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Nitrogen Release Characteristics from Biosolids-Derived Organomineral Fertilizers

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This study investigated the availability of nitrogen (N) following soil application of a novel biosolids-derived organomineral fertilizer (OMF_{15}—15:4:4) in comparison with urea (46% N). OMF_{15} is produced by coating biosolids granules (particle size range: 1.10–5.50 mm in diameter) with urea and potash [60% potassium oxide (K_2O)], which increase the concentration of mineral N and potassium (K), respectively, resulting in a balanced fertilizer material suitable for application in cereal and grass crops. The study comprised two soil types of contrasting characteristics which were incubated over a period of 90 days at 25 °C and maintained near field capacity. Nitrogen was applied at rates equivalent to 0 (control), 150, and 300 kg ha^{-1}, and soil mineral N measured routinely using standard laboratory techniques. Results showed that the majority of N was released from OMF_{15} within 30 days from application (range: 40% to 72% of total OMF_{15}-N applied) with a further 10% to 28% in the following 60–90 days. OMF_{15} required an accumulated thermal time of 2250 degrees-day to release between 68% and 79% of the total OMF_{15}-N applied. From this, it was inferred that mineralization of the organic-N fraction in OMF_{15} is likely to progress beyond harvest of winter cereal crops in-field conditions in England. The results of this study aided the development of fertilization strategies for the best use of OMF in winter cereal and grass crops.

Keywords Biosolids granules, nitrogen availability, organomineral fertilizers (OMF), soil N dynamics, winter cereal and grass crops

Introduction

This article, the first in a series of two, reports the results of a laboratory investigation into the availability of nitrogen (N) following soil application of a novel biosolids-derived organomineral fertilizer (OMF) known as OMF_{15} (15:4:4) (Antille et al. 2013c). The second article (Antille, Sakrabani, and Godwin 2014) deals with aspects relating to phytoavailability of fertilizer-phosphorus following soil application of OMF_{15}. These two

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articles comprise experimental data, which aided the development of best management practices for the use of OMF in cereal and grass crops in England.

In England and Wales, the disposal of sewage sludge (biosolids) follows a number of routes which include, most importantly, recycling to farmland (72%), incineration (18%), land reclamation and restoration (6%), and landfill (1%), with a total production (dry solids basis) estimated at 1.6 million tonnes per year (DEFRA 2011). Sewage sludge production is set to increase in response to growth of population, adoption of cleaner technology for treatment of effluents, and stringent legislation, which restricts the opportunities for disposal (Moseley et al. 1998). The disposal route to farmland is the least expensive available option (Antille 2011, based on 2007 figures), and it is widely accepted in Europe that recycling of organic materials, including biosolids, to land is the best practicable environmental practice (Edge 1999). Recycling aims to complete the natural nutrients and carbon cycles (Chambers et al. 2003; Powlson et al. 2012) while it mitigates the demand for finite resources (Weikard and Seyhan 2009; Goss, Tubeileh, and Goorahoo 2013). Meeting recycling targets in the longer term will require that wastewater operators are committed to improving the quality (physical and chemical properties) of biosolids to: (1) reduce environmental concerns, and (2) increase their fertilizer value and acceptability by farmers. Technology is available for the production of OMFs which can be obtained by coating biosolids granules with urea (46% N) and potash [60% potassium oxide (K_2O)] to provide a balanced compound fertilizer material with suitable physical characteristics (Antille, Gallar-Redondo, and Godwin 2013a; Antille et al. 2013c).

