elongation phase increased during June and correspond to the effects on grain and straw DM yields (Figs 1 and 2). Thus, low DM yields were directly related to a poor fertilizer value of the applied residue.

Heavy metals

The increased focus on waste water treatment and heavy metal sources since 1986 (CEC 1986) has reduced the heavy metal concentration in sewage sludge significantly (Evans 2001) compared with the quality in the 1960s and 1970s (McGrath 1984; Larsen & Petersen 1993). SS1 is representative of the upper range of concentrations of heavy metals in sewage sludge applied to agricultural land in Denmark, whereas SS2 represents the average concentration (Anon. 1999–2001). Despite differences, both sludges may be characterized as lightly contaminated with heavy metals. Thus, in combination with the reduced application rates of today, the amount of heavy metals applied in municipal residues may be described as very low and without any influence on crop growth. In contrast, pig manure is the major source of soil contamination with Cu and Zn in Denmark, due to high metal concentrations and high application rates as well as common and widespread use.

Fertilizer value of residue N

Application of nitrogen-containing organic matter on agricultural land affects the nitrogen turnover in the soil, and the influence depends on the type of matter applied (Smith *et al.* 1998*a*, *b*; Ambus *et al.* 2002). Detailed knowledge about nitrogen turnover in the soil is important, but is less so in a comparison of crop response to organic matter applications with that of mineral fertilizer treatments. Obviously, such a 'black box' simplification reduces the possibility of explaining the effects, but the estimated nitrogen fertilizer replacement value (NFRV) is a quantification of practical significance for correct use of organic residues as a nutrient source in agriculture. The estimated NFRV is supposed to express the fertilizer value related to mineral N plus the turnover of the organic N pool mineralized within the first year assuming humid, temperate conditions (Smith *et al.* 1998*b*). Mineralization of the slowly degradable pool in the following years was not measurable (Smith *et al.* 1998*b*; Ambus *et al.* 2002) and therefore assumed to be without influence on the NFRV.

Residue without supplementary N

The present experiments were performed near fields used for an experiment testing two types of sewage sludge during 1974–79 (Larsen & Petersen 1993, summarized by Petersen (2001*a*)). They estimated an NFRV of 25–30% based on the DM : total-N relation, but the estimates for the present experiments were twice as high (Table 6). There may be several explanations for the difference. First, changes and development in the waste water treatment process. In the 1970s, before the introduction of the EC directive concerning urban waste water treatment (CEC 1991*a*), only the oxygen demand was reduced at the waste water treatment works followed by precipitation of insoluble matter. Today, more than 95% of the waste water in Denmark is digested by microorganisms and they contribute significantly to organic matter in sludge. Mineralized N from this easily degradable fraction may, together with the content of ammonium-N, be used for prediction of the NFRV (Smith 1996). Second, the residues used in this experiment were applied just before the growing season. Larsen & Petersen (1993) applied the sewage sludge in November/December, 3–4 months prior to the growing season, thereby introducing the risk of reduction of NFRV by turnover and N leaching. This hypothesis is supported by the works of Edgar *et al.* (1995) and Misselbrook *et al.* (1996) who obtained the highest herbage yield with a late winter or spring application of liquid sludge, as a result of N leaching following autumn application (Misselbrook *et al.* 1996). Third, incorporation into the soil using a rotary harrow increased the residue-soil contact, and reduced the size of residue patches compared with incorporation by ploughing used in the 1974–79 experiment. This may have increased the turnover and release of N available for the crop. Finally, drought was recorded in several of the growing seasons during 1974–79, some with severe drought during July, some with a long period of precipitation deficit starting early in the spring. These conditions may have reduced the nitrogen mineralization and thereby the DM yields used for estimation of the NFRV. Drought was not observed in the present experiment, and precipitation deficit was neutralized on the sandy soil by irrigation.

Nitrogen offtake expresses plant-available N released from the residue-soil mixture, assuming complete uptake of mineral N and excluding N losses from the above-ground parts of the crop. Regarding SS1, SS2 and FYM, the higher NFRV for the DM : total-N relation compared with the N-offtake : total-N relation (Table 6) may be caused by the inclusion of more than just a nitrogen response in the DM yield. Such an additional manurial effect may be explained by other nutrients accompanied in proportion to the applied nitrogen, especially for the FYM for which the greatest difference between the estimates was observed. Similarly, additional effects on N-offtake may take place, but only caused by different pools and turnover rates of the applied residue N (Smith *et al.* 1998*b*).

