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#### Review

## A critical review of nitrogen mineralization in biosolids-amended soil, the associated fertilizer value for crop production and potential for emissions to the environment



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#### HIGHLIGHTS

- Mineralizable N decreased with increasing biological stabilization of biosolids.
- Air-drying or extended storage resulted in low total and mineralizable N contents.
- Internationally the mineralizable N of similar biosolids types was highly variable.
- Differences in upstream wastewater treatment processes may affect mineralizable N.
- Soils in warmer climates may have adapted to access more recalcitrant forms of N.

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#### ABSTRACT

International controls for biosolids application to agricultural land ensure the protection of human health and the environment, that it is performed in accordance with good agricultural practice and that nitrogen (N) inputs do not exceed crop requirements. Data from the scientific literature on the total, mineral and mineralizable N contents of biosolids applied to agricultural land under a wide range of climatic and experimental conditions were collated. The mean concentrations of total N (TN) in the dry solids (DS) of different biosolids types ranged from 1.5% (air-dried lime-treated (LT) biosolids) to 7.5% (liquid mesophilic anaerobic digestion (LMAD) biosolids). The overall mean values of mineralizable N, as a proportion of the organic N content, were 47% for aerobic digestion (AeD) biosolids, 40% for thermally dried (TD) biosolids, 34% for LT biosolids, 30% for mesophilic anaerobic digestion (MAD) biosolids, and 7% for composted (Com) biosolids. Biosolids air-dried or stored for extended periods had smaller total and mineralizable N values compared to mechanically dewatered types. For example, for biosolids treated by MAD, the mean TN (% DS) and mineralizable N (% organic N) contents of air-dried materials were 3% and 20%, respectively, compared to 5% and 30% with mechanical dewatering. Thus, mineralizable N declined with the extent of biological stabilization during sewage sludge treatment; nevertheless, overall plant available N (PAN = readily available inorganic N plus mineralizable N) was broadly consistent across several major biosolids categories within climatic regions. However, mineralizable N often varied significantly between climatic regions for similar biosolids types, influencing the overall PAN. This may be partly attributed to the increased rate, and also the greater extent of soil microbial mineralization of more stable, residual organic N fractions in biosolids applied to soil in warmer climatic zones, which also raised the overall PAN, compared to cooler temperate areas. It is also probably influenced by differences in upstream wastewater treatment processes that affect the balance of primary and secondary, biological sludges in the final combined sludge output from wastewater treatment, as well as the relative effectiveness of sludge stabilization treatments at specific sites. Better characterization of biosolids used in N release and mineralization investigations is therefore necessary to improve

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*Abbreviations:* AD, anaerobic digestion; AeD, aerobic digestion; BMPs, best management practices; *CEC*, cation exchange capacity; CoGAP, code of good agricultural practice; Com, composted; DMAD, dewatered mesophilic anaerobic digestion; DS, dry solids; DTA–AD, Dewatered thermophilic aerobic–anaerobic digestion; GHG, greenhouse gas; GWHC, gravimetric water holding capacity; IPCC, International Panel on Climate Change; LMAD, liquid mesophilic anaerobic digestion; LT, lime treated; MAD, mesophilic anaerobic digestion; N<sub>e</sub>, nitrogen equivalency; NLBAR, nitrogen limited biosolids application rate; N<sub>m</sub>, mineralizable N; NVZs, nitrate vulnerable zones; PAN, plant available nitrogen; TA–AD, thermophilic aerobic–anaerobic digestion; TD, thermally dried; TN, total N; VS, volatile solids; WAS, waste activated sludge; WWTP, wastewater treatment plant.

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comparison of system conditions. Furthermore, the review suggested that some international fertilizer recommendations may underestimate mineralizable N in biosolids, and the N fertilizer value. Consequently, greater inputs of supplementary mineral fertilizer N may be supplied than are required for crop production, potentially increasing the risk of fertilizer N emissions to the environment. Thus greater economic and environmental savings in mineral N fertilizer application are potentially possible than are currently realized from biosolids recycling programmes.

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#### 1. Introduction

Biosolids are the treated and stabilized solid organic residual byproducts (sewage sludge) from urban wastewater treatment. Large quantities of biosolids are produced globally and it is estimated that Europe and the United States of America (USA) produce approximately 10 million t dry solids (DS) year<sup>-1</sup> (Gendebien et al., 2008) and 7.2 million t DS year<sup>-1</sup> (NEBRA et al., 2007), respectively, whereas Australia, having a smaller population density, produces 330,000 t DS year<sup>-1</sup> (ANZBP, 2013).

Land application is the favoured practice for biosolids management internationally as it takes advantage of the soil improving properties and the fertilizer replacement value of the material whilst avoiding the environmental and economic impacts associated with other disposal options such as landfill or incineration. In the EU, on average 36% of the total biosolids produced are used in agriculture, although the range of utilization varies considerably, from no land application (Greece, Netherlands, Romania, Slovenia, Slovakia), to 50% or more (Belgium, Denmark, Spain, France, Ireland, United Kingdom (UK)) (Gendebien et al., 2008). In the UK almost 80% of biosolids are applied to land, with agriculture being the dominant route (77%) and a small fraction is used in land restoration (2%) (Water UK, 2010). In the USA, approximately 49% of the biosolids produced are used in agriculture (44.5%), landscaping or horticulture (2.5%), land restoration (1.5%) or forestry (0.5%) (NEBRA et al., 2007), whilst in Australia, 69% of biosolids produced are used in agriculture (59%), composting for landscaping (6%) or land rehabilitation (4%), and 20% is stockpiled, with the remainder sent to landfill (3%), discharged to the ocean (1%) or managed by an unspecified route (7%) (ANZBP, 2013). As in other regions of the world, the implementation of beneficial use strategies for biosolids across

Australia varies widely. For example, 34% of the biosolids produced in the State of Victoria are used in agriculture or are composted for land application, and the remainder is currently stockpiled. By contrast, all of the biosolids produced in the State of South Australia are used beneficially on land (ANZBP, 2013).

A substantial body of research has been undertaken to understand the risks to public health and the environment from biosolids land application, centred around pathogens, heavy metals, nutrient enrichment and organic pollutants (Smith, 1996). This research provides the foundation to international biosolids controls on management practices, treatment requirements, microbiological and pollutant quality criteria and application rates that ensure sustainable land application (ADAS, 2001; CEC, 1986; DoE, 1996; NWQMS, 2004; US EPA, 1993). Research about the potential risks of biosolids land application is ongoing and focuses on areas such as emerging organic contaminants (Clarke and Smith, 2011; Rigby et al., 2015) and the potential selection of antibiotic resistant genes in the environment (Munir and Xagoraraki, 2011).

The land application of biosolids in developed countries is highly controlled to prevent risks to public health and the environment. Sewage sludge must be treated and 'stabilized' to reduce odour, pathogen content and vector attraction, and is usually also mechanically dewatered or airdried to reduce bulk, to generate a product acceptable for beneficial use. Biosolids treatment may be through either one or a combination of the following generic methods:

- (1) Biological processes (anaerobic/aerobic digestion, composting)
- (2) Chemical processes (lime treatment)
- (3) Physical processes (pasteurization, thermal hydrolysis, thermal drying, air/solar drying).

Different sludge treatment processes are classified depending on the extent of pathogen removal achieved. For instance, mesophilic anaerobic digestion (MAD) must achieve a 2 log<sub>10</sub> reduction in Escherichia coli and is described as a 'conventional' treatment process by the UK Safe Sludge Matrix (ADAS, 2001), which is synonymous with the 'Class B' pathogen reduction requirements of US EPA (1993). Mesophilic anaerobic digestion is extensively used by the Water Industry for the treatment of sewage sludge and is particularly favoured due to the production of renewable energy in the form of biogas containing methane. Other methods, described as 'enhanced' treatment processes (for example, lime treatment or heating or drying at high temperatures), provide a greater degree of pathogen removal and must achieve  $\geq 6 \log_{10}$  reduction in E. coli (ADAS, 2001), which is effectively equivalent to the 'Class A' pathogen reduction requirements of US EPA (1993). Sludge treated to the conventional or Class B standard is used following a multi-barrier approach to protect human health from the transmission of infectious pathogens, by also establishing land use, cropping and harvesting restrictions to allow the natural attenuation of pathogens in the environment to take place (Lang et al., 2007). Enhanced-treated, or Class A biosolids, on the other hand, can be used for a wider range of crops, and in this case, less stringent controls on planting and harvesting intervals apply. In some jurisdictions, biosolids treated to this standard are permitted for use without restriction as they provide a single, complete barrier to pathogen transmission (US EPA, 1993). Increasingly, pretreatments are applied to improve sludge digestibility, biogas yields, and/or pathogen removal by anaerobic digestion (AD) processes. For example, thermal hydrolysis achieves both, pre-pasteurization destroys pathogens primarily and sonication increases digestibility (through secondary sludge biomass disruption), but does not affect pathogen destruction. Extensive descriptions of different sewage sludge treatment processes can be found in Gurjar (2001), Spinosa and Vesilind (2001) and Sanin et al. (2011). There is a diverse range of process options for sewage sludge treatment and understanding the extent to which these influence the nutrient content, availability and fertilizer value of different biosolids products is a prerequisite to support sustainable land application practices and agricultural utilization in particular.

Biosolids are a source of the major plant nutrient elements: nitrogen (N), phosphorus (P), sulphur (S), magnesium (Mg), and calcium (Ca) and trace elements such as copper (Cu), zinc (Zn) and boron (B) (Chambers et al., 2003; Christie et al., 2001), although they are not a significant source of potassium (K) due to the elutriating effect of wastewater treatment on soluble components (Pierzynski et al., 1994). Nitrogen is generally the most limiting nutrient to crop production (Graham and Vance, 2000), and the addition of industrially produced N based fertilizers in agriculture has contributed to increased crop yields globally (Tilman et al., 2002). Whilst the nutrients supplied in standard inorganic fertilizers are readily available for crop uptake, the N supplied by biosolids is present as both inorganic (principally ammonium  $(NH_4^+)$ ) and occasionally nitrate (NO<sub>3</sub><sup>-</sup>)) and organic forms. The availability of N for crop uptake in biosolids-amended soil is therefore dependent upon the inorganic N content and the forms and availability of organic N present. The available fraction of organic N in biosolids may be influenced by several factors such as the method of treatment or stabilization, soil properties, and environmental conditions. Estimating the nutrient release properties of biosolids is critical, as it is for any form of organicallybased nutrient supplement/replacement to mineral fertilizer, to avoid crop deficiencies and economic loss or the over-application of nutrients and the potential detrimental environmental effects associated with offsite movement of accumulated nutrients through leaching, runoff and erosion (Burgos et al., 2006; Elliott et al., 2002; Esteller et al., 2009; Ojeda et al., 2006). Other mechanisms promoting N losses to the environment include gaseous emissions via ammonia (NH<sub>3</sub>) volatilization, which are a function of biosolids type and application method, or denitrification, that are potentially promoted by the interaction between soil type and biosolids type and environmental conditions (Donovan and Logan, 1983; Pu et al., 2010; Rigby ad Smith, 2014; Robinson and Polglase, 2000). Hence, the investigation of N dynamics in biosolids-amended soils to determine appropriate application rates and best management practices according to local circumstances and conditions has been a focus of extensive international research.

Land application of biosolids, and other biowastes, is promoted as an opportunity to close the nutrient loop (Green Alliance, 2007), reduce consumption of raw materials and contribute towards food security, and the practice is consistent with developing policies to increase resource efficiency (EC, 2011). At the same time, it is essential to minimize adverse effects on the environment and to enhance soil quality. Despite the body of work investigating N supply from biosolids, there remains significant scope to improve the efficiency of N management in biosolids (and other sources of N) applied to soil and to further control nutrient losses to air and water (Al-Dhumri et al., 2013; Eldridge et al., 2008; Pu et al., 2008; Rigby et al., 2010). Therefore, the overall aim of this study is to critically review the literature on N behaviour in land applied biosolids under a range of environmental conditions, and to complete a robust assessment of N management recommendations for biosolids to improve N use efficiency. The specific objectives are to examine: (1) the impact of sewage sludge treatment method on the total, mineral and mineralizable N contents of treated biosolids, (2) the impact of different environmental factors on the rate and extent of N mineralization in biosolids-amended soil, (3) the rate and extent of crop uptake of N in biosolids-amended soil, and (4) the losses of N from biosolids-amended soil.

#### 2. International biosolids nitrogen limits and availability estimates

When biosolids are used in agriculture, best management practices (BMPs) are followed to ensure environmental protection. In some countries, such as Australia, these are specified directly in the controls on biosolids management (NSW EPA, 1997; Victoria EPA, 2004; DEC, 2012). Other regions recommend, and in specific circumstances, enforce, land management requirements for nutrients across a more comprehensive range of farming practices and manure and fertilizer inputs, including biosolids, such as the Code of Good Agricultural Practice (CoGAP) to

protect water, soil and air in the UK (Defra, 2009a) and the European Union (EU) Nitrates Directive (CEC, 1991), which, amongst other measures, limits N inputs in organic manures to agricultural land in all EU Member States.

Best management practices require the assessment of land suitability for receiving manures as well as direct controls on their application (e.g. Defra, 2009a; EPA Victoria, 2004; US EPA, 1995). For example, spreading manure on land susceptible to waterlogging or flooding should be avoided. Land suitability also depends upon an evaluation of soil structure, pH, nutrient status, slope and water table depth. Sites considered as generally acceptable for manure application may also require the location of buffer zones to protect sensitive ground or surface water sources from potential nutrient emissions. Controls on the timing and management of N applications in organic manures are also adopted with the aim of reducing leaching and atmospheric losses. For example, applications should not coincide with heavy rainfall events, and for fallow land, they should be close to seeding to reduce nutrient losses (Defra, 2009b; EPA Victoria, 2004; US EPA, 1995). Biosolids are often rich sources of ammonium–N (NH₄–N), particularly those from AD stabilization, and ideally should be rapidly incorporated into the soil after spreading to reduce NH<sub>3</sub> volatilization, as well as potential run-off losses. In some Australian States, the guidelines on biosolids management indicate that where incorporation is not possible, for example in 'no-till' farming or forestry, the key risks from surface-application must be addressed by adhering to slope restrictions and providing buffer zones (DEC, 2012; EPA Victoria, 2004). Additionally, closed periods may be introduced when application is not permitted at times of the year when there is a significant risk of NO<sub>3</sub><sup>-</sup> leaching, typically during the late summer to mid-winter period. For example, in the UK (Defra, 2009b), a closed period prohibits spreading of organic manures with high readily available N content (e.g. slurry, poultry manure and liquid digested sludge) in designated Nitrate Vulnerable Zones (NVZs), representing catchment areas at risk of water pollution by  $NO_3^-$  leaching (CEC, 1991).

Biosolids controls in Australia set maximum application rates based on an estimated plant available N (PAN) content using Eq. (1):

$$PAN = NH_4 - N + [NO_3 - N + NO_2 - N] + N_m$$

$$\tag{1}$$

where:  $NH_4-N$ , ammonium-N;  $NO_3-N$ , nitrate-N;  $NO_2-N$ , nitrite-N and  $N_m$ , mineralizable N (the fraction of organic N that is converted into inorganic N in the soil).

The rate of agricultural application of biosolids is determined by the 'Nitrogen Limited Biosolids Application Rate' (NLBAR) (NSW EPA, 1997; DEC, 2012), where the amount of PAN applied in biosolids (calculated according to Eq. (1)) must not exceed the crop N requirement in the year of application.

In other countries, such as the UK, biosolids controls (DoE, 1996; SI, 1990) require that use on land is consistent with good agricultural practice and separate guidance is issued on the fertilizer value (Defra, 2010). In addition, limits on inputs of N to farmland may be stipulated in separate regulations that apply to biosolids, as well as other sources of N. For instance, the EU Nitrates Directive (CEC, 1991) establishes maximum limits on inputs of total N (TN) in organic manures, including sewage sludge, within designated NVZs. In the UK, there is a field-based application limit for TN of 250 kg ha<sup>-1</sup> in NVZs for biosolids and other non-livestock derived organic manures (Defra, 2009a), and the combined PAN of organic wastes and mineral fertilizer N application should match crop requirements (Defra, 2009b, 2010). Similarly in the USA, the Code of Federal Regulations 40 Part 503 (US EPA, 1993) states that biosolids applications must not exceed crop N requirements and estimates of PAN and mineralizable N are required to determine application rates.

The US EPA Process Design Manual for the land application of biosolids (US EPA, 1995) provides example mineralization factors in the first year of application, as a proportion of the organic N fraction, equivalent to 10% for composted biosolids, 20% for anaerobically digested biosolids and 30% for aerobically digested biosolids (Table 1). However, these are

provided for general guidance and the manual recommends that the agronomic rate should be calculated on a site-specific basis using mineralization factors recommended by State 'Agricultural Experiment Stations' and the 'Cooperative Extension Service' if relevant research has been conducted, as mineralizable N is dependent upon local climate conditions and soil type (US EPA, 1995). The loss of NH<sub>3</sub> due to volatilization is also factored into the PAN value, which ranges from 0–50% depending on the biosolids type and application method (US EPA, 1995). For surface applied liquid or dewatered sludge, 50% of the NH<sub>4</sub>–N content is assumed to be lost due to NH<sub>3</sub> volatilization, but when liquid sludge is injected, all of the NH<sub>4</sub>–N content is assumed to be available (US EPA, 1995).

The mineralizable N values recommended by the New South Wales biosolids guidelines in Australia (NSW EPA, 1997) for different biosolids types are in the same range as those indicated in the USA Process Design Manual, although slightly smaller values are used for anaerobically and aerobically digested biosolids (Table 1). In the UK, PAN values for organic wastes, including biosolids, are given in the Fertilizer Manual (Defra, 2010). Instead of estimates of mineralizable N, these recommendations give overall PAN values (mineralizable N plus inorganic N) taking account of a wider range of different biosolids types, including digested liquid, digested cake, composted, lime stabilized and thermally dried biosolids. The recommendations referring to digested biosolids specifically apply to anaerobically digested types since aerobic digestion is not practised in the UK Water Industry. For the purpose of comparison with the US and Australia, the mineralizable N values have been estimated by subtracting the concentration of mineral N supplied in the different biosolids types from their PAN values, which are both given in Defra (2010), and expressing this value as a proportion of organic N (Table 1). For digested biosolids, the mineralizable N values are significantly lower than those provided in the US and Australia (6% of the organic N for digested cake and 8% for digested liquid). For lime stabilized and thermally dried biosolids the mineralizable N values are 10.5% and 16% of the organic N, respectively. The mineralizable N value given for compost is 10% organic N, and is equivalent to US and Australian values. Following the review of the literature presented in Sections 4–7, these differences in N availability estimates between countries are examined further in Section 8.

The UK recommendations (Defra, 2010) also take into account the effects of application method and timing, soil type and autumn/winter rainfall following application. For digested cake, when incorporated into the soil, the percentage of TN available to the next crop following the application of biosolids is 10% when applied in the autumn on a

#### Table 1

Mineralizable nitrogen values (first year of application) presented in biosolids fertilizer recommendations.