The development of new fertilizer products requires determining key properties of the material that affect their agronomic and environmental performances. Previous studies (e.g., Bowden and Hann 1997; Cordovil, Cabral, and Coutinho 2007) had indicated the need to investigate nutrients availability of organic materials applied to agricultural soils with a view to determining their fertilizing value (e.g., Sommers 1997). Smith, Woods, and Evans (1998) highlighted the need to better understand the role and fate of nutrients in soils amended with organic materials, which is an important consideration for economic (return from applied nutrients) and environmental reasons [leaching and gaseous losses of N, and phosphorus (P) transport to water courses]. This requires determining the nutrient release characteristics from applied OMF to increase fertilizer use efficiency by crops and to mitigate nutrient losses to the environment. In the UK, current recommendations for spring (February to April) applications of thermally dried biosolids to agricultural soils (DEFRA 2010) are based on the assumption that about 15% (surface-applied) or 20% (soil-incorporated) of the total N content in the material is available to the next crop following application irrespective of soil type.

Soil incubation techniques have been extensively used to determine nutrients availability from organic materials applied to agricultural soils (e.g., Chescheir, Westerman, and Safley 1986; Cordovil et al. 2005; Fangueiro, Chadwick, and Bol 2008). A possible disadvantage of these methods is the occurrence of large concentrations of inorganic-N in non-leached systems, which can disrupt mineralization processes and underestimate potentially mineralizable N content (Garau, Felipó, and Ruiz de Villa 1986). Despite the numerous studies on the subject and current agronomic guidelines (DEFRA 2010), there is a pressing need for robust quantitative data that describe soil behavior and determine the potential fertilizer value of organic amendments, particularly for novel fertilizer materials.

This article adopts the approach by Honeycutt and Potaro (1990), and brings together incubation dataset and combines it with measured field-scale temperature data to determine the N availability from biosolids-derived OMFs. The objective of this study was to determine the availability of N from OMF_{15} (15:4:4) following application to the soil under controlled laboratory conditions of temperature and soil moisture content (SMC) by comparing the release characteristics of OMF_{15}-N with urea-N. It was hypothesized that following soil application of OMF, the urea-N fraction would be released relatively rapidly whereas the organic-N fraction (biosolids-N) would be released at a slower rate. The development of a strategy for the best use of OMF in cereal and grass crops (Antille, Sakrabani, and Godwin 2013b; Antille, Sakrabani, and Godwin 2014) benefited from the results reported in this work.

Materials and Methods

Description of the Soils

The soils used in the study were a *Cottenham series* sandy loam and a *Holdenby series* clay loam (King 1969) (Table 1). These soils were selected because they have contrasting physical and chemical characteristics, suitable for the incubation study and due to their frequent occurrence in the NW region of England (Ragg et al. 1984), which is the area of operation of a leading water company in this part of the country. The soils were collected from the farm at Cranfield University (Silsoe, England); they had been under winter cereal and oilseed rape cropping (Antille 2011), and subjected to standard fertilization regimes (DEFRA 2010). The following determinations were conducted in soil samples taken from the farm prior to the start of the experiment: soil texture (British Standard 1377 Section 2.0 1990), SMC corresponding to field capacity (FC) at 0.05 bar (British Standard 7755 Section 5.5 1999), and soil bulk density (Blake and Hartge 1986) which was required to determine quantities of fertilizer added to incubation pots.

Fertilizer Materials

The availability of nitrogen in OMF_{15} (15:4:4) was compared with that of urea (46% N) following soil application. The fertilizers were applied at rates equivalent to 0 (control), 150, and 300 kg ha^{-1} of N to provide a suitable range that would cover N-fertilization regimes recommended for most arable and forage crops in England (DEFRA 2010). The actual fertilizer quantities applied to individual pots are shown in Table 2 . The experiment was replicated three times (trials 1–3) and all trials were conducted under the same experimental conditions. All treatments, including controls, were replicated four times. Therefore, the values reported for each treatment correspond to the mean of the three experiments and include 12 observations ($n = 12$). The differences in the application rate

Table 1

Soil textural analysis, soil moisture content corresponding to field capacity (mass basis) and soil bulk density for the two soils used in the incubation studies (sampling depth: 200 mm)