Comparing the estimated NFRV for the ammonium-N based relations (Table 6) an additional manurial effect was obtained for FYM only. This was less pronounced than for the relations using total-N as the independent variable, but still assumed to be due to the high accompanying loads of P and K for this residue. On the other hand, mineralization and uptake of residue N may elapse continuously during the growing season being out of step with the growth stage related crop demand, as indicated by the difference in the ammonium-N based NFRV obtained for SS2 (Table 6). A similar result was not obtained for SS1, but the high NFRV based on ammonium-N indicates that both types of sewage sludge make some contribution through easily degradable N unaccounted for by the ammonium-N analysis. Smith *et al.* (1998*b*) found that this easily degradable fraction was mineralized within approximately 500 °Cd corresponding to the time until ear emergence in the present experiment. The separate waste water-treatment works produce sludge of a consistently homogeneous quality, but the differences in treatment processes are very important for the sewage sludge quality, and the differences between SS1 and SS2 in this experiment may be related to the anaerobic and aerobic digestion procedures. Also Smith *et al.* (1998*b*) obtained differences in the mineralization course for two dewatered digested sewage sludges.

Nitrate leaching from the CAN reference may be a potential error causing the high ammonium-N based NFRV. Using the EVACROP computing model (Olesen & Heidmann 1990) drainage below 1 m was estimated less than 20 mm in 1998 and 1999 during the first 4 weeks after spring barley emergence (E. M. Hansen, personal communication). In 2000 the estimated drainage was about 30 mm during the 3rd and 4th week after emergence. However, maximum uptake of applied mineral N may be expected within 4 weeks of application (Petersen 2001*b*). Thus, the potential erroneous nitrate leaching originating from CAN is assumed to be small, but an overestimation of NFRV cannot be excluded. Hypothetically, leaching of 50% of the nitrate applied in CAN implies that $q\{i\}=1$ in Eqn (1) for the mineral reference represents only three-quarters of the applied N, meaning that the NFRV estimates (based on ammonium-N in residue) have to be either compared with a reference having $q\{i\}=4/3$ or reduced by 33%. Even under such an unfavourable condition, the NFRV for SS1 and SS2 significantly exceed 100, emphasizing that the content of easily degradable N in sewage sludge makes an appreciable contribution to the ammonium-N based NFRV.

Despite common occurrence, FYM is not a well-defined reference regarding the content of plant-available N due to the diffuse production and storage compared with the centralized treatment of waste water. The ammonium-N concentration is often used for prediction of the NFRV for spring applied animal slurry incorporated for a cereal crop (Petersen 1996*a*), but the values below 100 obtained in the present study indicate that the ammonium-N analysis overestimates the NFRV. Thomsen & Olesen (2000) reported net immobilization of anaerobically stored FYM whereas the remaining N in composted FYM leads to net mineralization, indicating that the ammonium-N concentration on its own may be misleading for the prediction of the NFRV. Thus, the anaerobic storage conditions for the used FYM had an immobilization potential causing overestimation of NFRV. However, a more likely explanation may be N loss by ammonia volatilization caused by incomplete incorporation. The suboptimal incorporation was accepted in consideration of representative soil sampling for studies of organic contaminants (Petersen *et al.*, in press). The risk for ammonia volatilization was most significant for FYM, due to 2–3 times greater ammonium-N : total-N ratio (Table 4) and application rate (fresh weight as well as ammonium-N) (Table 5).

Taking these reservations into consideration, it may be concluded that the NFRV of the sewage sludges is similar to FYM. In contrast, the NFRV of Compost is poor. Despite an NFRV for Compost close to 100 using ammonium-N as a basis, these estimates are accompanied by a high variation linked to the very low ammonium content of this residue. The low NFRV based on total-N (Table 6) is in agreement with incubation experiments using composted sewage sludge/straw mixture (Smith *et al.* 1998*b*) who measured a net mineralizable fraction of 8–10% of total-N. Thus, more than 50 t/ha Compost has to be applied for covering the N demand of a cereal crop.

Combination of residue and mineral N

Annual application rates of 5–20 t/ha of organic matter corresponding to 200–1200 kg total-N/ha were common in previous field experiments (Johnston & Wedderburn 1974; Larsen & Petersen 1993), and the high loads of mineral-N were sufficient to cover the N requirement of most crops. The introduction of EC directives (CEC 1986, 1991*b*) has reduced the annual application rate, and experiments done during the 1990s used 1–10 t DM/ha corresponding to a total-N rate in the range 50–250 kg/ha (Edgar *et al.* 1995; Misselbrook *et al.* 1996; Smith *et al.* 1998*a*; the present study). With these restricted rates, the applied amounts of mineral-N in residues were suboptimal, introducing the requirement for supplementary mineral N to obtain satisfactory yields of cereal crops (Petersen 1996*b*). The desired residue rates were expected to supply 15–30 kg mineral N/ha corresponding to about 20% of the fertilizer norm. The remaining 80% was applied as supplementary mineral N to fulfil the norm and thereby prevent N deficiency. Normally, the risk of lodging is low using the nitrogen norm, but the level of supplementary N was slightly high for the oat crop.