Biosolids type	Mineralizable	nitrogen (N <sub>m</sub> ) (% organic nit	trogen)					
	[Values indicated in brackets are Plant Available N (PAN) <sup>a</sup> % total N]							
	US	New South Wales, Australia	UK					
	US EPA (1995)	NSW EPA (1997) <sup>b</sup>	Defra (2010) <sup>c</sup>					
Aerobically digested	30	25	-					
Anaerobically digested	20	15	-					
Digested liquid			8 (45)					
Digested cake			6 (20)					
Composted	10	10	10 (15)					
Lime stabilized			10.5 (20)					
Thermally dried			16 (20)					

<sup>a</sup> Plant Available N (PAN) = mineral N (NH<sub>4</sub>-N + NO<sub>3</sub>-N) + mineralizable N.

<sup>b</sup> These controls only apply in the State of NSW, other Australian States have their own controls, although many adopt the same values as NSW. The Australian National Guidelines (NRMMC, 2004) do not provide direct values or limits.

<sup>c</sup> Applied in Spring and incorporated. Potentially mineralisable N was calculated from values of potentially available N (%) minus values of readily available N (% total N) supplied in biosolids provided in Defra (2010), and expressed as a proportion of organic N.

sandy/shallow soil and increases to 20% when applied in Spring (on all soils). However, for surface applications, it is assumed that only 15% of TN is available when applied in the Spring period.

The USA guidelines suggest that, following the year of application, the amount of N mineralized decreases by 50% each year until 3% is reached (US EPA, 1995). The recommendation in the UK is that digested cake supplies 10% of the TN applied in the second year and around 5% in the third year (Defra, 2010). In Australia, no recommendations are given for the organic N mineralization in subsequent years; however, it is suggested that the residual N in the soil should be taken into account on sites that receive frequent applications of biosolids (NSW EPA, 1997).

Hence, there are notable differences between international recommendations for N management for land-applied biosolids, particularly with respect to the mineralizable N or PAN values. In Sections 3–6 we review evidence in the scientific literature on mineralizable N and PAN in biosolids-amended soil for a range of biosolids types, soil and environmental conditions. In the light of this evidence we complete a critical assessment of mineralizable N values in Section 8 to assist improvements in the N use efficiency of biosolids application to land.

#### 3. Total and mineral nitrogen concentrations in biosolids

The TN concentrations for all biosolids types reported in the literature are listed in Tables 2–8 and were in the range 0.7–15% on a DS basis; the overall mean and median TN values were 4.1% DS and 4.4% DS, respectively. On average, therefore, biosolids typically contain more TN than cattle manure, which has a TN content of 1.0–3.9% DS (mean 2.5% DS) (Antil et al., 2009; Chadwick et al., 2000; Defra, 2010; Hristov et al., 2009), but less than poultry manure, which contains 4.4–5.3% of TN in the DS (mean 4.9% DS) (Chadwick et al., 2000; Nicholson et al., 2003; Defra, 2010). Whereas most of the N in commercial N fertilizers is in readily available inorganic forms, the greater part of N in the majority of biosolids types is organically combined in proteins, chitins, amino sugars and nucleic acids (Pierzynski et al., 2005), and must be mineralized before becoming available for plant use; this process is discussed in Section 4.

The concentration of inorganic N in biosolids was 0–50% of the TN concentration (mean 9.7% TN; median 6.6% TN). Most of the inorganic N was present in the form of  $NH_4$ –N whereas nitrate–N ( $NO_3$ –N) was rarely present.

Seven categories of sewage sludge/biosolids were identified: 1) untreated sludge (raw primary and activated sludge), which is usually considered unsuitable for use in agriculture and therefore falls outside the definition of biosolids; 2) aerobic digestion (AeD) (treatment temperature, e.g. thermophilic, was rarely specified); 3) MAD (two pre-treatment processes were also identified: pre-pasteurization and thermal hydrolysis); 4) thermophilic aerobic–anaerobic digestion (TA–AD); 5) thermally dried (TD) (raw, AeD or MAD); 6) lime-treated (LT) (raw, AeD or MAD); and 7) composted (Com). These categories were further sub-divided depending on whether they were liquid, dewatered, or had undergone air-drying or an extended period of storage (in a stockpile or lagoon) (Tables 2–8).

The biodegradation of organic matter, and the chemical processes occurring during biosolids treatment, influenced the overall TN content. For example, the mean TN concentration for raw (Table 2), AeD (Table 3), MAD (Table 4) and TD (Table 6) biosolids (excluding airdried or stockpiled/lagoon-stored sludges/biosolids) was >4% DS, whereas Com (Table 8), and dewatered LT (Table 7) biosolids contained 2.2% and 3.3% TN on average in the DS, respectively. The smaller TN values determined for Com and dewatered LT biosolids are due to a combination of process losses and dilution from the addition of bulking agents such as woodchips or municipal green waste during composting, and alkali reactants during lime stabilization, which are typically added at a rate of at least 30% DS during lime treatment of sewage sludge (Aubain et al., 2001).

Sewage sludge treatment and dewatering processes also affected the proportion of mineral and organic N in the biosolids. Dewatered mesophilic anaerobic digestion (DMAD) biosolids had a mean inorganic N content of 14.4% of the TN content (Table 4), which was greater compared to dewatered raw sludge (3.2% TN) (Table 2) due to mineralization of organic N during digestion. Certain processes, such as lime treatment, thermal drying or composting, which promote losses of NH<sub>3</sub> through increased pH, temperature or mechanical agitation, respectively, produced biosolids with a mean inorganic N content of 4.6–7.2% TN (Tables 6–8). Dewatered, thermophilic aerobic-anaerobic digestion (DTA–AD) biosolids had a greater inorganic N content than other dewatered biosolids types, equivalent to 21% of TN (Table 5), which may be due to enhanced biodegradation rates as a consequence of high temperatures during treatment (Cogger et al., 2004; Gilmour et al., 2003); however, there were very few examples of this type of

#### Table 2

Total nitrogen (TN) and mineral N (ammonium–N,  $NH_4$ –N and nitrate–N,  $NO_3$ –N) values reported in the scientific literature for raw (unstabilized) sludge, arranged according to dewatering method (liquid; dewatered; air-dried/stockpiled/lagoon stored). Values reported on a dry solid (DS) basis, apart from mineral N (% TN).

Further description	n	TN %		NH <sub>4</sub> –N mg k	g <sup>-1</sup>	NO3-N 1	ng kg <sup>-1</sup>	Mineral N	Reference
		Mean	Range	Mean	Range	Mean	Range	%TN	
AS, liquid	1	6.0							Sommers et al. (1976)
Primary undigested, liquid	1	3.6		5700				15.8	Smith and Tibbett (2004)
AS, alum dosed, liquid	1	6.5		624				1.0	Corrêa (2004)
AS, liquid	1	1.4		3900				27.9	Beltrán-Hernández et al. (1999)
Liquid mean		4.4		3408				14.9	
Liquid median		4.8		3900				15.8	
Liquid range		1.4-6.5		624-5700				1.0-27.9	
Raw, dewatered	1	6.1		1916		2.8		3.2	Rigby et al. (2009)
AS, air-dried	1	2.5							Nasmith and Mckay (1918)
AS, dried	11	5.6	4.7-6.3						Noer (1926)
AS, dried	1	2.0		1200					Muller (1929)
AS, dried	8	5.4	4.7-6.4						DeTurk (1935)
Primary, air/oven-dried	1	4.4		2160				4.9	Parker and Sommers (1983)
AS, air/oven-dried	2	5.6	4.2-7.0	2710	1700-3720			4.8	Parker and Sommers (1983)
Primary & AS, air/oven-dried	1	4.5		850				1.9	Parker and Sommers (1983)
Oxidation ditch and clarifier, air dried	1	4.9		8000				16.3	Cogger et al. (2004)
Storage lagoon, dewatered	2	2.5	2.3-2.6	2400	2100-2700			9.6	Gilmour et al. (2003)
Activated, solar dried, alum dosed	3	6.5		803				1.2	Corrêa (2004)
Air-dried mean		4.4		2821		2.8		8.1	
Air-dried median		4.7		2280		2.8		4.9	
Air-dried range		2-6.5		803-8000				1.2-16.3	

AS: activated sludge.

Total nitrogen (TN) and mineral N (ammonium–N, NH<sub>4</sub>–N and nitrate–N, NO<sub>3</sub>–N) values reported in the scientific literature for aerobic digestion (AeD) biosolids, arranged according to dewatering method (unspecified; liquid; dewatered; air-dried/stockpiled/lagoon stored). Values reported on a dry solid (DS) basis, apart from mineral N (% TN).

Further description	n	TN %		NH <sub>4</sub> -N mg kg <sup>-</sup>	- 1	NO <sub>3</sub> -N n	ng kg <sup>-1</sup>	Mineral N	Reference
		Mean	Range	Mean	Range	Mean	Range	%TN	
	38 (TN) 33 (NH <sub>4</sub> -N) 8 (NO <sub>3</sub> -N)	4.9	0–5–7.6	950		300	7–830	2.6	Sommers (1977)
	1	2.7		1800				6.7	Bozkurt et al. (2006)
Liquid	1	6.8		884				1.3	Magdoff and Chromec (1977)
Liquid	1	4.8							Adiei and Rechcigl (2002)
Thermophilic, liquid	1	8.5		27,700		< 0.1		21.3	Wang et al. (2003b)
Liquid	1	4.8							Sigua et al. (2005)
Liquid mean		6.2		14,292				8.0	
Liquid median		5.8		14,292				4.7	
Liquid range		4.8-8.5		884-27,700				1.3-21.3	
Dewatered	1	6.1		2460				4.0	Robinson and Polglase (2000)
Thermophilic, dewatered	6	4.3						20.6	Cogger et al. (2001)
Centrifuge, drying bed	1	5.6		1400				2.5	Gilmour et al. (2003)
Dewatered	1	5.8		11,200				19.3	Petersen (2003)
Drying bed	2	5.2	4.7-5.6	1500	1-2000			2.9	Cogger et al. (2004)
Dewatered	1	4.0		7900		0.3		19.8	Eldridge et al. (2008)
Dewatered	3	6.2		2800		2		4.5	Pu et al. (2008)
Belt-pressed	1	7.3		6700				9.2	O'Shaughnessy et al. (2008)
Dewatered by filter-press	6	4.6	4.3-4.9						Esteller et al. (2009)
Dewatered	3	6.1		2612		10		4.3	Pu et al. (2012)
Dewatered mean		5.7		4572		4.1		8.3	
Dewatered median		5.3		1980		27		4.4	
Dewatered range		4.0-7.3		1400-11,200		0.3-10		2.5-19.8	
Air/oven-dried	1	2.7		1360		44		5.2	Parker and Sommers (1983)
Air-dried	6	2.9	1.5-4.7	990	102-2378	53.3		3.6	Serna and Pomares (1992)
Air-dried	1	5.4		241				0.4	Hseu and Huang (2005)
Dewatered (stockpiled 18 months)	1	2.0		900		780		8.3	Al-Dhumri et al. (2013)
Air-dried mean		3.3		873		292		4.4	
Air-dried median		2.8		945		53.3		4.4	
Air-dried range		2.0-5.4		241-1360		44-780		0.4-8.3	

biosolids found in the literature reflecting its generally limited deployment in practice as a sludge treatment technology.

The variations in TN and mineral N contents within specific biosolids types may be partially explained depending on whether the biosolids were dewatered, and by the method of dewatering. Liquid biosolids, for example liquid mesophilic anaerobic digestion (LMAD) biosolids, generally had greater TN (mean 7.5% DS; range 4.3-15% DS) and inorganic N contents (mean 33.9% TN; range 14.7-44.2% TN) compared to dewatered types. Thus, dewatering removes a substantial fraction, equivalent to up to 60%, of the NH<sub>4</sub>-N content of anaerobically digested biosolids. In general, biosolids dewatered by air-drying had lower TN and inorganic N contents compared to mechanical dewatering. This is because organic N mineralization continues during the extended treatment times involved in air-drying sludge and losses of mineral N by NH<sub>3</sub> volatilization are also encouraged by air-drying processes. For example, the mean TN content in air-dried MAD biosolids was 2.8% DS, whereas mean TN in examples of mechanically dewatered MAD biosolids was 5.0% DS. Rouch et al. (2011) found that air-drying LMAD biosolids and storage reduced the TN concentration to <1% DS, an overall decrease of 80% in comparison to fresh liquid digested biosolids.

#### 4. Mineralizable nitrogen content and methods of determination

#### 4.1. Nitrogen mineralization

Nitrogen mineralization occurs through the hydrolysis and biodegradation of organic matter when the N content of the substrate exceeds the metabolic requirements of microbial cells for N, leading to the release of  $NH_4^+$  ions, which is a process mediated by heterotrophic soil microorganisms (Pierzynski et al., 2005). Ammonium ions may be directly absorbed by plant roots or oxidised by aerobic, nitrifying bacteria to  $NO_2^-$  and ultimately to  $NO_3^-$ , which is also used directly by plants (Pierzynski et al., 2005). The proportion of organic N in biosolids that becomes plant available is referred to as the 'mineralizable' fraction. On the basis that most of the N is usually present in organic forms (this is the case for dewatered biosolids for instance), predicting the mineralizable fraction of biosolids N is necessary to calculate the PAN and fertilizer replacement value.

#### 4.2. Laboratory and in situ soil incubation

Laboratory-based, aerobic soil incubation techniques are useful to determine the overall proportion of mineralizable biosolids-N. There are two methods commonly employed; in the first approach, biosolidsamended soil is packed into a column, maintained under controlled conditions and periodically flushed under vacuum with a dilute salt solution (typically 0.01 M CaCl<sub>2</sub>) to leach mineralized inorganic N (e.g. Magdoff and Amadon, 1980). In the second system, direct sampling and extraction of incubated biosolids-soil mixtures, to measure the different forms of inorganic N, is carried out during the incubation period (e.g. Tester et al., 1977). Both methods generally give similar results; however, a potential disadvantage of the leaching procedure, as noted by Parker and Sommers (1983), is that soluble organic N is also leached along with inorganic N, and may underestimate the mineralizable N content. By contrast, Garau et al. (1986) and Wang et al. (2003a) found the leaching procedure overestimated mineralizable N compared to the non-leached method, despite the removal of soluble organic N in the leachate. This may be because soil leaching also removed soluble organic carbon, which reduced the potential for microbial immobilization of mineral N in the soil, thus increasing the overall apparent net N mineralization (Wang et al., 2003a). The duration of reported laboratory incubation studies ranged between 42-480 days with evidence suggesting that most of the mineralizable organic N was released within the first 50 days, particularly where warmer temperatures (for example,  $\geq 25$  °C) were applied (Smith et al., 1998a;

Total nitrogen (TN) and mineral N (ammonium–N, NH<sub>4</sub>–N and nitrate–N, NO<sub>3</sub>–N) values reported in the scientific literature for mesophilic anaerobic digestion (MAD) biosolids, arranged according to dewatering method (unspecified; liquid; dewatered; air-dried/stockpiled/lagoon stored). Values reported on a dry solid (DS) basis, apart from mineral N (% TN).

Further description	n	TN %		NH <sub>4</sub> –N mg kg	-1	NO3-N mg	$\rm g~kg^{-1}$	Mineral N	Reference
		Mean	Range	Mean	Range	Mean	Range	%TN	
	85(TN) 67(NH <sub>4</sub> -N) 35(NO <sub>3</sub> -N)	5	0.5-17.6	9400	120–67,600	520	72-4900	19.8	Sommers (1977)
	1	2.9		1580		2		5.5	Epstein et al. (1978)
Liquid	4	6.3	4.3-7.8	26,900	9500-42,800			42.7	Gilmour et al. (2003)
Liquid	1	4.3		6300		< 0.01		14.7	Wang et al. (2003b)
Liquid	1	4.3		19,000				44.2	Smith and Tibbett (2004)
Liquid	4	15	11.3-18.2						Rouch et al. (2011)
Liguid mean		7.5		17,400		< 0.01		33.9	
Liquid median		5.3		19,000		< 0.01		42.7	
Liquid range		4.3-15		6300-26.900				14.7-44.2	
Dewatered	3	7.0							Wong et al. (1998)
Dewatered	2	47	41-45	1105	910-1300			24	Robinson and Polglase
Deviatered	2	-1.7	4.1 4.5	2004	510 1500	110		2.4	(2000)
Dewatered	1	3.9		2604		110		7.0	Polprasert (2002)
Dewatered	2	3.5	3.3-3.8	7050	6900-7200	N.D.		20.1	Adegbidi and Briggs (2003)
Belt-filter	3	6.3	5-7.4	12,433	10,900-14,600	<0.1		19.7	Gilmour et al. (2003)
Dewatered	5	6.0	5.2-6.8	8940	6200-13,100			14.9	Gilmour et al. (2003)
Dewatered	3	3.2		5500				17.1	Petersen (2003)
Dewatered	9	5.6		9121		0.01		16.3	Morris et al. (2003)
Dewatered	3	5.9	5-6.7	11,300	9000-13,000			19.2	Cogger et al. (2004)
Dewatered	1	5.3		4700				8.9	Smith and Tibbett (2004)
Dewatered	2	3.0		2311	1567-3005	5.7	0.3-11	7.7	Whatmuff et al. (2005)
Dewatered	1	3.7		2000				5.4	Mendoza et al. (2006)
Centrifuge dewatered	1	47		6750				14.4	Oieda et al. (2006)
Dewatered	6	54	44-63	3145	1300-6108	69	02-27	5.8	Pritchard and Collins (2006)
Dewatered	3	61	111 010	1200	1500 0100	2	012 217	2.0	Pu et al (2008)
Centrifuge dewatered	1	5.2		9100		-		17.5	$\Omega$ 'Shaughnessy et al. (2008)
Dewatered	1	6.0		9236		< 0.1		15.4	Rigby et al. (2009)
Dewatered	2	4.0		20,000		20.1		50.1	Cottschall et al. (2009)
Dewatered	1	4.6		4300		12		9.4	$V_{\rm res}$ et al. (2010)
Dewatered	1	4.0 6.1		4300		1.2		5.4 20.9	$P_{\rm H}$ of al. (2010)
Dewatered mean	5	5.0		7026		14		20.8	Fu et al. (2012)
Dewatered median		5.0		6750		18.0 E 7		14.4	
Dewatered median		0.5 2 7		1105 20.000		D.7		14.9	
Dewalered fallge	11	20	11 24	1105-20,000	56 2760	0.01-110	10 2100	2.0-50.1	Darling and Commons (1082)
Oven/an-urieu	11	2.0	1.1-3.4	11/1	20-3700	441	16-2100	8.0	Parker and Sommers (1983)
Alf-difed	6	2.2	2.1-3.4	2080	32-3084	.0.1		9.5	Seria and Poinares (1992)
Drying bed	6	2.6	1.6-3.6	4300	5000	<0.1		16.5	Barbarick and Ippolito (2000)
Drying bed	2	4.5	4.3-4.7	3100	1900-4300	<0.1		6.9	Gilmour et al. (2003)
Lagoon	2	1.9	1.6-2.2	3150	2300-4000			16.6	Gilmour et al. (2003)
Drying bed	1	4.3		4000				9.3	Cogger et al. (2004)
Lagoon, dewatered	1	2.2		4000				18.2	Cogger et al. (2004)
Air-dried	1	6.4		824		76.2		1.4	Hseu and Huang (2005)
Lagoon	1	0.89		306		1486		20.1	Heemsbergen et al. (2007)
Agitated air-dried	1	1.3		90		2000		16.1	Heemsbergen et al. (2007)
Drying pans (8–9 months)	3	4.4	3-5.8						Rouch et al. (2011)
Drying pans (11–12 months)	2	2.8	2.7-2.8						Rouch et al. (2011)
Stockpiled (12–36 months)	16	0.8	0.4-1.8						Rouch et al. (2011)
Stockpiled (1–3 months)	1	2.3		320		84		1.8	Al-Dhumri et al. (2013)
Air-dried/stockpiled mean		2.8		2217		405		11.6	
Air-dried/stockpiled median		2.3		2590		0.1		12.8	
Air-dried/stockpiled range		0.8-6.4		90-4300		0.1-2000		1.4-20.1	
Thermal hydrolysis pre-treatment, DMAD	2	5.1	5.14-5.39	8578	8368-8788	<0.01		16.8	Morris et al. (2003)
Pre-pasteurized, DMAD	2	4.1		5637		< 0.01		14.1	Morris et al. (2003)

DMAD: dewatered mesophilic anaerobic digestion.