Determination	Soil type and series	
Soil textural analysis	Clay loam (*Holdenby*)	Sandy loam (*Cottenham*)
Sand (%, w w^{-1})	46	67
Clay (%, w w^{-1})	25	13
Silt (%, w w^{-1})	29	20
Field capacity (%, w w^{-1}, 0.05 bar)	30.4	26.6
Soil bulk density (g cm^{-3})	1.22	1.34

Table 2
Fertilizer added to incubation pots for the study of N availability from soil application of OMF_{15} (15:4:4) and urea (46% N)

	Fertilizer material (mg of fertilizer product per pot)					
Soil type	Clay loam			Sandy loam		
Fertilizer	OMF_{15}	OMF_{15}	Urea	OMF_{15}	OMF_{15}	Urea
Treatment	1st trial	2nd and 3rd trials	All	1st trial	2nd and 3rd trials	All
Control (zero-fertilizer)	0	0	0	0	0	0
150 (kg N ha^{-1})	125.1	76.6	26.7	113.9	69.7	24.3
300 (kg N ha^{-1})	250.2	153.2	53.5	227.8	139.5	48.7

of OMF_{15} between the first, and the second and thirds trials (Table 2) are due to differences in the concentration of N in the fertilizer batches available for the study. A detailed characterization of the fertilizer materials used is given in Antille et al. (2013c). Differences in fertilizer quantities applied to the clay loam and sandy loam soils are due to differences in the values of soil bulk density encountered in each of the two soil types (Table 1).

Soil Incubation

Incubation pots of 0.25 L capacity were filled with 200 g of air-dried soil previously ground to pass a 2 mm sieve, mixed with fertilizer and wetted-up with de-ionized water to reach FC. Subsequently, the pots were placed in an incubator in the absence of light and the temperature in the chamber adjusted to 25 ± 0.1 °C (Tejada, Benitez, and Gonzalez 2002). SMC was regularly replenished by adding de-ionized water on a mass basis (Smernik, Oliver, and McLaughlin 2004), and the soil maintained between FC and 75% of FC throughout the experiment. This technique has been satisfactorily used in earlier studies (e.g., Flavel and Murphy 2006). The study was conducted for a period of 90 days following the guidelines outlined in OECD (2002). Conversion of soil mineral N (SMN) from mg kg^{-1} to kg ha^{-1} (field equivalent) was done based on the density of the soil and assuming a depth of 200 mm (Table 1).

Measurements and Analyses

Soil sampling was conducted at the start of the experiment (before fertilizer application), and every 30 days thereafter. The sampling technique was non-destructive (Johnston 2008) as the same pots and soil were used throughout the experiment over the 90 days period. The soils were analyzed for determination of total carbon (C) (British Standard 7755 Section 3.8 1995) and total N (Dumas 1831; British Standard EN 13654-2 2001) prior to fertilizer application and at the end of the 90 days incubation period. SMN (MAFF 1986 Method No.: 53) was determined prior to fertilizer application and every 30 days thereafter. Results are reported in the form of (mean) SMN recorded at times corresponding to sampling events at 0, 30, 60, and 90 days, respectively. Soil organic matter (SOM) (MAFF 1986 Method No.: 56) and soil pH (MAFF 1986 Method No.: 32) were determined before fertilizer application. The percentage N available ($\%N_{available}$) was estimated with Equation (1), relative to total N applied (Kokkora, Hann, and Godwin 2008) at 30, 60, and 90 days:

$$\%N_{available} = \frac{\text{Mineral N fertilised soil} - \text{Mineral N control soil}}{\text{Total N applied}} \times 100 \quad (1)$$

where $\%N_{available}$ is net available nitrogen in soil determined at 30, 60, and 90 days after fertilizer application. SMN includes ammonium nitrogen ($NH_4{}^+$-N) and nitrate nitrogen ($NO_3{}^-$-N) (Mengel and Kirkby 1987) therefore both N fractions were computed for the calculation of total mineral N at any given time.