A supplementary N rate four times the residue mineral N rate masks the N effect of the residue, causing variable residue effects on DM yield and N-offtake in relation to the 80%N reference treatment (Table 7) sometimes resulting in negative values for ANR (Table 8). An explanation of the variable results may be the ratio of immobilization capacity of the residue-soil mixture in relation to the amount of mineral N, either applied as mineral N or mineralized organic N during the growing season. The results in Tables 7 and 8 indicate that Compost has a high immobilization : mineralization ratio. This assumption is supported by findings of Ambus *et al.* (2002), who measured the N turnover of 3 × SS1 and 3 × Compost in bare-field microplots using soil from Askov. During the first 2 months after application, the initial gross immobilization was *c.* 1·3 and 2 mg N/kg soil/day for the SS1 and Compost, respectively. In contrast, the SS1 caused a three-fold higher gross N mineralization than Compost, viz. 13·8 *v.* 4·8 μg N/mg residue-N/day, indicating more readily mineralizable N in SS1 despite comparable C/N ratios. More readily available N was applied using the residues SS2 and FYM (Table 5), which was reflected in the yield for the FYM treatment (Table 7). On the contrary, only the N-offtake was increased for SS2, supporting the previous discussion on NFRV that the organic N in this residue was continuously mineralized. This may be the reason for the significant decrease in ANR for this residue when applying supplementary N. Reduced value of residue N was also reported by Petersen (1996*a*) for spring application of pig slurry supplied with mineral fertilizer. However, it is not possible to determine from either the present experiments or Petersen (1996*a*) whether the reason is reduced utilization of residue N or fertilizer N or both.

Phosphorus and potassium

The nutrients P and K were normally applied at rates corresponding to the offtake by the crops at harvest, and deficiency problems seldom occur in well-fertilized soils. The application rate of residue was determined on the basis of the legislative 40 kg P/ha/year, which often is higher than the offtake of a cereal crop. Thus, P deficiency may not have occurred in the experiment and therefore the effects on the crop P concentration as well as the offtake were insignificant. The use of a P balance sheet based on crop offtake may be an appropriate tool for decision of the P fertilizer rate.

Retention of K in the sewage sludge is not possible, resulting in a very low concentration in this residue. Thus, the applied amount of K was about one tenth of the crop offtake. This may have resulted in the lower K concentration in the crop of sewage sludge treatments, despite the K status of the soils being medium–high. In contrast, the applied amount of K in Compost and FYM was ample compared with the crop offtake, especially at the high application rate. The K-offtake is affected, on the one hand, by the possibility of luxury uptake and, on the other hand, K-loss by leaching from stem and leaves during the grain-filling period, and therefore K-offtake at maturity is not useful for predicting the crop K requirement.

CONCLUSION

The typical concentration of heavy metals in Danish municipal residues do not limit their agricultural use, but in practice the crop choice is limited to spring sown cereals. Application of the permitted annual rate is not able to provide an adequate N-supply to cereals, and supplementary mineral N is required. The need for supplementary N was reduced using a three-fold application rate equivalent to the permitted rate every 3 years. Thus, the aerobically digested sewage sludge (SS2) as well as the FYM yielded satisfactorily compared with the mineral reference treatments whereas the anaerobically digested sewage sludge (SS1) still needed some supplementary N.

The residue rate-independent estimates of nitrogen fertilizer replacement value (NFRV) demonstrate that sewage sludge has a potential manurial value comparable to FYM, taking supposed ammonia volatilization from FYM into account. In contrast to the total-N based NFRV of 49–68% for sewage sludge, the NFRV of Compost was less than 10%. To avoid over-consumption of fertilizer N and the risk of lodging, application of supplementary N has to take the NFRV of the residue into account. The ammonium concentration of organic residue is often used as an estimate for the NFRV, but in this experiment water extractable ammonium-N underestimates the crop response to digested sewage sludge, probably due to content of insoluble but easily degradable organic matter. The effect of residue application on P- and K-offtake was not substantial, but the applied amounts have to be taken into account in the fertilizer planning.

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