Smith and Durham, 2002). However, longer incubation periods may be necessary to fully characterize the mineralization patterns of the most recalcitrant forms of organic N.

First order kinetic models are frequently applied to describe the net change in mineralized N concentration in soils with incubation time (Bernal et al., 1998; Garau et al., 1986; Smith et al., 1980; Stanford and Smith, 1972). However, improved predictions of the rate and extent of N mineralization when organic wastes are added to soils may be provided by kinetic models that consider degradable organic N as two or more separate pools with different mineralization rate constants (Deans et al.,

1986; Lindemann and Cardenas, 1984; Smith et al., 1998a, 1998b). In this case an exponential equation is fitted to the data, and separate rate constants are estimated for slowly decomposable or recalcitrant organic N and readily decomposable or labile organic N pools. Generally the largest fraction of mineralizable organic matter is released rapidly in biosolids-amended soil. Gilmour et al. (1996a) proposed a sevenday test to predict long term decomposition of biosolids and constructed a model to estimate the percentage of C in the rapidly and slowly mineralized fractions with the rate constants based on the percentage of C mineralized within 7 days. Predictions of long-term decomposition

Total nitrogen (TN) and mineral N (ammonium–N, NH<sub>4</sub>–N and nitrate–N, NO<sub>3</sub>–N) values reported in the scientific literature for dewatered thermophilic, aerobic–anaerobic digestion (DTA–AD) biosolids.

Values reported on a dry solid (DS) basis, apart from mineral N (% TN).

Further description	n	TN%	$\rm NH_4-N~mg~kg^{-1}$	$NO_3$ -N mg kg <sup>-1</sup>	Mineral N %TN	Reference
Belt-filter dewatered	1	4.7	9700	<0.1	32.6	Gilmour et al. (2003)
Dewatered	1	4.7	10,000		24.8	Cogger et al. (2004)
Mean		4.7	9850	<0.1	28.7	
Median		4.7	9850	<0.1	28.7	
Range			9700-10,000		24.8-32.6	

were found to correspond well to observed values. This study was focused specifically on C mineralization; however, there may be potential to develop a similar short-term soil incubation test to also predict N mineralization patterns in biosolids-amended soil.

A number of researchers have also used *in situ* incubation methods to investigate N mineralization processes in the field (Hanselman et al., 2004; Eldridge et al., 2008). Hanselman et al. (2004) compared four field incubation methods to a non-leached laboratory incubation procedure: i) a buried bag method, which involved filling plastic bags with soil and burying at a shallow depth in the field for several days or weeks; ii) a covered-cylinder method using a perforated PVC cylinder covered by a cap to exclude rainfall; iii) an ion-exchange resin to capture mineralized N as it leaches from soil contained within a PVC cylinder; and iv) a newly developed soil and ion-exchange resin trap which was designed to improve drainage in comparison to the standard resintrap method. Soils were amended with poultry manure, biosolids and yard waste compost. However, as may be expected with field barrier techniques, the soil water content dynamics were not fully representative of ambient water conditions in field soil and affected the N mineralization measurements. Buried bag methods initially gave similar results to laboratory incubation measurements of mineralized N; however, degradation of the bags over time resulted in low N recovery and increased water content in the bag. The alternative soil-resin trap maintained ambient field water conditions; however, recovery of N was low during extended incubation periods, and the resin cartridge required regular replacement (frequency < 45 days) to improve estimates of N mineralization.

#### 4.3. Nitrogen budgets and mass balance

Biosolids N mineralization has been estimated from N budgets and mass balances in amended soils measured under field or glasshouse conditions, with or without the presence of a crop (Smith and Hadley, 1989; Pu et al., 2008, 2012). This resource intensive method estimates

the proportion of TN originating from the biosolids in the different N fractions, presented in Eq. (2), and the biosolids N contribution is calculated by subtracting the N present in each fraction in unamended control treatments. This allows a specific assessment of the rate and extent of N mineralization relevant to the particular conditions of the experiment. Nevertheless, quantification of all of the potential N pools operating within the system presents particular technical challenges.

$$N_{m} = \left[ \left( N_{crop} + N_{min1} + MBN + N_{d} + N_{v} \right) - \left( N_{min0} \right) \right] / N_{org} * 100$$
(2)

where: N<sub>m</sub>, mineralizable N (% biosolids organic N); N<sub>crop</sub>, N taken up by the crop (% TN); N<sub>min1</sub>, mineral N of biosolids origin remaining in the soil profile at the end of the growing season (%TN); MBN, microbial biomass N (%TN); N<sub>d</sub>, N lost by denitrification (%TN); N<sub>v</sub>, N lost through ammonia volatilization (%TN); N<sub>min0</sub>, mineral N originally present in the biosolids (%TN); N<sub>org</sub>, proportion of organic N originally supplied in the biosolids (% TN).

A fundamental assumption of the N mass balance approach is the equivalency of N dynamics in background, unamended control and amended soils. However, inputs of mineral or organic N to soil can have a priming influence on the mineralization of native soil organic N, thus complicating and potentially overestimating the biosolids mineralizable N pool mass balance (Kuzyakov et al., 2000).

#### 4.4. Nitrogen equivalency method

The nitrogen equivalency  $(N_e)$  of biosolids N is calculated from the relative crop response patterns (in terms of yield or N uptake) obtained for biosolids N and inorganic fertilizer N supplied at increasing rates to controlled experimental plots under field conditions (Al-Dhumri et al., 2013; Barbarick and Ippolito, 2000, 2007; Bowden et al., 2007; Morris et al., 2003; Rigby et al., 2010; Smith et al., 2002). Only the nutrient under investigation (N) is limiting in the soil and all other nutrients

#### Table 6

Total nitrogen (TN) and mineral N (ammonium–N, NH<sub>4</sub>–N and nitrate–N, NO<sub>3</sub>–N) values reported in the scientific literature for thermally dried (TD) biosolids. Values reported on a dry solid (DS) basis, apart from mineral N (% TN).

Further description	n	TN %		NH4-N mg kg	5-1	NO3-N mg kg	g <sup>-1</sup>	Mineral N	Reference
		Mean	Range	Mean	Range	Mean	Range	%TN	
Activated	1	5.4							Rudolfs (1928)
AS, alum dosed	3	6.5		356				0.6	Corrêa (2004)
AeD	1	5.5		3310				6.0	Eldridge et al. (2008)
MAD	63	4.5							Cogger et al. (2001)
MAD	9	5.6	4.5-6.6	2233	700-3200	< 0.01		4.0	Gilmour et al. (2003)
MAD	5	4.7	4.0-5.1	1162	800-8576	< 0.01		2.5	Morris et al. (2003)
MAD	3	5.4	4.6-6.6	2300	1000-3000			4.3	Cogger et al. (2004)
MAD	1	4.5		3360				7.5	Ojeda et al. (2006)
MAD	1	4.5		3360		0.04		7.5	Tarrasón et al. (2008)
MAD	1	3.4		517		6.85		1.5	Rigby et al. (2009)
MAD	3	5.9		4540		4		7.7	Pu et al. (2012)
Mean		5.1		2349		3.6		4.6	
Median		5.4		2300		4		4.3	
Range		3.4-6.5		356-4540		0.04-6.85		0.6-7.7	

AS: activated sludge; AeD: aerobic digestion; MAD: mesophilic anaerobic digestion.

Total nitrogen (TN) and mineral N (ammonium–N, NH<sub>4</sub>–N and nitrate–N, NO<sub>3</sub>–N) values reported in the scientific literature for lime treated (LT) biosolids, arranged according to dewatering method (liquid; dewatered; air-dried/stockpiled/lagoon stored). Values reported on a dry solid (DS) basis, apart from mineral N (% TN).

Further description	n	TN %		NH <sub>4</sub> -N mg k	g <sup>-1</sup>	NO <sub>3</sub> -N	mg kg <sup>-1</sup>	Mineral N	Reference
		Mean	Range	Mean	Range	Mean	Range	%TN	
Liquid	2	3.3	3.2-3.4	1500	1200-1800			4.5	Gilmour et al. (2003)
MAD, liquid	1	4.0							Adjei and Rechcigl (2002)
AS, alum dosed, liquid	3	4.0		93.8				0.2	Corrêa (2004)
LMAD	1	4.8							Sigua et al. (2005)
Liquid mean		4.0		797				2.4	
Liquid median		3.5		1236				4.0	
Liquid range		3.3-4.8		93.8-1500				0.2-4.5	
Centrifuged	1	4.6		2800				6.0	Gilmour et al. (2003)
Raw, dewatered	3	1.7	1.13-2.01	718	< 0.01			4.3	Morris et al. (2003)
AeD, dewatered	1	3.7		600				1.6	Mendoza et al. (2006)
Raw, dewatered	1	2.4		2171		43		9.2	Rigby et al. (2009)
MAD, dewatered	1	2.1		2000				9.5	Magdoff and Chromec (1977)
MAD cake	1	3.9							Adjei and Rechcigl (2002)
MAD, belt-filter press, lime added to cake	2	4.7	3.9-5.4	700	400-1000			1.5	Cogger et al. (2004)
MAD, dewatered	1	2.0		827.0		4.9		4.2	Whatmuff et al. (2005)
MAD, dewatered	1	3.9						0.0	Sigua et al. (2005)
MAD, belt filter	4	3.7	2.6-5.4	1175	400-2100	< 0.1		3.2	Gilmour et al. (2003)
MAD, dewatered	1	1.8		1400				7.8	Gilmour et al. (2003)
Dewatered mean		3.3		1459		24.0		4.8	
Dewatered median		3.7		1288		24.0		4.2	
Dewatered range		1.8-4.7		600-2800				0-9.5	
Raw, air/oven-dried	1	2.3		62		42		0.3	Parker and Sommers (1983)
MAD, air/oven-dried	2	0.7	0.5-0.8	308	26-590	59	28-90	4.4	Parker and Sommers (1983)
Air-dried mean		1.5		185				2.3	
Air-dried median		1.5		185				4.9	
Air-dried range		0.7-2.3						0.3-4.4	

MAD: mesophilic anaerobic digestion; AS: activated sludge; LMAD: liquid mesophilic anaerobic digestion; AeD: aerobic digestion.

are adequately supplied according to crop requirements. The  $N_e$  is an estimate of the PAN and can be used to determine the proportion of mineralizable N, based on the mineral and organic N contents in the applied biosolids and assuming the entire mineral N fraction contributes to the PAN, as follows:

to include a factor for losses of N through  $NH_3$  volatilization (e.g. 50%);  $N_{org}$ , biosolids organic N (% TN).

Mineralizable N calculated using this method represents the net balance of the N taken up by the crop and that which is lost from the soil profile by leaching, runoff, erosion and gaseous emissions.

#### $N_{m}(\%) = (N_{e} - N_{min}) / N_{org} * 100$ (3)

where:  $N_m$ , mineralizable N (% organic N);  $N_e$ , nitrogen equivalency (equivalent to PAN) (% TN);  $N_{min}$ , the proportion of inorganic N ( $NH_4-$ N +  $NO_3-N$ ) (% TN) originally present in biosolids (this can be adjusted 4.5. Rapid chemical extraction procedures

Incubation procedures, and particularly glasshouse and field studies, are relatively time- and resource-consuming for the routine assessment of mineralizable N in biosolids. Therefore, chemical test methods have been proposed as an alternative approach to provide rapid, indicative

#### Table 8

Total nitrogen (TN) and mineral N (ammonium–N,  $NH_4$ –N and nitrate–N,  $NO_3$ –N) values reported in the scientific literature for composted (Com) biosolids. Values reported on a dry solid (DS) basis, apart from mineral N (% TN).

Further description	n	TN %		NH <sub>4</sub> -N mg	kg <sup>-1</sup>	NO3-N mg l	$kg^{-1}$	Mineral N	Reference
		Mean	Range	Mean	Range	Mean	Range	%TN	
	3	1.1	0.73-1.15						Tester et al. (1977)
Raw	1	1.4		502		<1		3.6	Epstein et al. (1978)
MAD	1	1.0		532		13		5.5	Epstein et al. (1978)
Raw, LT, oven/air-dried	1	1.6		970		20		6.1	Parker and Sommers (1983)
MAD	2	1.2	1.0-1.3	505	170-840	1115	90-2140	4.2	Parker and Sommers (1983)
	3	1.4	1.1-1.8	1547		18.5	9-26	11.2	Sims (1990)
AS, air-dried	1	4.9		7900		0.2		16.1	Gilmour et al. (2003)
Raw	3	1.5	1.47-1.54	225	14.5-532	512	173-1182	4.9	Morris et al. (2003)
	1	1.4		400		1800		15.7	Adegbidi and Briggs (2003)
Lagoon dewatered	2	2.5	2.3-2.6	3000	2000-4000			12.0	Cogger et al. (2004)
	1	1.6		277				1.7	Corrêa (2004)
	1	3.4		656				1.9	Ojeda et al. (2006)
	1	3.4		4450		1.88		13.1	Tarrasón et al. (2008)
	3	1.6		180		97		1.7	Corrêa et al. (2006)
	6	6.0	6.6-6.9						Esteller et al. (2009)
	1	1.2		36		312		3	Boen and Haraldsen (2011)
Mean		2.2		1513		344		7.2	
Median		1.6		518		58		5.2	
Range		1.0-6.0		36-7900		<1-1800		1.7–16.1	

MAD: mesophilic anaerobic digestion; LT: lime treated; AS: activated sludge.

measurements of the N availability in biosolids-amended soils. For example, Douglas and Magdoff (1991) found that the N released by a Walkley-Black acid-dichromate digest was significantly correlated to the fraction of organic N mineralized during soil incubations with 19 different organic residues. Magdoff and Chromec (1977) extracted the mineral N fraction in biosolids-amended soil with 0.01 M CaCl<sub>2</sub> following autoclave treatment for 16 h at 121 °C. Mineralizable N in DMAD, LT, and AeD biosolids determined by non-leached soil incubation was positively correlated to the autoclaving procedure. By contrast, Serna and Pomares (1992) observed no significant relationship between autoclave-extraction and the N mineralization of air-dried, ground biosolids from MAD or AeD measured by non-leached laboratory incubation, or the TN uptake by maize (Zea mays L.) in a growth chamber. However, pepsin or HCl were suggested by Serna and Pomares (1992) as alternative extractants for estimating available N release from biosolids as both showed good correlations with biological indices of N availability. Similarly, Parnaudeau et al. (2004) also found a significant relationship between acid soluble N and biosolids N mineralization.

Other chemical extraction procedures show promise for estimating soil mineralizable N pools and could also be applicable to characterizing biosolids N mineralization behaviour. For example, Curtin and Wen (1999) investigated soil organic matter pools contributing to mineralizable N using a series of chemical and physical operational extraction procedures. The soil N mineralization rate coefficient (k) correlated with the N pool extracted by KCl digestion (2 M at 100 °C for 4 h: hot KCl method), suggesting that hot KCl N may represent the most labile organic N fraction. Steam distillation in phosphate-borate (PB) buffer at pH 11.2 extracted approximately 3 times more N as hot KCl, and was also considered to be representative of the most labile forms of mineralizable N. A light fraction (LF) of soil organic N (extracted by NaI solution and elutriated with 0.01 M CaCl<sub>2</sub> solution) was the largest N type measured overall and was positively correlated with the total mineralizable N pool. However, it was not closely related to k, suggesting that it may represent more slowly mineralized N. The sum of both PB N and LF N was approximately equivalent to the total mineralizable N pool.

The development of a rapid extraction procedure that provides robust and reliable estimates of mineralizable N in different biosolids types applied to soil would offer significant advantages, compared to current soil incubation and other methods, and be potentially feasible for application as a method for routine characterization of biosolids N. This would enable a more comprehensive and accurate assessment of the overall fertilizer value of biosolids N than is currently possible with standard total and soluble nutrient chemical analysis, and could lead to refined fertilizer recommendation practices being implemented for specific biosolids sources.

## 5. Factors influencing nitrogen transformations in biosolids-amended soil

#### 5.1. Biosolids type

The literature survey indicated that the mineralizable N in biosolids, and the subsequent release of this available N pool in amended soil, were significantly influenced by the type of sewage sludge treatment process, dewatering and storage. A summary of the mineralizable N contents of different biosolids types and the experimental conditions reported are presented in Tables 9–15, and are discussed below.

#### 5.1.1. Raw unstabilized sludge

Landspreading of raw unstabilized sludge is not generally permitted in developed countries as a precaution against disease transmission (US EPA, 1993; ADAS, 2001). Nevertheless, it is allowed by the EU Sludge Directive 86/278/EEC (CEC, 1986) provided that additional land use precautions are taken, and was practised in some European countries, including the UK. Therefore, data were collected on the N fertilizer replacement value of unstabilized sludge, although most European countries no longer apply raw sludge to farmland and it was phased out in the UK by the Safe Sludge Matrix (ADAS, 2001). Unstabilized sludge, including primary sludge and secondary, waste activated sludge (WAS), had a high proportion of mineralizable N, compared to treated biosolids types (Table 9). The mean mineralizable N value reported in the literature for these unstabilized sludge types (excluding air-dried or lagoon stored biosolids) was 42.0% of the organic N content, with a range of 19.8-64.1%. Air-drying or storage of raw sludge in a lagoon facilitates microbiological stabilization over time and reduced the mineralizable N content (14.7%, range 0-29.7%); a clear relationship also exists between mineralizable N and the method of dewatering or storage and this is discussed further in Section 5.1.6. Most of the N in dewatered unstabilized sludge is in organic forms (96.8% TN, Table 2), much of which is labile organic matter since it has not been transformed or stabilized by microbial digestion or decomposition processes. Consequently, the increase in microbial activity with addition of unstabilized, raw sludge to soil as organic matter is degraded (Rigby et al., 2009; Sierra et al., 2001), coupled with the larger C:N ratio of this sludge type (e.g. typical C:N value of raw sludge, which is usually a mixture of primary sludge and WAS in the ratio 60:40 on a DS basis: 13:1; Smith and Tibbett, 2004) compared to treated, stabilized forms (e.g. MAD biosolids typical C:N value range: 7-9:1; Smith and Tibbett, 2004), may lead to a short-term immobilization of N causing an initial lag in net mineralized N (Smith et al., 1998a,b,c). The addition of unstabilized organic matter to the soil may also produce undesirable gaseous losses of N via denitrification under certain circumstances as discussed further in Sections 7.2 and 8.