Statistical Analyses

Statistical analyses were conducted using GenStat (2007) Release 10.1 and involved repeated measurement of analysis of variance (ANOVA) and least significant differences (LSD) to compare the means using a 5% probability level ($P < 0.05$). The analyses conducted were graphically verified by means of residual plots and normalization of the data was not required.

Results and Discussion

Initial Soil Analyses

Table 3 shows the results of soil chemical analyses conducted prior to the start of the experiment, which corresponded to the baseline level. The amounts of $NH_4{}^+$-N recorded in the analyses were consistently low and negligible compared with $NO_3{}^-$-N which suggested a rapid conversion of $NH_4{}^+$ to $NO_3{}^-$ under the prevailing experimental conditions.

Nitrogen Release Characteristics

Figure 1 shows SMN recorded at 0, 30, 60, and 90 days from the start of the experiment. There were significant differences in SMN between control and treatments ($P < 0.001$). Differences observed between the sandy loam and clay loam soils ($P < 0.001$) can be explained by differences recorded initially in the overall fertility status of the two soils,

Table 3
Soil analyses conducted prior to the start of the experiments in the laboratory

Determination	*n*	Sandy loam	Clay loam
Soil pH	3	6.90 ± 0.24	6.20 ± 0.19
SOM (%, w w^{-1})	3	3.80 ± 0.01	5.90 ± 0.02
Total C (%, w w^{-1})	3	1.80 ± 0.14	1.83 ± 0.01
Total N (%, w w^{-1})	3	0.14 ± 0.01	0.16 ± 0.1
C:N ratio	3	11.4 ± 0.35	10.6 ± 0.17
SMN (mg kg^{-1})	3	14.6 ± 0.14	31.1 ± 0.58

The standard deviation (*SD*) is shown as ± the mean value.