#### 5.1.2. Digested biosolids

Microbiological digestion treatment processes decompose labile organic N in sludge releasing NH<sub>3</sub>–N (Smith et al., 1998a), and may reduce the overall pool of mineralizable N compared to unstabilized sludge. Thus, MAD biosolids (excluding air-dried types) had a mean mineralizable N content equivalent to 29.8% of organic N (range 10.5-45.4%) (Table 11). However, biosolids from AeD treatment processes typically had larger mineralizable N fractions than anaerobically digested types (Garau et al., 1986; Parker and Sommers, 1983). For example, the mean mineralizable N content in AeD biosolids was 28.5% compared to 15.3% in biosolids treated by MAD after incubation in soil for 112 days (Parker and Sommers, 1983). The overall mean mineralizable N content of AeD biosolids (excluding air-dried types) reported in the literature was 47.2% of organic N (range 32.1–57.5%) (Table 10). Thus, in contrast to AD, biosolids treated by AeD had a mineralizable N pool broadly similar to raw, unstabilized sludge, indicating that the organic matter content was generally less effectively stabilized compared to MAD. Thermophilic aerobic-anaerobically digested biosolids also had marginally larger mineralizable N contents, equivalent to 34.0% (range 32.5-36.5%) (Table 12), compared to biosolids treated by MAD processes. Higher mineralizable N contents in aerobically digested biosolids types could be explained by the growth of new microbial biomass during aerobic treatment, which accounts for the relatively poor stability and significant N value of this sludge type (e.g. Sierra et al., 2001; Pu et al., 2012) compared to MAD, for instance, where metabolic constraints in the anaerobic environment limit microbial growth (Lester and Birkett, 2002).

Pre-treatment methods are applied to destroy pathogens and/or increase secondary sludge digestibility and may therefore potentially influence the mineralizable N content of treated biosolids. However, Morris et al. (2003) found that the mineralizable N fraction in dewatered thermally hydrolysed MAD biosolids was similar to products from conventional MAD treatment, and was equivalent to 16.2% and 17.2% of their organic N contents, respectively. Therefore, it is possible for effective, conventional MAD treatment processes to achieve comparable organic N stabilities to biosolids receiving more extensive pretreatments involving biomass disruption and hydrolysis reactions.

Mineralizable nitrogen (N<sub>m</sub>) content (% organic N) and experimental conditions reported in the scientific literature for raw sludge, arranged according to dewatering method (mechanically dewatered; air-dried/lagoon dewatered).

Description	Mean N <sub>m</sub> (% organic N) (range)	Rate (t ha <sup>-1</sup> )	Soil pH	Soil type(s)/texture(s)	Temp (°C)	Moisture <sup>a</sup>	Duration (days)	Method	Additional notes	Reference
Raw, mechanically dewatered	19.8 (18.2–21.3)	10	6.8/8.2	Sandy silt loam (Luvisol), silty clay (Rendzima)	10–31	15–29% DS	90	Field, no crop	Mean N <sub>m</sub> for two soil types	Rigby et al. (2009)
AS, alum dosed, belt press dewatered Mean Median Range	64.1 42.0 42.0 19.8–64.1	0-15	5	Sand			123	Field, RR, ryegrass	Mediterranean- type climate	Rigby et al. (2010)
AS, lagoon dewatered	0.0	6.7		Sandy loam (Puyallup)	25	40% MWHC	62	NL inc.		Gilmour et al. (2003)
AS, lagoon dewatered	4.5 (3.6–5.4)			Sandy loam (Puyallup)				Field, RR, fescue <sup>b</sup>	Washington, 1 growing season	Gilmour et al. (2003)
AS, air dried	19.0	6.7		Sandy loam (Puyallup)	25	40% MWHC	62	NL inc.		Gilmour et al. (2003)
Oxidation ditch, air dried	29.7			sandy loam (Puyallup)				Field, RR, fescue <sup>c</sup>	Washington, 1 growing season	Gilmour et al. (2003)
Mean	14.7									
Median	14.5									
Range	0.0-29.7									

Field: field experiment; RR: relative response; NL inc.: non-leached laboratory incubation; L inc.: leached laboratory incubation.

AS: activated sludge. MWHC: Maximum water holding capacity.

<sup>a</sup> Units as reported.

<sup>b</sup> Mineralizable N was calculated from nitrogen equivalency value and mineral N/organic content of biosolids, assuming 50% volatilization of ammonia for surface applied biosolids and no volatilization for soil-incorporated biosolids, according to guidelines given in US EPA (1995).

Across the international literature, major differences are reported in the mineralizable N contents of biosolids from AeD and MAD processes in different regional areas. For example, in the UK, a mineralizable N content of 12.5-17.3% is consistently found for MAD biosolids in crop and laboratory investigations (Morris et al., 2003; Rigby et al., 2009; Smith et al., 1998a). Elsewhere, however, for example in the USA and Australia, larger mineralizable N values have been observed for MAD biosolids, typically in the range: 28.5-45.4% (e.g. Al-Dhumri et al., 2013; Gilmour et al., 2003; Pu et al., 2008). Plant available N results were also correspondingly increased in these regions; for example, an average PAN value for DMAD of 38% of the TN was reported in the USA (Gilmour et al., 2003) and 40-56% in Australia (Al-Dhumri et al., 2013; Eldridge et al., 2008; Pu et al., 2008), compared to approximately 30% of the TN in the UK (Morris et al., 2003) (Table 16). This behaviour suggested that the apparent increase in PAN may be explained because a greater proportion of the organic N supplied by the biosolids was available for use by crops, rather than it being accounted for by relative differences in the balance of mineral to TN in the biosolids. Research in Egypt has also indicated a greater PAN value than in the UK, of 50% for stockpiled mesophilic anaerobically digested biosolids (WRc, 1999) (Table 16). These observations in some cases may be explained due to soil and environmental factors, which are discussed further in Sections 5.4–5.7. However, although these factors have a significant effect on the rate of mineralization and release of biosolids N in soil, overall, the potentially mineralizable N fraction remains relatively consistent irrespective of varying soil type conditions in temperate regions (e.g. Rigby et al., 2009). The very close similarity in biosolids mineralizable N pools measured by laboratory incubation at optimum temperatures (e.g.  $\geq$  25 °C) and in field investigations within temperate regions (e.g. Gilmour et al., 2003) also suggests that temperature generally does not influence the overall amount of mineralizable N released from biosolids in temperate soils, but that it only affects the rate of N release. However, whilst soil temperature is commonly assumed to control the rate, but not the extent, of N mineralization (Benbi and Richter, 2002), there is evidence suggesting that temperature can influence the fundamental adaptation and capacity of soil microbial communities to mineralize more recalcitrant, stable forms of organic N (MacDonald et al.,

1995; Zogg et al., 1997). Thus more biosolids organic N may be potentially mineralizable in amended soil under warmer climatic conditions (Section 5.5). However, this explanation may only partially account for the apparent variability in mineralizable N observed between similar biosolids types in the international literature. Indeed, differences in N mineralization of equivalent digested biosolids types may also be potentially explained by varying upstream factors and processes during wastewater treatment, such as the composition of the sewage sludge, particularly the primary sludge to WAS ratio, as well as the operation of local sludge treatment processes, which affect the overall degree of organic matter stabilization achieved; this is explored further in Section 8.

#### 5.1.3. Lime-treated biosolids (alkaline stabilized)

The mean mineralizable N content of LT biosolids was 34.4% (range 2.6–65.1%) (Table 14), which is greater than biosolids from MAD (Table 11), but smaller on average compared to AeD (Table 10) and unstabilized sludge (Table 9) (excluding air-dried or lagoon stored AeD or raw types). The LT biosolids category included unstabilized (raw) sludge treated with lime and biosolids amended with lime as a supplemental treatment following AD or AeD. As may be expected, therefore, LT raw and AeD sludges had lower stabilities and, consequently, larger mineralizable N fractions compared to LT MAD biosolids. For example, lves et al. (2010) found that 62% of organic N from LTAeD biosolids was mineralized after 56 days of soil incubation, whereas values of 8–39% mineralizable N were reported for LTMAD biosolids (Parker and Sommers, 1983; Gilmour et al., 2003).

#### 5.1.4. Thermally dried biosolids

Thermally dried biosolids have similar mineralizable N contents to unstabilized, AeD and LT biosolids, with a mean value of 40.1% (range 26.0–71.0%) (Table 13). The largest overall mineralizable N value reported for this category of biosolids was 71.0% following a 168 day soil incubation test (Sierra et al., 2001). This may be partly explained because the biosolids were sampled from an AeD process, and therefore potentially had an intrinsically higher mineralizable N content compared to other biosolids types (e.g. MAD) (Section 5.1.2). However,

Mineralizable nitrogen (N<sub>m</sub>) content (% organic N) and experimental conditions reported in the scientific literature for aerobic digestion (AeD) biosolids, arranged according to dewatering method (unspecified; liquid; dewatered – method not specified; air-dried/lagoon dewatered).

Description	Mean N <sub>m</sub> (% organic N) (range)	Rate <sup>a</sup>	Soil pH	Soil type(s)/texture(s)	Temp (°C)	Moisture/rainfall <sup>b</sup>	Duration (days)	Method	Additional notes	Reference
AeD	50.5 (36.1–60.8)	150–900 mg N kg <sup>-1</sup>		Sandy loam (Winooski)	17		91	L inc.	Mean $N_{\rm m}$ for 3 rates	Magdoff and Chromec (1977)
AeD, liquid	54.0	103–127 kg ha <sup>-1</sup>	5.6-6.9	Sandy loam (Hadley)/loam (Nellis)	25	Field moist	119	L inc.	Mean $\mathrm{N}_\mathrm{m}$ for 4 rates and 2 soils	Magdoff and Amadon (1980)
AeD, liquid	55.0 (45 5–68 0)	103-508 kg N ha <sup>-1</sup>	5.6-6.9	Sandy loam (Hadley)/loam (Nellis)				Field	Mean N <sub>m</sub> for 4 rates, 2 soils and 2 crops	Magdoff and Amadon (1980)
AeD, thermophilic, liquid	(15.5° 50.5) 32.1 (19.6–50.4)	400 kg N ha <sup>-1</sup>	4.5/5.4	Sandy (tephric recent)/stoney silt	10–20			L inc.	Mean $N_m$ for 2 soils and 2 temps	Wang et al.
AeD, thermophilic, dewatered	(1010-0011) 32.5 (20-44.9)	2.2–6.6 t ha <sup>-1</sup>		Sandy Ioam (Puyallup)/Ioam (Buckley)			30–50	Field, tall fescue/perennial ryegrass <sup>c</sup>	Temperate climate; mean $N_{\rm m}$ for 3 rates and 2 sites	Cogger et al. (1999)
AeD, partially dewatered AeD, dewatered	52.0 52.1	$8.3 \text{ g kg}^{-1}$ 6-54	4.9 6.8	Clay (Oxisol) Alluvial clay loam (Vertosol)	40 15.7–29.4	30 kPa 417 mm rainfall	168 214	NL inc. Field, N budget, forage	Mean $N_{\rm m}$ for 4 rates	Sierra et al. (2001) Pu et al. (2008)
AeD, dewatered	(43.3–38.9) 57.5 (47–68)	14 t ha <sup>-1</sup>	5.9 (1:5 H <sub>2</sub> O)	Clay (Red Ferrosol)		360–634 mm	217	Field, maize/ unplanted	Subtropical climate	Pu et al. (2012)
AeD, dewatered	34.0	$22 \mathrm{t} \mathrm{ha}^{-1}$	5.2	silty clay loam (Red Chromosol)	11-22	665 mm rainfall	365	Field, turf	Biosolids incorporated	Eldridge et al.
AeD, dewatered	53.0	$22 \mathrm{t} \mathrm{ha}^{-1}$	5.2	Silty clay loam (Red Chromosol)	11–22	665 mm rainfall	365	Field incubation	Soil isolated in PVC tubes	Eldridge et al.
Mean Median Range	47.2 52.1 32.1–57.5									(2000)
AeD, oven-dried/air-dried	25	$30 \text{ t} \text{ha}^{-1}$	5.9	Silt loam (Fincastle)	23	0.3 bar	112	L inc.		Parker and Sommers (1983)
AeD, oven-dried/air-dried	32	$30 \text{ t} \text{ha}^{-1}$	5.9	Silt loam (Fincastle)	23	0.3 bar	112	NL inc.		Parker and Sommers (1983)
AeD, sand-bed dried	69.3 (27–109)	5–20 t ha <sup>–1</sup>	5.5–7.8	Sandy loam (Xerofluvent)/sandy clay loam (Xerofluvent)	30	34kPa	112	L inc.	$N_{\rm m}$ is mean of 4 rates and 2 soils	Garau et al. (1986)
AeD, sand-bed dried	33.9 (19–50)	5–20 t ha <sup>-1</sup>	5.5–7.8	Sandy loam (Xerofluvent)/sandy clay loam (Xerofluvent)	30	34kPa	112	NL inc.	$N_{\rm m}$ is mean of 4 rates, 2 soils	Garau et al. (1986)
AeD, air-dried	6.5 (5.8–7.2) <sup>d</sup>	122–135 mg N kσ <sup>-1</sup>	6.8	Sandy loam	25	40% MWHC	42	NL inc.	$Mean \ N_m \ for \ 2 \ sludges$	Iakimenko et al. (1996)
AeD, air-dried	34.3 (24–40)	28 g kg <sup>-1</sup>	basic	Sandy loam (Xerorthent)	25	66.7% field capacity	112	NL inc.	Mean $\ensuremath{N_{\mathrm{m}}}$ for 6 sludges	Serna and Pomares
AeD, dewatered (stockpiled for 18 months) Air-dried mean Air-dried median Air-dried range	11 (9–12) 33.5 33.0 6.5-69.3	0-25.2 t ha <sup>-1</sup>	5.6-6.3	Clay loam Red Sodosol/sandy loam (Brown Sodosol)	16.6–19.8	237–240 mm rainfall	120	Field; RR	Mean $N_{\rm m}$ for 6 rates and 2 soils	Al-Dhumri et al. (2013)

Field: field experiment; RR: relative response; NL inc.: non-leached laboratory incubation; L inc.: leached laboratory incubation.

MWHC: Maximum water holding capacity.

AeD: aerobic digestion.

<sup>a</sup> Application rate (units as reported).

<sup>b</sup> Moisture content/tension or rainfall (units as reported).

<sup>c</sup> Mineralizable N was calculated from nitrogen equivalency value and mineral N/organic content of biosolids, assuming 50% volatilization of ammonia for surface applied biosolids and no volatilization for soil-incorporated biosolids, according to guidelines given in US EPA (1995).

<sup>d</sup> Expressed as a proportion of total N (mineral N and organic N concentrations not given).

thermal conditioning also consistently increased the mineralizable N contents of treated biosolids materials. Thus, the findings indicated that TD biosolids had smaller inorganic N contents compared to mechanically dewatered sludge types, due to losses of NH<sub>3</sub>–N during thermal drying, but this was balanced by an increase in mineralizable N with heating so that, overall, the PAN value of these materials with widely contrasting physico-chemical properties was remarkably similar (Smith and Durham, 2002; Morris et al., 2003; Rigby et al., 2009). A possible explanation for this behaviour may be due to denaturation of organic N compounds at high temperatures increasing accessibility to mineralization by soil microorganisms.

#### 5.1.5. Composted biosolids

The smallest overall mineralizable N contents were reported for composted biosolids, with a mean value of 6.7% (range -10-24.5%) (Table 15), compared to the other biosolids types examined. Thus, effective sewage sludge treatment by composting processes produces a biosolids which is a strongly stabilized source of residual organic matter of low to very low N content and availability. The variable rates and extent of N mineralization of composted biosolids reported in the literature may be explained by differences in compost maturity, as labile forms of N and C are biodegraded into more stable forms during the composting process and maturation of the material (Bernal et al., 1998; Doublet et al., 2011). Immobilization of N is also possible in biosolids compost-amended soils (Boen and Haraldsen, 2011; Bowden et al., 2007; Sims, 1990; Tester et al., 1982). For example, in a pot trial with tall fescue grown in a silty clay loam soil amended with composted LT sludge, Bowden et al. (2007) estimated a net mineralization rate equivalent to -15 to -5% of organic N supplied, indicating the net immobilization of N. Nitrogen immobilization is often associated with inputs to soil of organic matter with high C:N ratio (for example > 20:1; Parker and Sommers, 1983), and this physico-chemical property of composted biosolids is governed by the bulking agent type and addition regime. The degree of compost stability also plays an important role in defining the net balance of N mineralization-immobilization processes in amended soil. Specifically, the C:N ratio of the labile fraction of organic matter is the critical factor influencing the N mineralization rate. For example, Bernal et al. (1998) observed immobilization of N in the first 10 days of an incubation experiment with soil amended with immature compost containing high labile C, whereas no N lock-up occurred in soil receiving maturated compost.

#### 5.1.6. Air-dried biosolids and storage

The methods employed to dewater biosolids and storage period profoundly impact the extent of sludge N mineralization (Gilmour et al., 2003). For example, the mean TN content of MAD biosolids after airdrying or extended storage was 2.8% DS compared to 5.0% DS for mechanical dewatering processes (Table 4). Total and mineralizable N values of air- or lagoon-dried biosolids decrease due to NH<sub>3</sub> volatilization losses and mineralization of organic N during the long retention periods associated with this passive form of dewatering process. For example, the mean mineralizable N value of air-dried MAD biosolids was 19.9% compared to 29.8% for mechanically dewatered types (Table 11).

Coker et al. (1987b) reported the mineralizable N content of LMAD may decrease from 15.0% to 9.0% following storage for a period of one year. The smallest mineralizable N values were recorded for lagoondried, pulverised and flash-dried MAD biosolids (Premi and Cornfield, 1971) and air-dried MAD biosolids (Chae and Tabatabai, 1986; Rouch et al., 2011; Smith et al., 1998a). Thus, Smith et al. (1998a) found that 7% of the organic N in air-dried MAD biosolids was mineralized after soil incubation for 73 days at 25 °C compared to 12.5% for mechanically dewatered, MAD biosolids.

Mineral N production by MAD biosolids, measured by a 70 day soil incubation procedure, decreased to 2.7% of the organic N fraction after pan-drying for 11 months (Rouch et al., 2011), although further storage in stockpiles for 24–36 months had little additional effect on the

proportion of mineralizable N. However, TN declined during both of these post-digestion treatment phases, from a high initial concentration reported of 15% TN DS in the input LMAD sludge, to a mean of 3.9% after pan-drying, decreasing to 0.8% in the stockpiles. Therefore, in addition to the volatilization of NH<sub>3</sub>–N, it is plausible that these extensive losses of N could also be explained by denitrification emissions of gaseous N during stockpile storage. Rouch et al. (2011) suggested the limited N release from pan-dried digested biosolids was attributed to increased organic matter stability compared to conventional AD with mechanical dewatering, due to the extended storage period; in this specific case it was also possibly linked to a lack of appropriate microbial flora to mineralize the applied organic N in the nutritionally poor incubated tenosol soil type.

There were many cases, particularly in earlier studies (e.g. Magdoff and Chromec, 1977; Parker and Sommers, 1983), where the method or type of sludge dewatering process was not reported. However, it is necessary to consider and include a description of this important operational sludge characteristic when investigating biosolids mineralizable N to ensure the robust and critical interpretation of biosolids N mineralization data.

#### 5.2. Carbon to nitrogen ratio

The relationship between the C:N ratio and N mineralization of biosolids is extensively reported in the literature (Er et al., 2005; Serna and Pomares, 1992; Parnaudeau et al., 2004). For example, Er et al. (2005) statistically evaluated the effects of biosolids type, organic N content, application rate, C:N ratio, soil organic N content, soil pH, time and temperature on N mineralization using a backwards regression selection procedure, and data from 12 published studies on AeD, AD and Com biosolids. The effect of biosolids C:N ratio on N mineralization was highly statistically significant and explained 35.3% of the total variability of mineralizable N data. Biosolids application rate and temperature were also significant explanatory variables accounting for 34.1% and 17.6% of the total variance in N mineralization results, respectively.

Specifically, the C:N ratio of the labile organic matter fraction in biosolids is the critical factor controlling the N mineralization process in amended soil. Parnaudeau et al. (2004) examined the chemical and organic C and N mineralization characteristics of 17 wastewater sludges, including 11 municipal wastewater sludges, both untreated and treated by a variety of methods including AD and lime amendment. The organic C to organic N ratios of the sludges were relatively low, from 5–19, and there was a significant relationship between the organic C:organic N ratio and N mineralization. In particular, the C:N ratio of the acidsoluble (12 *M* HCl) organic fraction was strongly correlated with the mineralizable N contents measured in the different types of sewage sludge examined. Acid-soluble fractions with low C:N ratios were assumed to be mainly composed of proteinic compounds and to contribute the majority of rapidly mineralizable N in biosolids-amended soil.

#### 5.3. Application rate

Nitrogen mineralization and nitrification activity in soil are potentially sensitive to the biosolids application rate (Ryan et al., 1973). For example, Premi and Cornfield (1971) reported that the recovery of inorganic N in an alluvial sandy loam soil after six weeks of incubation with lagoon dried MAD biosolids decreased from 4.2 to 2.3% as the rate of biosolids application increased from 5 to 20 g kg<sup>-1</sup>. This behaviour may be explained by the accumulation of NH<sub>3</sub>–N in soil when high rates of digested biosolids are applied, which is inhibitory to soil microbial activity including mineralization and nitrification processes (Epstein et al., 1978). For example, Ryan et al. (1973) applied high rates of digested sludge to soil supplying 378–756 mg kg<sup>-1</sup> of NH<sub>4</sub>–N and observed an initial lag in nitrification activity. High rates of biosolids application can also reduce the apparent net N mineralization rate by encouraging gaseous losses of N by denitrification (Epstein et al., 1978; Lindemann Mineralizable nitrogen (N<sub>m</sub>) content (% organic N) and experimental conditions reported in the scientific literature for mesophilic anaerobic digestion (MAD) biosolids, arranged according to dewatering method (liquid; dewatered – method not specified; mechanically dewatered; air-dried/lagoon dewatered), and pre-treatment methods.

Description	Mean N <sub>m</sub> (% organic N) (range)	Rate <sup>a</sup>	Soil pH	Soil type(s)/texture(s)	Temp (°C)	Moisture/rainfall <sup>b</sup>	Duration (days)	Method	Additional notes	Reference
LMAD	31 (28–34)	6.7 t ha <sup>-1</sup>		Silt loam (Conover loam)	25	40% MWHC	63–75	NL inc.	$Mean \ N_m \ for \ 2 \ sludges$	Gilmour et al. (2003)
LMAD	10.5 (-31.3-40.9)			Loam (Conover loam)				Field, RR, field corn	Michigan; biosolids incorporated, N <sub>m</sub> for 2 sludges and 2 seasons	Gilmour et al. (2003)
DMAD	12.5 (0–26)	50 t ha <sup>-1</sup>	6.9–8.0	Loamy sand (Aberford association), clay loam (Andover I association)	25	40% MWHC	73	NL inc.	Mean $N_{\rm m}$ for 2 sludges and 2 soils	Smith et al. (1998a)
DMAD	32 (27–37)	10 g kg <sup>-1</sup>	5.5-7.5	Silt loam (Fincastle)/silty clay (Chalmers)	21	— 1 bar	168	NL inc.	Mean $\ensuremath{N_{\mathrm{m}}}$ for two soils	Terry et al. (1981)
DMAD	31 (31.3–35.5)	2.78-3.63 t ha <sup>-1</sup>		Silt loam (Captina)				Field, sorghum sudangrass <sup>c</sup>	Mean $N_m$ for 2 years	Gilmour and Skinner (1999)
DMAD	36.5 (24.1–53.3)			Silt loam (Captina), sandy loam (Puyallup), loam (State)				Field, RR, field corn/ fescue <sup>c</sup>	Arkansas, Virginia, Washington; N <sub>m</sub> for 6 sludges and 2 seasons	Gilmour et al. (2003)
DMAD	35.5 (26.7–41.6)	0–1172 kg N ha <sup>–1</sup>	6.5	Clay (Acid sulphate)	22.1–31.6 (humid /tropical)	irrigated daily	121	Field incubation	Mean N <sub>m</sub> for 4 rates	Sripanomtanakorn and Polprasert (2002)
DMAD	43.5 (30–57)	25.8–46.8 t ha <sup>-1</sup>	7.7	Clay loam	25	Field capacity	252	Pot trial, planted/non-planted	Mean $\mathrm{N}_\mathrm{m}$ for 2 sludges	Adegbidi and Briggs (2003)
DMAD	17.2 (11.9–21.9)	0–13.3 t ha <sup>–1</sup>	5.9	Loamy sand	Temp-erate climate			Field	Mean N <sub>m</sub> for 3 sludges and 3 growing seasons	Morris et al. (2003)
DMAD	15.2 (5.9-29)	400 kg N ha <sup>-1</sup>	4.5 /5.4	Sandy (tephric recent)/stoney silt loam (pallic orthic brown soil)	10–20		182	L inc.	Mean N <sub>m</sub> for 2 soils and 2 temperatures	Wang et al. (2003b)
DMAD	45.4 (42.6–48.1)	16-72 t ha <sup>-1</sup>	6.8	Alluvial clay loam (Vertosol)	15.7–29.4	471 mm rainfall	214	Field, N budget, forage	Mean $N_m$ for 2 rates	Pu et al. (2008)
DMAD	35 (33–37)	14 t ha <sup>-1</sup>	5.9 1:5 H <sub>2</sub> O	Clay (Red Ferrosol)		360 mm (Trial 1)/ 634 mm (Trial 2)	217	Field, maize/unplanted	Subtropical climate; Mean N <sub>m</sub> for 2 trials	Pu et al. (2012)
DMAD	17.3 (14.2–20.4)	10 t ha <sup>-1</sup>	8.2 /6.8	Silty clay (Rendzima)/sandy silt loam (Luvisol)	10–31	15–29%	90	Field, no crop	2 soils, 3 reps	Rigby et al. (2009)
DMAD	38.1	0–15 t ha <sup>–1</sup>	6	Sand			123	Field, RR	Mediterranean-type climate	Rigby et al. (2010)
DMAD	35.0	7.5 t ha <sup>-1</sup>		Brown Sodosol	12.5	70	56	NL inc.		Ives et al. (2010)
DMAD, belt-filter Mean	28.5 (17–40) 29.8	4.5–6.7 t ha <sup>-1</sup>		Silt loam (Captina)/sandy loam (Puyallup)	25	40	75	NL inc.	Mean $N_{\rm m}$ for 2 sludges	Gilmour et al. (2003)

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Table 11 (continued)

Description	Mean N <sub>m</sub> (% organic N) (range)	Rate <sup>a</sup>	Soil pH	Soil type(s)/texture(s)	Temp (°C)	Moisture/rainfall <sup>b</sup>	Duration (days)	Method	Additional notes	Reference
Median	32.0									
Range	10.5-45.4		- 4	A11 · 1 · 1 · 1	20	50	10	NV .		D : 10 011
MAD, lagoon dried, pulverised, flash dried	3.2 (2.3–4.2)	5–20 g kg <sup>–1</sup>	7.1	Alluvial sandy loam	30	50	42	NL INC.	3 rates, 2 reps	(1971)
MAD, sand-bed dried	46.8 (23-68)	8.25–33 t ha <sup>–1</sup>	5.5–7.8	Sandy loam (Xerofluvent)/sandy clay loam (Xerofluvent)	30	34kPa	112	L inc.	Mean $N_m$ for 3 rates	Garau et al. (1986)
MAD, sand-bed dried	22.9 (16-31)	8.25–33 t ha <sup>-1</sup>	5.5–7.8	Sandy loam (Xerofluvent)/sandy clay loam (Xerofluvent)	30	34kPa	112	NL inc.	Mean $\ensuremath{N_{\mathrm{m}}}$ for 3 rates	Garau et al. (1986)
LMAD, lagoon matured 2–5 years	9 (4.7–12.2)	0–415 kg N ha <sup>–1</sup>		Loamy sand (Cottenham), silty loam (Milton), clay loam (Oak), calcareous loam (Aberford)				Field, RR, ryegrass	Temperate climate, surface applied, mean N <sub>m</sub> for 2 rates and 4 sites	Coker et al. (1987b)
MAD, sand-bed dried	28.5 (25–32)	0–11 t ha <sup>–1</sup>	6.9	Loam (Weld)				Field, RR dryland winter wheat	Mean $N_{\rm m}$ for 5 rates and 2 sites	Barbarick and Ippolito (2000)
LMAD, storage lagoon (2–8 years)	13.3 (7.1–19.5)			Sandy loam (Puyallup)				Field, RR, fescue	Washington; surface applied, 1 growing season; N <sub>m</sub> for 2 sludges,	Gilmour et al. (2003)
MAD (air-dried/oven dried)	15.2 (4–31)	$30 \text{ t ha}^{-1}$	5.9	Silt loam (Fincastle)	23	0.3 bar	112	L inc.	Mean N <sub>m</sub> for 11 sludges	Parker and Sommers (1983)
MAD (air-dried/oven dried)	15.4 (7–27)	30 t ha <sup>-1</sup>	5.9	Silt loam (Fincastle)	23	0.3 bar	112	NL inc.	Mean $\mathrm{N}_\mathrm{m}$ for 11 sludges	Parker and Sommers (1983)
MAD, air-dried	7.0 (1–13)	50 t ha <sup>-1</sup>	6.9–8.0	Loamy sand (Aberford association), clay loam (Andover I association)	25	40% MWHC	73	NL inc.	Mean $N_m$ for 2 soils	Smith et al. (1998a)
MAD, air-dried	27.2 (19.3–28.4)	15–30 g kg <sup>-1</sup>		Fine-silt/coarse-loam	35	0.01 Mpa	224	L inc.	Mean $\ensuremath{N_{m}}\xspace$ for 2 rates and 2 soils	Lindemann and Cardenas (1984)
MAD, air-dried	17.8 (1–51)	$50 \text{ t } \text{ha}^{-1}$	5.1-7.0	Silt-loam/silty clay loam	30		182	L inc.	Mean $\ensuremath{N_{\mathrm{m}}}$ for 3 sludges and 5 soils	Chae and Tabatabai (1986)
MAD, drying bed	38 (31–45)	6.7 t ha <sup>-1</sup>		Sandy loam (Puyallup)	25	40	63	NL inc.	Mean $N_{\rm m}$ for 2 sludges	Gilmour et al. (2003)
MAD, air-dried	28.4 (25.0–30.3)			Loam (Conover)				Field, RR, fescue	Michigan; surface applied; mean $N_m$ for 3 sludges and 2 seasons	Gilmour et al. (2003)
MAD, drying pan (11 months)	2.7	$10 \text{ t ha}^{-1}$	5.5	Tenosol	20	11-14%	70	Inc., NL		Rouch et al. (2011)
DMAD (stockpiled 24–36 months)	3.1 (0.8–6.93)	10 t ha <sup>-1</sup>	5.5	Tenosol	20	11-14%	70	Inc., NL	Mean $N_m$ for 5 sludges	Rouch et al. (2011)
DMAD (stockpiled 1–3 months) Mean Median	40 (39-41) 19.9 16.8	0-22.7 t ha <sup>-1</sup>	5.6–6.3	Clay loam (Red Sodosol)/sandy loam (Brown Sodosol)	16.6–19.8	237–240 mm rainfall	120	Field, ryegrass	Mean $N_{\rm m}$ for 6 rates and 2 soils	Al-Dhumri et al. (2013)
Range	2.7-46.8									
Pre-pasteurized, DMAD	27.8 (25.3–30.2)	0–13.3 t ha <sup>–1</sup>	5.9	Sandy loam				Field	Temperate climate; mean N <sub>m</sub> for 2 seasons	Morris et al. (2003)
Thermal hydrolysis, DMAD	16.2	0–13.3 t ha <sup>-1</sup>	5.9	Sandy loam				Field	Temperate climate; mean Nm for 2 seasons	Morris et al. (2003)

Field: field experiment; RR: relative response; NL inc.: non-leached laboratory incubation; L inc.: leached laboratory incubation.

MWHC: Maximum water holding capacity.

MAD: mesophilic anaerobic digestion; LMAD: liquid mesophilic anaerobic digestion; DMAD: dewatered mesophilic anaerobic digestion.

<sup>a</sup> Application rate (units as reported).

<sup>b</sup> Moisture content/tension or rainfall (units as reported).

<sup>c</sup> Mineralizable N was calculated from nitrogen equivalency value and mineral N/organic content of biosolids, assuming 50% volatilization of ammonia for surface applied biosolids and no volatilization for soil-incorporated biosolids, according to guidelines given in US EPA (1995).

Mineralizable nitrogen (N<sub>m</sub>) content (% organic N) and experimental conditions reported in the scientific literature for thermophilic aerobic-anaerobic digestion (TA-AD) biosolids, dewatered by filter belt press.

	Mean N <sub>m</sub> (% organic N) (range)	Rate (t ha <sup>-1</sup> )	Soil type(s)/texture(s)	Temp (°C)	Moisture (%MWHC)	Duration (days)	Method	Additional notes	Reference
	32.5 (20–44.9)		Sandy loam (Puyallup)/loam (Buckley)			30–50	Field, tall fescue/perennial ryegrass <sup>a</sup>	Temperate climate; mean $N_m$ for 3 rates at 2 sites	Cogger et al. (1999)
	33	6.72	Sandy loam (Puyallup)	25	40	63	NL inc.		Gilmour et al. (2003)
	36.5		Sandy loam (Puyallup)				Field, fescue	Washington; 1 growing season	Gilmour et al. (2003)
Mean Median Range	34.0 33.0 32.5–36.5								

Field: field experiment; NL inc.: non-leached laboratory incubation.

MWHC: Maximum water holding capacity.

<sup>a</sup> Mineralizable N was calculated from nitrogen equivalency value and mineral N/organic content of biosolids, assuming 50% volatilization of ammonia for surface applied biosolids and no volatilization for soil-incorporated biosolids, according to guidelines given in US EPA (1995).

and Cardenas, 1984; Pu et al., 2012), which is discussed further in Section 7.2.

#### 5.4. Soil texture and organic matter content

Soil physico-chemical and biological properties have important effects on the biosolids N mineralization rate including the soil textural class, particularly the balance between clay and larger soil particle fractions, as well as the microbial activity and processing rate (fertility) of soil. For example, Tester et al. (1977) found the net mineralization

rate of composted biosolids varied between 0–6% organic N in several contrasting soil types with the largest overall mineral N production occurring in a loamy sand (Evesboro C horizon) compared to silty clay (Christiana C horizon) and silty loam (Fauquier B horizon) soil types. The lower mineral N release in the silty loam was attributed to  $NH_4^+$  fixation by vermiculite clay fractions, but in the silty clay loam microbial immobilization of N was suggested as the mechanism responsible for lower  $NO_3^-$  recovery, since the kaolinitic clay present in this soil type is not normally associated with  $NH_4^+$  fixation. Indeed, several other studies (Hall, 1983; Hernández et al., 2002) report increased rates of

#### Table 13

Mineralizable nitrogen (N<sub>m</sub>) content (% organic N) and experimental conditions reported in the scientific literature for thermally dried (TD) biosolids.

De	scription	Mean N <sub>m</sub> (% organic N) (range)	Rate (t ha <sup>-1</sup> )	Soil pH	Soil type(s)/texture(s)	Temp (°C)	Moisture/rainfall <sup>a</sup>	Duration (days)	Method	Additional notes	Reference
Rav	w, TD	29.4 (11.1–41.9)	0-13.3	5.9 (H <sub>2</sub> O)	Loamy sand				Field, RR, ryegrass	Temperate climate; mean N <sub>m</sub> for 3 seasons	Morris et al. (2003)
TD	MAD	32.5 (20–44.9)	2.2-6.6	( 2 )	Sandy loam (Puyallup)/loam (Buckley)			30–50	Field, tall ryegrass/forage grass <sup>b</sup>	Temperate climate; mean N <sub>m</sub> for 2 sites and 2 sludges	Cogger et al. (1999)
TD		44.0	4.48		Silt loam	23–24	175 mm	30–60	Field, sorghum sudangrass <sup>b</sup>		Gilmour and Skinner (1999)
Ael d	D, heat Iried at 60 °C	71.0	10	4.9	Clay (Oxisol)	40	30 kPa	168	NL inc.		Sierra et al. (2001)
TD	MAD	35.6 (31.9–39.1)	0-13.3	5.9 (H <sub>2</sub> O)	Sandy loam				Field, RR, ryegrass	Temperate climate; mean N <sub>m</sub> for 3 seasons	Morris et al. (2003)
TD	MAD	26.0 (22–30)	5.2-6.7		Silt loam (Captina)/silt loam-loam (Celina-Conover)	25	40% MWHC	63	NL inc.	Mean $N_{\rm m}$ for 2 soils	Gilmour et al. (2003)
TD	MAD	30.5 (17.5–43.9)			Sandy loam (Puyallup)				Field, RR, fescue <sup>b</sup>	Washington; mean N <sub>m</sub> for 2 sludges and 2 seasons	Gilmour et al. (2003)
TD.	AeD	30.5 (21–40)	0-48.3		Silty clay loam (Red Chromosol)	11–22	665 mm rainfall	365	Field, turfgrass	Mean $\ensuremath{N_m}$ for 3 rates	Eldridge et al. (2008)
TD.	AeD	55.5 (51–60)	0-48.3		Silty clay loam (Red Chromosol)	35	Field capacity	216	L inc.	Mean $N_{\rm m}$ for 3 rates	Eldridge et al. (2008)
TD	AeD	49 (45–54)	0-48.3	5.2	silty clay loam (Red Chromosol)	11–22	665 mm rainfall	365	Field incubation	Mean $N_{\rm m}$ for 3 rates	Eldridge et al. (2008)
TD	MAD	35.6 (31.5–39.6)	10	8.2/6.8 (H <sub>2</sub> O)	Silty clay (Rendzima)/sandy silt loam (Luvisol)	10–31	15–29%	90	Field, no crop	Mean $N_m$ for 2 soils	Rigby et al. (2009)
ľ	Mean	40.1			. ,						
ľ	Median	35.6									
F	Range	26.0-71.0									

Field: field experiment; RR: relative response; NL inc.: non-leached laboratory incubation; L inc.: leached laboratory incubation.