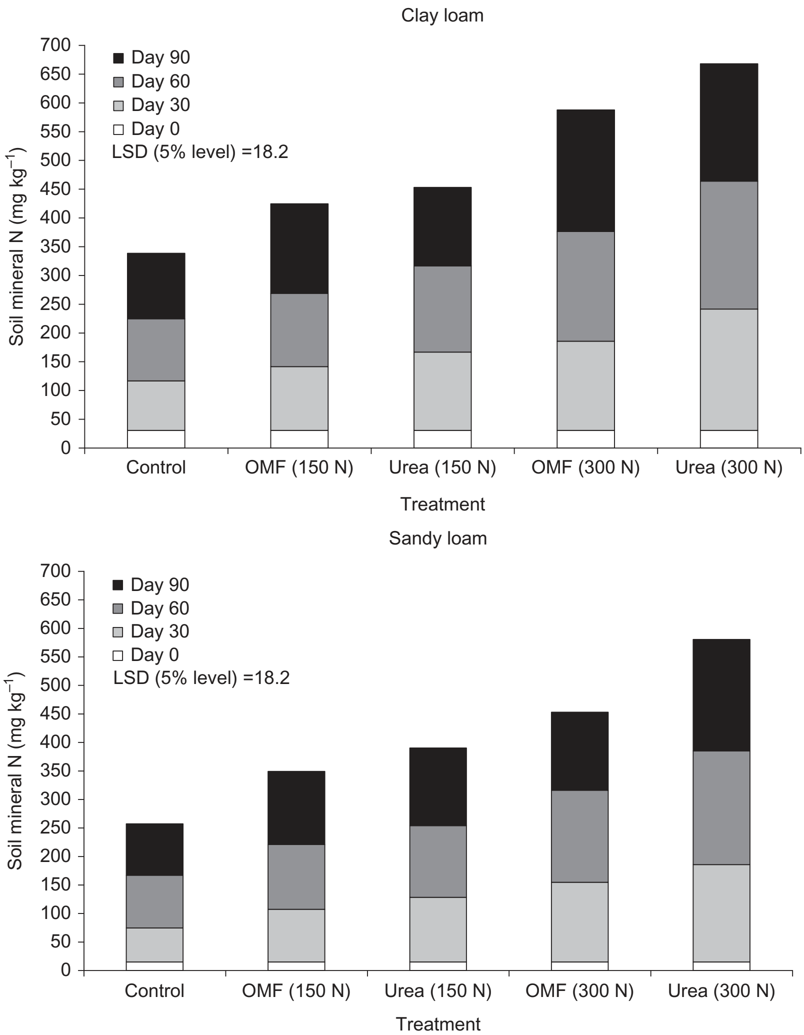

Figure 1. Soil mineral N recorded at days 0, 30, 60, and 90 after fertilizer application to clay loam (*top*) and sandy loam (*bottom*) soils, respectively (P = 0.43, n = 12). OMF is OMF_{15} followed by the corresponding N application rate expressed in kg ha^{-1}.

particularly, SOM levels (Table 3). Overall, there were significant differences with respect to fertilizer type and N application rates (P-values < 0.001), which were not observed when factoring in soil type (P-values > 0.05). This suggested that changes recorded in SMN as a result of fertilizer type or N application rate were of similar order of magnitude in both soil types when compared to controls. The interaction fertilizer type $\times$ N rate was significant ($P < 0.05$) but this was mainly due to the effect of urea-N applied at a field equivalent rate of 300 kg ha^{-1}. There was an overall effect ($P < 0.001$) of fertilizer type with respect to time. Throughout the experiment, urea-treated soils showed consistently greater SMN values than soils treated with OMF_{15}, particularly, at days 30 and 60 (range of 17% to 26% higher). At day 90, SMN levels were approximately the same in both treatments ($\approx$165 mg kg^{-1}, mean value across soil types and N application rates; n = 16). Gaseous losses of N by denitrification or volatilization can result in errors when providing estimates of N availability from organic amendments, as determined by laboratory incubation techniques (Smith, Woods, and Evans 1998). However, these losses of N are usually low when biosolids are thoroughly mixed with the soil (Terry, Nelson, and Sommers 1981), which agrees with the technique employed in this study. Therefore, it is reasonable to assume that N losses by volatilization of NH_3 were minimized as in this experiment the fertilizer materials were thoroughly mixed with soil in incubation pots.

Studies (Honeycutt, Potaro, and Halteman 1991; Smith, Woods, and Evans 1998; Griffin and Honeycutt 2000) have satisfactorily employed a technique based on accumulated thermal time (degrees-day, d°C) to predict the release of N from organic sources applied to soil. This approach provides a useful link between data obtained under controlled laboratory conditions and patterns of mineral N formation that may be expected in-field conditions (Honeycutt, Potaro, and Halteman 1991; Smith, Woods, and Evans 1998; Griffin and Honeycutt 2000). The relationship between thermal units and N mineralization is influenced by soil water content (Honeycutt, Zibilske, and Clapham 1988, Honeycutt, Potaro, and Halteman 1991; Honeycutt and Potaro 1990). Ability to predict N availability from organic amendments applied to soil is an important consideration for nutrient management, to enable the simultaneous occurrence of N supply and crop N demand (Griffin and Honeycutt 2000).

Based on the earlier works quoted above, an attempt was made to link the data derived from this incubation study with soil temperature series recorded in the field to provide estimates of expected N availability under such conditions. However, it is recognized that N release characteristics in-field conditions may be different given the numerous of factors influencing this process such as presence of crops, N uptake, and changing temperature and soil moisture conditions. Soil temperatures were recorded in the field at Cranfield University (Silsoe, England) at a depth of 200 mm and on an hourly basis over a period of 2 years corresponding to 2008 and 2009. On average, the period between 1 March and 30 June, which coincides approximately with the main growth period of winter cereal crops in Bedfordshire (England), showed an accumulated thermal time (d°C, base temperature = 0 °C) equivalent to 1593 d°C. This value is the average as calculated from mean monthly soil temperatures recorded in 2008 and 2009 (authors' own data).