MWHC: Maximum water holding capacity.

TD: thermally dried; TDMAD: thermally dried mesophilic anaerobic digestion; AeD: aerobic digestion; TDAeD: thermally dried aerobic digestion.

<sup>a</sup> Moisture content/tension or rainfall (units as reported).

<sup>b</sup> Mineralizable N was calculated from nitrogen equivalency value and mineral N/organic content of biosolids, assuming 50% volatilization of ammonia for surface applied biosolids and no volatilization for soil-incorporated biosolids, according to guidelines given in US EPA (1995).

Mineralizable nitrogen (N<sub>m</sub>) content (% organic N) and experimental conditions reported in the scientific literature for lime treated (LT) biosolids, arranged according to dewatering method (unspecified; liquid; dewatered – method not specified; mechanically dewatered; air-dried/lagoon dewatered).

Description	Mean N <sub>m</sub> (% organic N) (range)	Rate <sup>a</sup>	Soil pH	Soil type(s)/texture(s)	Temp (°C)	Moisture/rainfall <sup>b</sup>	Duration (days)	Method	Additional notes	Reference
Raw IT	22	$30  t  ha^{-1}$	59	Silt loam (Fincastle)	23	0.3 bar	112	Linc		Parker and Sommers (1983)
Raw IT	22	$30 \text{ t ha}^{-1}$	59	Silt Ioam (Fincastle)	23	0.3 bar	112	NI inc		Parker and Sommers (1983)
MAD IT	26	$30 \text{ t ha}^{-1}$	59	Silt Ioam (Fincastle)	23	0.3 bar	112	Linc	Mean N., for 2 sludges	Parker and Sommers (1983)
1411 12, 21	(0.1-5)	50 1 114	5.5	She fount (Fineastic)	25	0.5 bui	112	E me.	Mean Mm for 2 studges	Funcer and Sommers (1909)
MAD, LT	12.5	30 t ha <sup>-1</sup>	5.9	Silt loam (Fincastle)	23	0.3 bar	112	NL inc.	Mean N <sub>m</sub> for 2 sludges	Parker and Sommers (1983)
,	(2-23)								in the second	
Raw, LT, liquid	58.4			Silt loam-loam (Celina-Conover)				Field, RR, fescue <sup>c</sup>	Michigan; mean N <sub>m</sub> for 2 sludges	Gilmour et al. (2003)
· · · •	(43.8-70.1)									
Raw, LT, dewatered	51.1			Cobbly loam (Shottower)				Field, RR, fescue <sup>c</sup>	Virginia	Gilmour et al. (2003)
DMAD, LT, ferric	20.8	0-900	6.8	Sandy loam (Winooski)	17		91	L inc.	Mean Nm for 3 rates	Magdoff and Chromec (1977)
chloride	(13.7-25.2)	mg N kg <sup>-1</sup>								-
DMAD, LT,	36.3			Silt loam (Captina)/sandy loam	25	40% MWHC	63	NL inc.	Mean N <sub>m</sub> for 3 sludges	Gilmour et al. (2003)
	(22-59)			(State)						
DMAD, LT,	37.5			Silt loam (Captina)/sandy loam				Field, RR sorghum	Arkansas, Virginia, Washington;	Gilmour et al. (2003)
	(33.7-44.9)			(State)/sandy loam (Puyallup)				sudan grass/fescue	mean N <sub>m</sub> for 3 sludges, 2 seasons	
Raw, LT cake	34.1	0-13.3	5.9	Sandy loam				Field trial, RR,	Temperate climate; mean N <sub>m</sub> for 3	Morris et al. (2003)
	(23.2-41.8)	t ha <sup>-1</sup>	$(H_2O)$	5				ryegrass	seasons	
LT, AeD, dewatered	62.0	7.5 t ha <sup>-1</sup>		Brown Sodosol	12.5	70% MWHC	56	NL inc.		Ives et al. (2010)
Raw, LT, cake	16.4	$10 \text{ t ha}^{-1}$	8.2/	Silty clay (Rendzima)/sandy silt	10-31	15-29%	90	Field, no crop	Mean Nm for 2 soils	Rigby et al. (2009)
	(9.1-23.7)		6.8	loam (Luvisol)						
Raw, LT, cake	65.1	0–15 t	5	Sand				Field, RR, ryegrass	Mediterranean-type climate	Rigby et al. (2010)
		ha <sup>-1</sup>								
DMAD, LT,	38.3	3.94-10.24		Silt loam (Captina)	22.6-26.7	175 mm (year	60-90	Field, sorghum	Mean N <sub>m</sub> for 2 sludges, 2 seasons	Gilmour and Skinner (1999)
belt-pressed	(30.6–49.7)	t ha <sup>-1</sup>				1)-331 mm (year 2)		sudangrass <sup>c</sup>		
Mean	34.4					-,				
Median	33.5									
Bango	2.2.0									

Field: field experiment; RR: relative response; NL inc.: non-leached laboratory incubation; L inc.: leached laboratory incubation.

MWHC: Maximum water holding capacity.

AeD: aerobic digestion; MAD: mesophilic anaerobic digestion; DMAD: dewatered mesophilic anaerobic digestion; LT: lime treated.

<sup>a</sup> Application rate (units as reported).

<sup>b</sup> Moisture content/tension or rainfall (units as reported).

<sup>c</sup> Mineralizable N was calculated from nitrogen equivalency value and mineral N/organic content of biosolids, assuming 50% volatilization of ammonia for surface applied biosolids and no volatilization for soil-incorporated biosolids, according to guidelines given in US EPA (1995).

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 Table 15

 Mineralizable nitrogen (N<sub>m</sub>) content (% organic N) and experimental conditions reported in the scientific literature for composted (Com) biosolids.

Description	Mean N <sub>m</sub> (% organic N) (range)	Rate <sup>a</sup>	Soil pH	Soil type(s)/texture(s)	Temp (°C)	Moisture <sup>b</sup>	Duration (days)	Method	Additional notes	Reference
Raw, Com	2 (0-6)	0-60 g kg <sup>-1</sup>	3.8-5.7	Loamy sand (Evesboro C horizon), silt loam (Fauquier B horizon), silty clay (Christiana C horizon, laboratory sand)	22	0.33 bar	54	NL inc.	Mean $N_{\rm m}$ for 3 rates and 4 soils	Tester et al. (1977)
Com with cotton waste	4.6 (0-9)	48 t ha <sup>-1</sup>	7.8 (H <sub>2</sub> O)	Silt loam (Typical Calciorthid/Xeric Calciorthid)	28	60% MWHC	70	NL inc.	Mean N <sub>m</sub> for 2 soils and 3 composts	Bernal et al. (1998)
Raw, LT, Com	5	30 t ha <sup>-1</sup>	5.9	Silt loam (Fincastle)	23	0.3 bar	112	L inc.		Parker and Sommers (1983)
Raw, LT, Com	12	30 t ha <sup>-1</sup>	5.9	Silt loam (Fincastle)	23	0.3 bar	112	NL inc.		Parker and Sommers (1983)
MAD, Com	3	30 t ha <sup>-1</sup>	5.9	Silt loam (Fincastle)	23	0.3 bar	112	L inc.	Mean N <sub>m</sub> for 2 sludges	Parker and Sommers (1983)
MAD, Com	6.5 (4-9)	$30 t ha^{-1}$	5.9	Silt loam (Fincastle)	23	0.3 bar	112	NL inc.	Mean $N_{\rm m}$ for 2 sludges,	Parker and Sommers (1983)
Com	12	51.8 t ha <sup>-1</sup>	7.7	Clay loam	25	Field capacity	252	Pot trial, planted/non-planted	$Mean \ N_m \ for \ 2 \ sludges$	Adegbidi and Briggs (2003)
Com with garden waste	16.7 (0.12–29)	0–13.3 t ha <sup>–1</sup>	5.9	Sandy loam				Field, RR, ryegrass	Temperate climate; mean N <sub>m</sub> for 3 seasons	Morris et al. (2003)
MAD, Com	24.5 (10.9–38.1)	0-8 t ha <sup>-1</sup>	4.9-5 (H <sub>2</sub> O)	Sand (Orthod Spodosol)/silty clay loam (Ustox Oxisol)	25	Field capacity	161	NL inc.	$Mean \ N_m \ for \ 2 \ soils$	Corrêa et al. (2006)
Raw, LT, Com	-10 (-15-5)	7.82-25.32 g kg <sup>-1</sup>	5.6	Silty clay loam (Fauquier)	20–30	90% field capacity	168	Pot trial, tall fescue	$Mean \ N_m \ for \ 2 \ soils$	Bowden et al. (2007)
Com with park and garden waste	-2.9 (-4-1.3)	4–12 kg m <sup>-2</sup>	6.2		10.8-18.6	— 10–100 kPa		Pot trial, ryegrass <sup>c</sup>	One growing season, May-September	Boen and Haraldsen (2011)
Mean Median Range	6.7 5 									

Field: field experiment; RR: relative response; NL inc.: non-leached laboratory incubation; L inc.: leached laboratory incubation. MWHC: Maximum water holding capacity.

MAD: mesophilic anaerobic digestion; LT: lime treated; Com: composted.

<sup>a</sup> Application rate (units as reported).

<sup>b</sup> Moisture content/tension (units as reported).

<sup>c</sup> Mineralizable N was calculated from nitrogen equivalency value and mineral N/organic content of biosolids, assuming 50% volatilization of ammonia for surface applied biosolids and no volatilization for soil-incorporated biosolids, according to guidelines given in US EPA (1995).

Selected plant available N (PAN) (% total N) values reported in the literature across different regions.

Biosolids type	Plant available N (PAN) (% total N) <sup>a</sup>											
	UK (South East England)	USA (Michigan, Arkansas, Virginia, Washington)	Australia (NSW)	Australia (Queensland)	Australia (Victoria)	Egypt						
Raw, air-dried		17.0										
Raw, solar dried						20						
AeD, dewatered			40	55								
AeD, air-dried					17.5							
DMAD	30.5	38.1		56	41							
DMAD, stockpiled						50						
TD	34.4	32.7	32.5 <sup>b</sup>									
LT, dewatered	37.0	41.1										
Com	21.0											
Reference	Morris et al. (2003)	Gilmour et al. (2003)	Eldridge et al. (2008)	Pu et al. (2008)	Al-Dhumri et al. (2013)	WRc (1999)						

AeD: aerobic digestion; DMAD: dewatered mesophilic anaerobic digestion; TD: thermally dried; LT: lime treated; Com: composted.

<sup>a</sup> Plant available N (PAN): mineralizable N + mineral N.

<sup>b</sup> Mean for surface applied and incorporated treatments.

mineralization of biosolids N with sandy soil textures compared to soils with higher clay contents and attribute this to better aeration in sandy soils. However, the opposite behaviour has also been observed where mineralization and nitrification rates increase in soils with high clay contents, which typically also have larger organic matter contents that promote greater microbial activity (Chae and Tabatabai, 1986; Corrêa et al., 2006; Jha et al., 1996; Rigby et al., 2009). Rigby et al. (2009) investigated the influence of two contrasting soils, a silty clay (Rendzima overlying chalk) and a sandy silt loam (Luvisol overlying brickearth), on N availability in biosolids-amended soil (DMAD, TDMAD and dewatered raw sludge), and found that soil type influenced the rate of N mineralization, but not the overall extent of net N release. Thus, the mineral N accumulation rate was reduced in the sandy soil type due to the slower microbial turnover of organic N compared to the silt loam, however, the final, overall net mineral N production was similar for each applied biosolids product irrespective of the contrasting soil type conditions.

Additionally, soil properties can affect the availability of biosolidsderived N in soils due to losses of N through leaching, runoff, erosion or gaseous emissions. Many of these soil type effects on N availability are common to all sources of fertilizer N (both conventional mineral forms and organic sources); however, the interaction between soil and biosolids properties can also have a significant influence upon these processes (Section 7).

#### 5.5. Soil temperature

Temperature has already been established (Section 5.1.2) as a key factor influencing the rate of organic N decomposition and transformations in soil treated with biosolids and other organic N fertilizer sources (Gilmour and Gilmour, 1980; Honeycutt et al., 1991; Smith et al., 1998a, 1998b, 1998c; Sierra et al., 2001; Terry et al., 1981). Sierra et al. (2001) found that mineralization and nitrification rates were raised with increasing soil temperature between 20–40 °C; however, the magnitude of the response was greater between 20–30 °C than 30–40 °C. Nitrogen mineralization rates increased between temperatures of 15-30 °C in a laboratory incubation study with two soil types (a Fincastle silt loam (fine silty, mixed, mesic Aeric Ochraqualf) and a Chalmers silty clay loam (fine-loamy, mixed, mesic Typic Argiaquoll)) amended with AD biosolids, and at the end of the incubation period of 168 days, the N mineralized at 30 °C was three times larger compared to the 15 °C temperature condition (Terry et al., 1981). Similarly, Wang et al. (2003b) found the mineralization rate of organic N in AeD and AD biosolids in a laboratory incubation experiment with New Zealand forest soils, including a stoney silt loam (pallic orthic brown soil) and a volcanic sandy soil (tephric recent), was significantly increased at a mean soil temperature of 23 °C compared to 10 °C over 26 weeks. Nitrogen mineralization also proceeds in cool soil temperature conditions <5 °C, albeit at a slow rate (Cookson et al., 2002; Opperman et al., 1989). This temperature dependent behaviour has important implications for biosolids N management to avoid NO<sub>3</sub><sup>-</sup> formation and potential losses through leaching from amended soils in regions with cooler, wet winters, due to the slower plant growth and decreased nutrient demands under these conditions.

Kinetic models of soil organic N mineralization generally assume that temperature affects the mineralization rate, but not the size of mineralizable N pool (Bernal et al., 1998; Garau et al., 1986; Smith et al., 1980; Stanford and Smith, 1972). However, more accurate predictions of N mineralization may be provided when the labile C or N pool are also considered as temperature dependent (Benbi and Richter, 2002; Ellert and Bettany, 1988; MacDonald et al., 1995; Zogg et al., 1997). For example, MacDonald et al. (1995) investigated the effects of temperature between 5-25 °C on N mineralization patterns in four forest soils during a 32 week incubation experiment. Contrary to other findings (e.g. Smith et al., 1980; Stanford and Smith, 1972), the rate constant estimated from a first order kinetic model of mineral N production  $(R^2 = 0.94, P = 0.001)$  was not consistently related to temperature, however, the mineralizable N pool was highly temperature dependent and increased under warmer soil incubation conditions. This behaviour suggested a temperature dependent factor may operate that can influence microbial access to soil organic N. It is possible, for instance, that a functional shift in the composition of soil microbial populations (Zogg et al., 1997) occurs in response to temperature increasing the degradation of more recalcitrant forms of organic N in warm soils. This important phenomenon, which may partially account for the larger mineralizable N fraction in biosolids-amended soils sometimes apparent in warmer climates (Tables 9–15), is further discussed in Section 8.

#### 5.6. Soil moisture

Increasing soil moisture up to field capacity raises the N mineralization rate of soil organic N at temperatures greater than 5 °C (Stanford and Epstein, 1974). However, the effect of moisture content on N availability in biosolids-amended soils may be more complex due to the presence of a source of labile organic matter. Pritchard and Rigby (2010) performed a short-term (45 day) laboratory incubation experiment and measured increased mineral N accumulation in unamended control soils with increasing soil moisture content between 25–100% of the gravimetric water holding capacity (GWHC), from 10 to 18 mg N kg<sup>-1</sup> dry soil (ds) in a sandy soil and from 16 to 25 mg N kg<sup>-1</sup> ds in a sandy loam soil. This represented an increase in the mineralization of soil organic N equivalent to 13% in the sandy soil and 7.5% in the sandy loam. Raising the moisture content of sandy soil amended with DMAD biosolids between 25-50% of the GWHC increased the net mineralization of biosolids organic N (i.e. subtracting mineral N supplied by biosolids and background soil mineral N) and the accumulation of soil mineral N from 29 to 33 mg kg<sup>-1</sup> dry soil (ds), but mineral N decreased to 22 mg N kg<sup>-1</sup> ds at 100% GWHC. In the sandy loam, soil mineral N accumulation from net mineralization of biosolids organic N decreased from 39 mg N kg<sup>-1</sup> ds at 25% GWHC to 11 mg N  $kg^{-1}$  ds at 100% GWHC. The smaller net accumulation of mineral N at 100% GWHC is likely to be a consequence of the loss of gaseous N through denitrification. This is caused by the formation of anaerobic microsites due to a greater proportion of water filled pore space and the presence of a source of labile organic matter to support heterotrophic microbial growth, which depletes soil O2 and promotes denitrification activity, the main mechanism of gaseous denitrification emissions from soil (Maag and Vinther, 1996; Rigby and Smith, 2013), discussed further in Section 7.2.

#### 5.7. Soil pH

Several reports demonstrate that, within the acidic to slightly alkaline range (<pH 8), raising the soil pH value increases N mineralization rate in biosolids-amended soil (Garau et al., 1986; Hseu and Huang, 2005; Huang and Chen, 2010). For example, Hseu and Huang (2005) found 3–34% of the TN content supplied by MAD and AeD biosolids was mineralized over a 48 week incubation period; more mineralizable N was released in a silt loam (fine-silty, mixed, hyperthermic Typic Hapludalfs) with pH 6.5, compared to a clay soil (fine, mixed, hyperthermic Plinthic Paleaquults) and a sandy soil (sandy, mixed, hyperthermic Typic Udipsamments), with acidic pH values of 4.7 and 5.7, respectively. Contrary to these findings, Terry et al. (1981) found no significant effect of pH values between 5.3–7.5 on the decomposition rates of sludge in silty clay and silt loam soil types.

The optimal pH range for many soil microbial oxidisers is pH 7–9, and nitrification is negligible below pH 4.5 (Nieder and Benbi, 2008). Nitrification of biosolids compost N was inhibited at soil pH values of 5–6 (Tester et al., 1977; Terry et al., 1981), and in an acidic (pH 4.5), sandy soil (tephric recent) amended with aerobically and anaerobically digested biosolids (Wang et al., 2003b). Thus, soil pH is unlikely to be a major factor controlling N mineralization in biosolids-amended soil at typical managed agricultural soil pH conditions (>pH 6.0 H<sub>2</sub>O); below this range sludge application is restricted to protect soil from the long-term accumulation of potentially toxic elements (DoE, 1996).

#### 6. Nitrogen fertilizer replacement value

#### 6.1. First year nitrogen uptake

The N fertilizer value of dewatered biosolids is strongly dependent on the mineralizable N fraction, as most of the N is present in organic forms, whereas, in liquid biosolids, the N fertilizer value depends more upon the NH<sub>4</sub>–N content. Adjei and Rechcigl (2002) conducted an experiment with Bahia grass (*Paspalum notatum*) on a fine sandy Pomona soil and compared the agronomic value of liquid AeD biosolids, liquid LT biosolids, LT biosolids cake and ammonium nitrate supplied at rates of 90 and 180 kg N ha<sup>-1</sup>, and an unfertilized control. They found that forage production from the liquid biosolids was similar to inorganic fertilizer, whereas crop yield was approximately 30% less for the cake treatments (at equivalent rates of TN application).

Many studies demonstrate biosolids are efficient sources of N for crop growth (Adegbidi and Briggs, 2003; Adjei and Rechcigl, 2002; Akdeniz et al., 2006; Cogger et al., 2001; Morris et al., 2003). Mineralizable N values of various biosolids types determined through field investigations of crop uptake of N in biosolids-amended soils (in the first year after biosolids application) are presented in Tables 9–15. Mineralizable N determined by laboratory incubation of biosolids-amended soil to track soil mineral N accumulation kinetics also displays similar patterns to the N uptake from, and N equivalency of, biosolids applied to field production crops. Thus, laboratory incubation is an effective and reliable means of predicting available N release from biosolids and provides an adjunct to developing fertilizer recommendations relevant to biosolids applications under field crop production conditions. For example, mineralizable N values for DMAD biosolids types (excluding air-dried types) obtained by field investigation, in the year following application, were in the range 10.5–45.4% organic N, and those determined by laboratory incubation were similar and generally in the range 15.2–43.5% (Table 11). The consistent results observed between both field and laboratory incubation tests therefore also demonstrate that, in most situations, the majority of mineralizable N is released rapidly in biosolids amended soil in the year of application and typically over a much shorter time period.

The reproducibility of crop response to biosolids N was investigated using perennial ryegrass (Lolium perenne) as a test crop by Morris et al. (2003) for a wide range of biosolids types, including those produced from conventional sludge treatments, such as MAD, and enhanced treatments and pretreatments, for example, thermal drying and thermal hydrolysis, respectively. Biosolids were applied to a loamy sand/sandy loam soil to separate experimental plots in three consecutive years of field trials and the ryegrass response (yield and N offtake) was compared to inorganic N fertilizer. This major experimental programme in the UK showed that the biosolids performance was relatively consistent between years and that, despite differences in mineralizable N values, PAN was broadly comparable between most biosolids types treated by different processes and pretreatments (Table 16), with the exception of composted material, which had a much smaller N availability, as reported in many other studies (e.g. Lynch et al., 2004). For example, the PAN content was consistent for DMAD biosolids from three major wastewater treatment plants (WWTPs) and was in the range of 27-34% of TN, equivalent to a mineralizable N content of 16-21% organic N; the overall mean PAN for the DMAD biosolids tested was 30.5% TN (Table 16). For thermally dried MAD biosolids (TDMAD) the range of PAN was 34-38% of TN, equivalent to 32-37% of organic N. Thermally dried MAD and raw sludge types gave broadly similar mean PAN values equivalent to 34.9% and 29.4%, respectively, with an overall mean equivalent to 34.4% TN (Table 16). A substantial mineral N fraction is lost by volatilization during thermal drying, however, N availability was maintained in TDMAD biosolids due to the increased size of the mineralizable N pool upon heating. This was also supported by the observations of Smith and Durham (2002) in a soil incubation study, which showed that TDMAD prepared with digested sludges from different WWTP contained consistently larger pools of mineralizable N compared to equivalent mechanically dewatered materials.

Further evidence of the consistent effects of biosolids applications on crop N response and as an effective N fertilizer source for agricultural production has also been provided through other extensive fieldbased research. For example, Barbarick and Ippolito (2000) conducted a six-year investigation at two different sites to determine the N fertilizer equivalency of sand-bed dried MAD biosolids supplied to wheat. They found that R<sup>2</sup> values of regression relationships between biosolids application rate and N uptake by grain were generally highly significant, and first-year N mineralization rates were relatively consistent between years, in the range of 25-32% organic N. Subsequently, however, the variation in N uptake by wheat increased, but this was not necessarily linked to the variability in biosolids N supply, as significant compounding effects from crop water stress due to drought conditions and also hail damage were noted (Barbarick and Ippolito, 2007); therefore, these extenuating factors reduced first-year biosolids N mineralization rates to a degree and in the range of 21-27% organic N.

Mineralizable N values vary between different biosolids types (Section 4.1), however, research in the US (Cogger et al., 2006; Gilmour et al., 2003) considering the effects of climate on biosolids decomposition in soil also found that, with the exception of extensive stabilization methods, such as composting or lagoon storage, sludge treatment process did not have a significant effect on the overall PAN value (mineralizable N plus mineral N) of different types of biosolids product (e.g. Gilmour et al., 2003, Table 16). Due to the similarity in N release across a range of biosolids types, Gilmour et al. (2003) used mean biosolids laboratory decomposition data and weather data to model PAN during the growing season across the US, and recommended this as a more suitable approach than defining PAN based on biosolids treatment process. This study particularly demonstrated the importance of quantifying biosolids N mineralization at a local regional level and taking seasonal and environmental factors into consideration.

These research programmes demonstrate that the PAN content of biosolids is generally relatively consistent between different treatment processes (except for composting processes or long-term storage, which consistently give much smaller PAN values) within regional areas (Table 16). Nevertheless, there appear to be quite significant variations in mineralizable N (and hence PAN values) reported between different regions internationally. For instance, mineralizable N and PAN data from the UK are generally smaller than the values measured elsewhere as shown in Tables 9–16, and as discussed in Section 5.1.2. It is possible that this reflects regional and local differences in the operational practices employed in upstream wastewater treatment, such as the proportions of primary and activated sludge types entering sludge treatment, and the relative effectiveness of operation of sludge treatment-stabilization processes at different WWTPs (Smith, 2014) (Section 8). Further work is necessary to elucidate the impacts of these upstream management factors on biosolids mineralizable N, and researchers are also encouraged to provide more comprehensive descriptions of these conditions to better characterize specific biosolids types used in N fertilizer research to assist interpretation of biosolids N mineralization results.

#### 6.2. Residual and slow-release value

Consolidated biosolids types produced by mechanical or other forms of dewatering or drying process are generally considered to provide a 'slow-release' source of N (Adegbidi et al., 2003). Whilst the rate of N release from organic N mineralization in biosolids is slower than standard soluble inorganic fertilizers, the organic N fraction typically mineralizes within a few weeks in soil and is mostly released during the first season of application as discussed in Section 6.1. The US and the UK biosolids recommendations on fertilizer value (US EPA, 1995; Defra, 2010) suggest that for DMAD biosolids, 10 and 5% of biosolids organic N is mineralized in the second and third years, respectively. However, earlier research on which the recommendations were partly based was often conducted with greater rates of biosolids than are typically applied for agricultural purposes and due to regulatory N restrictions. For example, the UK biosolids fertilizer guidelines were partly based on research involving large rates of application equivalent to 28-84 t DS ha<sup>-1</sup> and 742–2230 kg N ha<sup>-1</sup> (Hall, 1983) and, under such conditions, it is possible to apparently develop a slowly mineralizable residual N pool in the amended soil. For example, Boyle and Paul (1989) reported a mineralizable N fraction remained three years after the cessation of anaerobically digested cake application at high rates of between 11.3 and 45.0 t DS ha<sup>-1</sup> year<sup>-1</sup> for a period of 8 years. Gilmour et al. (1996b) investigated the effects of biosolids inputs to a dedicated disposal site over the previous 7 years on fresh AeD biosolids decomposition in the subsequent year, to improve predictions of the release of N from the residual biosolids fractions in the amended soil. A labile fraction of the residual biosolids equivalent to 6 to 10% of the total residual biosolids C pool was detected, and first-order rate constants of the total pool of residual biosolids C were similar to those determined for more labile portions of soil organic matter. Consequently, there was evidence of residual biosolids decomposition in the period following application, but the rates of biosolids supplied were extremely high and equivalent to a total input of 418–647 t ha<sup>-1</sup> DS over a period of seven years and were therefore unrepresentative of current agronomic biosolids practice. Decomposition of the fresh biosolids addition followed a two phase process, with rapid and slow phases, but the first-order rate constants for these phases and percentages of fresh biosolids in the rapid fraction were independent of previous additions of biosolids.

Other reports of the residual N value of biosolids applications may be explained by the apparent conservation of the readily mineralizable N fraction due to a combination of biosolids management and environmental reasons that slow or prevent microbial mineralization transformations. For example, in a field experiment to assess N recovery from heat-dried and dewatered biosolids, surface-applied to forage grasses at rates of 200–600 kg TN ha<sup>-1</sup>, Cogger et al. (1999) found substantial mineralization of biosolids N in the second year. In that case, the mean N fertilizer equivalency increased from 22 to 60% at one site and 38 to 54% at the second site, in the first and second years of the experiment, respectively. This behaviour was probably explained by the occurrence of dry conditions at the soil surface in the summer followed by cool winter temperatures, which delayed the mineralization of biosolids N until the following Spring. In later work, Cogger et al. (2004) found that first year PAN for tall fescue growth on a well-drained fine sandy loam at a site near Seattle was similar across a range of biosolids treatment types (surface-applied at rates of 400–500 kg TN ha^{-1}) and was 37  $\pm$ 5%, with the exception of lagooned biosolids, where the PAN ranged from 8 to 25% depending on the age of the biosolids. Second year PAN was dependent upon biosolids treatment type, and was  $13 \pm 2\%$  for dewatered biosolids and was 50% smaller for thermally dried biosolids, demonstrating a rapidly mineralizable N pool in this biosolids type. Second year N values observed in both of these studies (Cogger et al., 1999, 2004) were probably a consequence of reduced rates of mineralization in the first year, which have been observed for surface applied biosolids (Castillo et al., 2011; Jaynes et al., 2003), further exacerbated by unfavourable environmental conditions. Evidently, N availability of surface-applied biosolids is difficult to predict and a residual value can be highly variable and dependent upon site specific conditions, due to local environmental and management factors that influence the rate of mineralization of N, rather than due to intrinsic slow-release properties of biosolids themselves.

Research completed in the UK (Morris et al., 2003; Rigby and Smith, 2014) has shown that, for biosolids incorporated into soil in the Spring and exhaustive uptake cropping by ryegrass, all of the agronomically significant N was released in the year of application and there was no detectable residual N value in subsequent years for biosolids applied at standard agronomic rates. The apparent residual N effects sometimes recorded for biosolids in temperate regions (Boyle and Paul, 1989; Hall, 1983; Soon et al., 1978) could therefore be potentially explained by the carry-over of unused mineral N into the second season rather than from a significant mineralization of residual organic N in the year following application. Significant residual N release from mineralization of a very slowly degradable pool in subsequent years following biosolids application may be detectable and has been reported when atypical, large rates of biosolids are supplied that would normally exceed crop requirements for N under standard agronomic practice. Consequently, considering the residual N fertilizer value of biosolids is generally irrelevant in typical agricultural situations. However, the residual fraction has value in land reclamation where large rates of addition (e.g. equivalent to  $\geq 1$  t TN ha<sup>-1</sup>) are necessary to establish soil ecological restoration (Bradshaw and Chadwick, 1980; Coker et al., 1982). Other circumstances could be envisaged that lead to an apparent residual effect, due to conservation of the readily mineralizable N, for example, where dried biosolids are applied to dry soil, or where cold environmental conditions limit microbial activity slowing the rate of N mineralization thus conserving the readily mineralizable N for a longer period until more favourable conditions return.

The similarity between first year mineralization rates measured in the field compared to mineralizable N contents determined by laboratory incubation shown in Tables 9–15 provides further confirmation that most of the labile organic N is released in the first year of application for soil-incorporated biosolids and that there is negligible residual N supplied from standard agronomic rates of biosolids addition. This behaviour can therefore simplify agricultural N fertilizer recommendations for incorporated biosolids, which can exclude second and third year N availabilities for the practical purposes of calculating supplementary mineral N fertilizer application rates.

#### 7. Losses of nitrogen from biosolids-amended soil

#### 7.1. Established nitrogen loss mechanisms

Nitrogen may be lost from biosolids-amended soil by several established mechanisms, which include leaching, runoff or gaseous emissions, caused by ammonia volatilization and denitrification. Leaching losses of N from soils occur mainly as NO<sub>3</sub><sup>-</sup> because of the low capacity of most soils to retain anions, whereas NH<sub>4</sub><sup>+</sup> is less mobile and forms exchangeable associations with negatively charged soil minerals (Tester et al., 1977; Shen et al., 1997). Leaching of fertilizer NO<sub>3</sub><sup>-</sup> to groundwater is a significant route of N loss from agricultural systems, and a major cause of groundwater and surface water NO<sub>3</sub><sup>-</sup> contamination (Addiscott, 2005). Nitrate leaching and contamination of water bodies is a function of a combination of many complex factors, including: N fertilizer rate and timing, rainfall, irrigation, soil type, depth of the aquifer and geology. In general, sandy, coarse-textured soil types have lower water holding capacities than fine-textured soils with greater clay and organic matter contents, and hence movement of soluble  $NO_3^-$  through the soil profile in sandy soils is more likely (Corrêa et al., 2006; Rigby et al., 2009); however, the presence of a crop reduces leaching losses due to the uptake of N and reduced soil-water transport by evapotranspiration (Cogger et al., 2004; King and Morris, 1974; Santibanez et al., 2007).

Timing of biosolids applications is critical to minimize leaching losses associated with the seasonal differences in rainfall (Fumagalli et al., 2013; Hall, 1983, 1984). Under UK conditions, Hall (1983, 1984) observed that 50-70% of the N in liquid sludges was available in the first year of application when applied in Spring and incorporated into the soil, whereas in early winter relative availability was reduced to 20-30% due to N losses through leaching by winter rainfall. Two major research programmes were completed by WRc (Smith et al., 1994, 1995) and ADAS (Misselbrook et al., 1996; Shepherd, 1996) in the 1990s in the UK to investigate N losses from biosolids-amended soil. In the UK, declining soil temperatures from October reduce the rate of nitrification,  $NO_3^-$  accumulation and the potential risk of N leaching from biosolids-amended soil. Thus, Smith et al. (1994) found that N recovery from LMAD applied and incorporated into the soil increased between October-December from 25 to 55% of total applied N over this period for winter sown wheat. Additionally, when biosolids were shallow injected, placing the available N in close proximity to crop roots, more than 50% of the available N supplied in biosolids in August, September and October was recovered by the winter wheat crop (Smith et al., 1995). As may be expected, sewage sludge varied in leaching potential depending on the available N content (Misselbrook et al., 1996; Shepherd, 1996; Smith et al., 1995). Thus, losses were greatest for LMAD (11–19% TN), followed by liquid undigested sludge (8–12%), with the smallest N loss recorded for DMAD (6-8%) (Smith et al., 1995). Surface application of biosolids may reduce leaching losses of N by 50% compared to soil incorporation (O'Brien and Mitsch, 1980; Smith et al., 1994), however, this is largely because it promotes N emissions via NH<sub>3</sub> volatilization, which are also undesirable.

Following applications of biosolids, N  $(NH_4^+, NO_3^-, organic N)$  in soluble and particulate forms, may be lost by soil erosion by wind or water or run-off of surface waters when the rate of precipitation exceeds the soil infiltration capacity (Pierzynski et al., 2005; Ojeda et al., 2006). This is also dependent upon factors such as the gradient of the soil surface, soil structural properties and crop cover (Ojeda et al., 2006).

Leaching and runoff are common risks to all sources of fertilizer N (both conventional and organic sources), and BMPs (outlined in Section 2) include restrictions that are designed to prevent such nutrient losses. For instance, in the UK these include closed application periods for organic manures with high N availability (>30% of TN is in readily available forms) (Defra, 2009b). These are extended for more vulnerable soil types and on tillage land. However, the effectiveness of BMPs at controlling NO<sub>3</sub><sup>-</sup> concentrations in water resources has been inconsistent and difficult to quantify, emphasizing the need for sciencebased performance assessments to determine the most suitable measures to protect the water environment (CCA, 2013). An advantage of organic sources of N, such as dewatered biosolids, over inorganic forms, is that they may reduce the loss of N from the crop root zone, because mineralizable N is released over a period of weeks, in contrast to the high solubility and transport risk of mineral NO<sub>3</sub><sup>-</sup> fertilizers (Eldridge et al., 2009; Esteller et al., 2009). Thus, to some extent, the release of mineralizable N from biosolids may more closely match crop uptake in comparison to applications of inorganic fertilizer. However, if applied in excess of crop requirements, the mineralization of biosolids organic-N may exceed crop uptake of N leading to surplus  $NO_3^-$  in the soil, which is vulnerable to leaching by winter rainfall. Thus, accurate estimation of the mineralizable N in biosolids is necessary to ensure efficient utilization of combined biosolids and supplementary fertilizer N inputs to agricultural systems to avoid wastage and losses of N to the environment.

Nitrogen may be lost from the soil as a gas via NH<sub>3</sub> volatilization (Pierzynski et al., 2005), promoted by surface application of  $NH_4^+/NH_3$ containing fertilizers, high soil pH, which increases the free NH<sub>3</sub> concentration, drying weather conditions, increased temperature and wind speed, and low soil cation exchange capacity (CEC) (Freney et al., 1983; Pierzynski et al., 2005). However the risk of NH<sub>3</sub> volatilization can be eliminated by incorporating N sources into the soil by tillage or injection (Beauchamp et al., 1978; Robinson and Polglase, 2000), or significantly reduced by spreading to moist soil with a crop cover (Ojeda et al., 2006). Surface applied, dewatered biosolids are less able to penetrate the soil surface than liquid types and volatilization losses of 68-81% of NH<sub>3</sub>-N from dewatered MAD and AeD biosolids have been observed in the first week after spreading (Robinson and Polglase, 2000), compared to 60% for LMAD biosolids (Beauchamp et al., 1978). Significant volatilization losses of NH3 released from the mineralization of organic N in surface applied thermally-dried biosolids also provide a likely explanation for the reduced yield performance of surface applications of these biosolids types compared to soil incorporation (Smith and Hadley, 1988).