The soils used in the incubation study reached this accumulated thermal time of 1593 d°C at approximately 60 days from the start of the experiment. From Figure 1, it is deducted that at day 60, net SMN levels for the clay loam soil treated with OMF_{15} at 150 and 300 kg ha^{-1} of N were 20 mg kg^{-1} (field equivalent = 50 kg ha^{-1}) and 83 mg kg^{-1} (field equivalent = 200 kg ha^{-1}), respectively. For the sandy loam soil, net SMN levels recorded at day 60 for the same N application rates were 21 mg kg^{-1} (field equivalent = 55 kg ha^{-1}) and 70 mg kg^{-1} (field equivalent = 185 kg ha^{-1}), respectively. This simple analysis suggests that OMF_{15} has some potential to supply relatively large amounts of (available) N to the crop during the main part of the growing season. OMF_{15} required an accumulated thermal time of 2250 d°C to yield the percentages available N reported in Table 4 at day 90 from fertilizer application. The majority of N that was released from OMF_{15} became available within 30 days from the start of the experiment, which is attributed to a rapid release of N from the fraction carrying urea. At this time (30 days after fertilizer application), the accumulated thermal time was 750 d°C. Nitrogen applications to winter cereal crops in England are normally conducted between late February and middle of April comprising a window of about 60 days (Berry et al. 2004; DEFRA 2010). Based on the same soil temperatures dataset indicated earlier for the field, the accumulated thermal time for this period (late February to middle of April) reported an average of

Table 4
Available nitrogen relative to total N applied as fertilizer for soils treated with OMF_{15} (15:4:4) and urea (46% N).

$\%N_{available}$ (%, w w^{-1})				
Soil type	Clay loam			
Fertilizer type	OMF_{15}		Urea	
Time/N-rate	150 kg N ha^{-1}	300 kg N ha^{-1}	150 kg N ha^{-1}	300 kg N ha^{-1}
30 days	40.5	56.6	81.6	99.9
60 days	35.6	67.3	65.8	93.0
90 days	68.4	79.4	39.3	93.0
Mean	48.2	67.7	62.2	95.2
SD	17.7	11.4	21.4	4.1
Soil type	Sandy loam			
Fertilizer type	OMF_{15}		Urea	
Time/N-rate	150 kg N ha^{-1}	300 kg N ha^{-1}	150 kg N ha^{-1}	300 kg N ha^{-1}
30 days	58.6	71.6	96.1	99.1
60 days	37.4	61.3	59.1	95.4
90 days	68.3	70.3	83.8	94.0
Mean	54.8	67.8	79.7	96.2
SD	15.8	5.6	18.7	2.6

SD is standard deviation. Use n = 12.

585 d°C for 2008 and 2009. The value of 585 d°C falls earlier than that corresponding to the first sampling event conducted at 30 days after fertilizer application, which is equivalent to 750 d°C. This makes it impossible to produce estimates of available N corresponding to that thermal time (585 d°C) with the information available. Therefore, it is acknowledged that soil sampling at shorter time intervals as well as within 7 days from the start of the experiment would have provided a valuable indication of N transformations that may be expected in the field.

The accumulated thermal time of 2250 d°C, recorded at 90 days in the incubation study, is likely to be reached in early August in-field conditions in Bedfordshire (England). This suggests that mineralization of OMF_{15}-N will continue beyond senescence of winter cereal crops, which has implications upon fertilizer N management. On the one hand, subsequent crops sown in early autumn may benefit from residual N mineralized between pre- and post-harvest of previous crop. This can result in reduced N requirements for the establishment of some crops in soils with relatively low soil N supply (SNS), e.g., SNS ≤ 2 (DEFRA 2010). On the other hand, unutilized N that is mineralized between pre-harvest and crop establishment is prone to leaching during autumn, which can occur, for example, in late-sown or second winter wheat crops. Hence, application of OMF_{15} in early spring (late February) should be considered as part of the fertilization strategy to allow for sufficient time for the organic-N fraction in OMF_{15} to mineralize during the main growth period of the crop. Nitrogen carried over after harvest should be accounted for when computing N requirements of the following crop in the rotation in soils with low SNS, e.g., oilseed rape established in the autumn. When the total N rate is to be split into two applications, which is the recommended practice for N rates exceeding 120 kg ha^{-1} (DEFRA 2010), OMF_{15} may be applied in the first dressing (late February) and supplemented with a mineral N source in the second dressing, typically around middle of April. The second application of N should be adjusted according to the N requirement of the crop, stages of growth and overall fertility status of the soil at the time of fertilizer application to minimize risk of lodging later in the season (Tottman and Broad 1987; Berry et al. 2004; Antille 2011).

Due to the scale and characteristics of this experiment, the set of observations outlined earlier requires validation with data obtained in-field conditions. However, preliminary studies at the field-scale (Antille 2011) showed that a first winter wheat crop benefited from combined applications of mineral N as urea-ammonium nitrate solution (28% N), and OMF_{15} in the first and second dressings, respectively. The earlier study showed that grain yield in OMF_{15}-treated crop was not significantly different ($P > 0.05$) from the crop that received mineral N only (yield range: 10.4–10.7 t ha^{-1}). These results demonstrate the advantage of using both N sources as part of the fertilization strategy. However, the timing of fertilizer application requires further investigation to optimize fertilizer use efficiency.

Despite the relatively large differences recorded in SOM levels between the two soil types (Table 3), it can be seen from Table 4 that mean SMN values in the sandy loam soil were comparable to those recorded for the clay loam soil. Incubation studies conducted by Cordovil et al. (2005) demonstrated that sandy loam soils amended with organic materials can undergo intense N mineralization initially but approximately 30 days after application; N mineralization rates may decrease significantly. This pattern of N mineralization agrees with results reported by El-Gharous, Westerman, and Soltanpour (1990) and it helps to explain the relatively high levels of N available encountered in the sandy loam soil. This soil was therefore able to sustain higher N mineralization rates than the clay loam soil, which reflects a textural effect on N availability following soil application of OMF_{15}. This result contrasts with current recommendations for application of thermally dried biosolids to agricultural soils (DEFRA 2010) which assume a fixed value of 15% (surface application) or 20% (soil incorporation) N availability, relative to total N applied, for all soils in spring applications (February to April). From Table 4 it can be inferred that availability of OMF_{15}-N relative to that of urea-N ranged approximately between 69% and 77% on average over the 90 days incubation period, depending on soil type and N application rate.

Conclusions

The findings from this study suggest that OMF_{15} has potential to supply relatively large amounts of available nitrogen during the main part of the growing season if fertilizer application is conducted in early spring. Nitrogen release from OMF_{15} occurs mainly within 30 days following soil application accounting to about 40% to 70% relative total nitrogen applied as fertilizer, with a further 10% to 30% approximately in the following 30–60 days, respectively.

OMF_{15} requires an accumulated thermal time of 2250 degrees-day to release about 70% to 80% of total OMF_{15}-N following soil application depending on soil type and N application rate. Given that this accumulated thermal time is likely to be reached in early August in-field conditions in Bedfordshire (England), it is expected that mineralization of the organic-N fraction in OMF_{15} would continue after harvest of winter cereal crops in that part of the country. Therefore, OMF_{15} may be applied in the early part of the spring to enable for simultaneous occurrence of fertilizer nitrogen availability and high rates of uptake by crop. The use of thermal units provided a useful link between data obtained under controlled laboratory conditions and expected patterns of mineral N formation from applied OMF_{15} in-field conditions.

Based on the results of this study, and those reported by the authors on fertilizer-phosphorus (Antille, Sakrabani, and Godwin 2014) there appears to be potential for further development of OMF products. The product formulation and nutrient release characteristics make OMF_{15} suitable for application in winter cereal and grass crops. The timing of OMF application requires further investigation in-field conditions to enable optimizing crop responses and minimizing nutrient losses to the environment.

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