#### 7.2. Denitrification emissions

A further pathway of N loss from the soil is denitrification, the reduction of  $NO_3^-$  or  $NO_2^-$  to  $N_2$  gas. The process is mediated by chemoheterotrophic bacteria under anaerobic conditions, where  $NO_3^-$  is used as an alternative electron acceptor to oxygen ( $O_2$ ) during organic matter decomposition (Pierzynski et al., 2005). Denitrification is enhanced under  $O_2$  deficient conditions, in waterlogged soils, for example, or as a result of rapid depletion of  $O_2$  by soil microorganisms due to high organic C availability (Mendoza et al., 2006; Thangarajan et al., 2013). The final product of denitrification, dinitrogen gas ( $N_2$ ) is inert; however, the intermediate nitrous oxide ( $N_2O$ ) possesses 310 times the global warming potential of  $CO_2$  (Brown et al., 2012).

As is the case with any organic manure type, incorporation of biosolids into the soil may encourage losses of N via denitrification (Mendoza et al., 2006). There are few data available that directly quantify gaseous N losses from biosolids by denitrification. However, in a laboratory investigation, Pu et al. (2010) examined the effects of biosolids type, soil type and polymer addition on the mechanisms and extent of both denitrification and NH<sub>3</sub> volatilization at a soil incubation temperature of 30 °C. The experimental procedure included wetting and drying cycles at moisture contents between 75%–150% of GWHC. Ammonia losses over 72 days were minimal, accounting for <4% of the applied NH<sub>4</sub>–N; however, 24% of TN applied in MAD biosolids and 29% for AeD biosolids was lost through denitrification over 105 days. Thus, denitrification represents potentially a major pathway of gaseous N losses, particularly under warm and moist soil conditions (Pu et al., 2010).

Hsieh et al. (1981) investigated N mineralization kinetics in a sandy loam soil amended with air-dried and ground activated sludge and MAD biosolids applied at rates between 0–12% ds under two different moisture conditions, 0.33 to 0.06 bar (0.1 bar is typically equivalent to approximately 100% GWHC). No inorganic N accumulated in the 0.06 bar moisture condition at the high biosolids application rates, although organic N was depleted. The results showed that the more stabilized MAD biosolids-N was less susceptible to denitrification of mineralizable N in soil at high moisture content compared to the highly labile C source supplied by activated sludge. Indeed, in moist soil conditions at the 12% ds application rate, all of the apparently mineralized N applied in activated sludge was unaccounted for compared to 70% in the soil amended with MAD biosolids.

More recently, Rigby and Smith (2013) investigated N transformations in biowastes-amended soil in a short-term soil incubation study; digestates of food waste, farm waste, biodegradable municipal solid waste (MSW) and sewage sludge biosolids (DMAD) were incubated with different soil types under constant temperature and moisture conditions (25 °C and 50% GWHC). This investigation provided indirect evidence that substantial gaseous loss of N occurs, with the associated increased risk of greenhouse gas (GHG) emissions, due to the simultaneous nitrification-denitrification of N under conditions of high labile C supplied in organic residuals, and/or the granulate physical structure of digestate materials, in fine-textured soil types. However, stabilized DMAD biosolids were shown to represent a smaller potential risk of denitrification losses and emissions than other aerobic or granular digestate types from municipal sources. This can be explained by the more effective stabilization of biosolids organic matter during AD treatment of sewage sludge compared to aerobic digestion processes, and the ability to disperse the material effectively into the soil compared to more granulate forms. Understanding of these mechanisms is limited, therefore, further research is required to quantify the interactive effects between soil type and biowaste stability and physical properties and subsequent denitrification losses and associated GHG emissions.

The International Panel on Climate Change (IPCC) inventory guidelines provide N<sub>2</sub>O emissions factors for different sources of N in soils including N applied in fertilizers (IPCC, 2006). The default value for N<sub>2</sub>O emissions from soil fertilized with synthetic N fertilizers or organic N fertilizers, including biosolids, is 1% of the TN applied. A key benefit of using biosolids as alternative N sources to inorganic fertilizers is the offset of GHG emissions produced during synthetic fertilizer manufacture. Denitrification is dependent on mineral N availability in the soil and since only a proportion of the N supplied in biosolids, as well as other organic N sources, is available and released over a period of weeks, only a fraction is potentially accessible to denitrification mechanisms at any one time, whereas mineral N fertilizers represent a source of immediately available N. For stabilized biosolids, the N availability measured in different temperate soil systems is consistent and predictable (e.g. Morris et al., 2003; Rigby et al., 2009). Therefore, it may be more reasonable to base N<sub>2</sub>O emission factors on PAN release kinetics rather than the TN content of biosolids or other organic manures. Thus, it is likely that GHG inventories for biosolids (and other types of organic manure) applications based on the current 1% default value overestimate the contribution of these soil emissions to the overall life cycle C emissions such that full credit may not be received for the GHG mitigation potential of land application. Better understanding of these emissions is required to improve the accuracy of carbon-footprint estimates (e.g. Barber, 2008) used to inform investment decisions and selection of optimal management and treatment strategies for bioresources with the potential for land application.

# 8. Scientific analysis and development of nitrogen management recommendations for biosolids to improve nitrogen use efficiency

#### 8.1. Nitrogen recommendations for biosolids in temperate regions

Some inconsistencies were identified from the review of international literature between recommended PAN or mineralizable N values in national guidelines and actual measured values (Tables 1 and 9-16). In the UK, for instance, N release data for different biosolids types provided by a comprehensive programme of field experiments (Morris et al., 2003) suggest that PAN may be underestimated in national fertilizer recommendations for certain types of biosolids (Defra, 2010). For example, over three growing seasons, Morris et al. (2003) found mean mineralizable N values for DMAD of 17.2%, nearly three times the value of mineralizable N implied by the PAN value given in Defra (2010) (Table 1), and this was also confirmed in other field trials with DMAD biosolids applied to contrasting soil types by Rigby et al. (2009). An explanation for the apparent inconsistency may be provided by understanding the historical development of the fertilizer guidance for sewage sludge in the UK (Hall, 1986). This is because the advice initially proposed for mechanically dewatered digested biosolids was based on earlier field trials with air-dried, digested biosolids types (for example, see Coker et al., 1982; Hall and Williams, 1983), which contain little mineral N due to the volatilization of the NH<sub>3</sub> content, and it was assumed that soluble nutrients were largely removed during mechanical dewatering, therefore, a PAN value of 15% of TN (later increased to 20% of TN), corresponding approximately to only the mineralizable fraction, was assumed as the basis to the fertilizer recommendation for digested sludge cake (Hall, 1986).

#### 8.2. Nitrogen recommendations for biosolids in warm climates

Inconsistencies were also identified between recommended PAN or mineralizable N values and actual measurements of these biosolids-N properties in warmer climates, which have probably arisen for different reasons. For instance, field investigations conducted in Australia, in Victoria, New South Wales, Western Australia and Queensland (Al-Dhumri et al., 2013; Eldridge et al., 2008; Rigby et al., 2010; Pu et al., 2012), indicate that the first year mineralization rate is much greater than the 15% and 25% of organic N for anaerobic and aerobically digested biosolids, respectively, suggested in Australian guidelines (e.g. NSW EPA, 1997; DEC, 2012). For example, in Queensland, where the climate is sub-tropical, field experiments with arable crops on clay soils (Red Ferrosol, Grey Vertosol, Yellow Chromosol, Black Vertosol) (Pu et al., 2012) indicated that the mean N mineralization of anaerobic and aerobic biosolids over approximately 12 months was equivalent to 35% and 58% of organic N, respectively. Furthermore, there was evidence of extremely rapid rates of mineralization, which meant that a large amount of mineral N was potentially vulnerable to loss from the soil unless the release coincided with uptake by a growing crop. In another example, Eldridge et al. (2008) investigated the proportion of biosolids organic N mineralized over 12 months in a field incubation study using PVC tubes to isolate the amended soil from plant root access, on a silty clay loam (Red Chromosol) in South West Sydney. The mineralized N was equivalent to 45–54% of organic N for TD biosolids (Table 13) and 53% organic N for dewatered AeD biosolids (Table 10), with most of this mineralizing rapidly at the start of the incubation (Eldridge et al., 2008).

Incubation studies with biosolids-amended soils, performed at optimal temperatures for microbial activity (25 °C) using soils from temperate climates (Smith et al., 1998a, 1998b, 1998c), correlate with estimates of biosolids N mineralization measured under these field conditions (Morris et al., 2003; Rigby et al., 2009). Therefore, as discussed above (Sections 4.2, 5.1.2 and 5.5), it would be reasonable to assume that soil temperature increases the rate, but not the overall extent, of applied organic N mineralization because the most recalcitrant forms of organic N are not accessible to microbial attack (e.g. Stanford and Smith, 1972). However, much greater mineralization of organic N is frequently observed for similar sludge types in warmer climate conditions (Tables 9–15). This may be due to differences in sludge treatment and stability properties, however, there is also evidence suggesting a possible correlation between soil temperature and the size of the mineralizable N pool in soil types in warmer climates (Benbi and Richter, 2002; Ellert and Bettany, 1988; MacDonald et al., 1995; Zogg et al., 1997). Therefore, the increased mineralization of biosolids N observed in warmer climates may reflect an adaptation of soil microbial communities to decompose more recalcitrant forms of organic N under these conditions and soil types where rapid mineralization rates are observed.

There is a limited amount of fundamental soil process evidence available to support this suggestion. For example, Naramore (2010) detected larger concentrations of lignin-degrading enzymes in heated soils compared to control soils from a long term soil-warming experiment where a forest soil was heated in situ by 5 °C above the ambient temperature using thermal cables (Bradford et al., 2008). Therefore, soil microbial communities in sub-tropical and warm climates such as Queensland and Western Australia could be adapted to more efficiently exploit soil organic N reserves due to the competition for substrate resources under these conditions. Consequently, they may be potentially more effective at mineralizing organic N applied to soil in biosolids compared to temperate soils and this behaviour may partially explain the greater mineralizable N values observed in these regions.

#### 8.3. Effects of upstream wastewater and sludge treatment processes

Larger pools of mineralizable N have also been determined in other regions of Australia, such as New South Wales, Victoria and Tasmania (Eldridge et al., 2008; Al-Dhumri et al., 2013; Ives et al., 2010) with more temperate climates compared to values for similar biosolids types measured in the UK for instance. For example, the overall mean temperatures at the two experimental sites of Al-Dhumri et al. (2013) were 15.6–16.4 °C over the growing season, which is within the range of mean daily temperatures of 10-31 °C observed by Rigby et al. (2009) in the UK. Nevertheless, Al-Dhumri et al. (2013) measured a mineralizable N content in DMAD biosolids equivalent to 40% of the organic N compared to a consistent value of approximately 17.0% determined in the UK for the same biosolids treatment type (Morris et al., 2003; Rigby et al., 2009). In a laboratory investigation study of the N mineralization characteristics of different biosolids types produced in Tasmania, Australia, Ives et al. (2010) observed a mineralizable N content for DMAD biosolids of 35% organic N in a Brown Sodosol after 56 days of incubation at 12.5 °C.

Factors other than soil environmental conditions also therefore potentially dictate the size of the mineralizable N pool in similar applied biosolids types. Indeed, the apparent differences in mineralizable N contents of similar types of biosolids may be potentially explained by varying upstream treatment processes applied to sewage sludge and the overall level of stabilization of the organic matter achieved during treatment. For instance, activated sludge tends to contain greater concentrations of N, and has a mean TN content of 5% DS compared to 2.5% in primary sludge (Smith, 2014). Furthermore, activated sludge is less susceptible to AD processes compared to primary sludge, and the volatile solids (VS) destruction of activated sludge is approximately 25% less compared to primary sludge. This is because activated sludge consists of a large proportion of microbial biomass and the cell walls provide protection against biodegradation (Smith, 2014). Therefore, one possible explanation is that the relative proportions of activated sludge and primary sludge mixed together for digestion could significantly influence the overall level of stabilization, and hence the apparent mineralizable N present in the final digested product. The effectiveness of the sludge digestion process itself is a further factor controlling the mineralizable N content of the treated biosolids. For example, the DMAD biosolids investigated by Al-Dhumri et al. (2013) was apparently less well stabilized (VS content equivalent to 66% on a DS basis), and this may have contributed to the larger mineralizable N content (40% of organic N), and PAN content (41% TN, Table 16) compared to DMAD samples from the UK (VS range: 55–65% DS; mineralizable N: 17% of organic N; PAN: 30.5% TN) (Morris et al., 2003; Rigby et al., 2009). Unfortunately, although the VS content of sludge is a routinely measured parameter in operational practice at WWTP, it is rarely reported in biosolids N mineralization studies.

Plant Available N values were also correspondingly raised in regions where mineralizable N was increased (Table 16), demonstrating that larger mineralizable N contents do not simply represent a shift in the balance of mineralizable to mineral N, rather a greater overall total proportion of available N. However, the critical relationship between wastewater treatment processes, the balance of primary and activated sludge, and the efficiency of sludge stabilization processes and the extent and behaviour of the mineralizable N fraction in biosolids has largely been neglected in biosolids N fertilizer investigations. This is a fundamental and critical area requiring further study and is essential to fully interpret biosolids N mineralization behaviour. Research is also necessary to quantify the impact of advanced AD conditions and sludge pretreatments on mineralizable N in these biosolids types.

As may be expected, the range of biosolids types presented in the international controls (Table 1) generally reflect the types of biosolids produced and used in practice in specific regions. However, there are some cases where important biosolids management practices are omitted from regional guidelines. For instance, pan-drying and long-term storage is widely adopted for sludge consolidation at major WWTPs in Australia and this practice profoundly affects and reduces the total and mineralizable N content in biosolids (Rouch et al., 2011). Australian controls do not currently include specific advice on the N fertilization properties of these biosolids types; however, this supplementary information would assist local operators to supply appropriate rates of biosolids treated by these processes and mineral fertilizers to meet crop requirements for N.

#### 8.4. Best management practices

Gaseous emissions are a potential route of N loss from biosolidsamended soils (Section 7). International biosolids guidelines recommend incorporation of biosolids into the soil, and this practice effectively eliminates NH<sub>3</sub> volatilization losses (Beauchamp et al., 1978; Robinson and Polglase, 2000). Denitrification is potentially a significant pathway for loss of N in warm, moist and fine-textured soils, and further research is required to determine the extent of denitrification in biosolids-amended soils in a range of climate types, in particular to define the interactions between soil type and biosolids type. For example, the stability of biosolids organic N may have a critical impact on potential denitrification losses in fine-textured soil types (Rigby and Smith, 2013). Furthermore, clay soils may frequently be targeted for biosolids applications to reduce leaching of NO<sub>3</sub>-N that occurs to a greater extent in sandy soil types. Hence, further research is required to examine the influence of soil type and biosolids type on denitrification losses from amended agricultural soils to also inform BMPs that minimize denitrification losses of N, improve fertilizer replacement value, and reduce potential climate change impacts of N<sub>2</sub>O emissions. This is also necessary to ensure default values for N emissions used in carbon-footprint calculations (IPCC, 2006) for the agricultural utilization of biosolids are accurate and appropriate.

#### 9. Conclusions and recommendations

#### 9.1. Key outcomes of the review

 The most robust approach to defining the N fertilizer value of different biosolids types is by field investigation, but soil incubation and pot trials are also reliable means of determining the potentially mineralizable N content.

- The amount of mineralizable N in biosolids is proportional to the total organic N content, and the degree of organic matter stability. The survey indicated a mean mineralizable N fraction for biosolids stabilized by microbiological treatment processes of approximately 47.2% organic N (range 32.1–57.5%) for AeD biosolids, 29.8% organic N (range 10.5–45.4%) for MAD biosolids and 6.7% organic N (-10-24.5%) for Com biosolids.
- The mean mineralizable N value for LT biosolids is 34.4% organic N (range 2.6–65.1%), and varies depending on whether the biosolids have previously undergone biological stabilization.
- Thermally dried biosolids have similar mineralizable N values to AeD biosolids, equivalent to 40.1% organic N (range 26.0–71.0%); this may be due to thermal changes in the structure of organic compounds increasing microbial accessibility for decomposition.
- Storage and drying methods have a significant impact on the total and mineralizable N contents found in biosolids. Lagoon stored and airdried biosolids have lower TN and mineralizable N contents, for instance, compared to mechanical dewatering, due to the degradation and stabilization of organic N and gaseous emissions of N that occur during extended treatment periods.
- The overall PAN values obtained for dewatered biosolids types (except for compost) are generally similar, within climatic regions, and decreases in mineral N content (as in TDMAD compared to DMAD biosolids) are thus matched by an increase in or greater mineralizable N and vice versa. Composted and other exceptionally stabilized biosolids types consistently have much small PAN and mineralizable N values.
- The rate of N mineralization in biosolids-amended soil is influenced by the application rate, soil type, temperature, moisture content and pH value. Nevertheless, the overall N value of similar biosolids products from different WWTP appears relatively consistent within regional areas and for different soil types. However, wide variations were apparent in international measurements of mineralizable N and PAN for similar biosolids types across different regions, which are difficult to reconcile. Climatic factors, particularly soil temperature, influence the rate of mineralization. However, the overall extent of mineralization is also potentially increased in soils in warmer climates compared to cooler, temperate conditions. This may be explained by the adaptation of microbial communities to degrade more recalcitrant forms of organic N in warmer climatic regions compared to temperate soils. However, soil environmental effects do not fully account for the differences in overall mineralizable N observed.
- Variations in upstream wastewater treatment processes and, in particular, the relative proportions of primary and activated sludge, may also influence the final stability and N properties of treated biosolids. The operation of sludge stabilization treatments at different WWTPs further impact the mineralizable N properties of similar biosolids types and, together, these operational factors could contribute to the apparent variation observed in mineralizable N and PAN between regions. Therefore, accurate estimates of the mineralization rate and mineralizable N fraction for a given biosolids type depend on local measurements of biosolids N mineralization to accommodate the effects of regional seasonal, environmental and operational factors.
- Evidence from the literature suggests that mineralization rates of biosolids N are relatively rapid. First year mineralizable N values calculated from field data are similar to laboratory incubation results and show that most of the mineralizable fraction of organic N is rapidly released and available for crop uptake in the first year after application.
- Recent research in the UK has indicated that reports of residual N in temperate regions are likely to be associated with the carry over of unused mineral N into the second season, or atypical and large application rates, rather than from mineralization of slowly released residual organic N at normal agronomic rates of biosolids application. However, several investigations have observed second year mineralization of biosolids N, but under circumstances where there may be a delayed release of the readily mineralizable fraction

of N due to unfavourable climatic and seasonal conditions following application, such as low temperature or moisture content conditions, which restricted the rate of N mineralization in the first year.

- Reducing NO<sub>3</sub><sup>-</sup> leaching from agricultural land and particularly from land receiving applications of organic manures, including biosolids, has been a prime focus of BMP development. These measures quantitatively reduce drainage losses and wastage of applied N, but their effectiveness at controlling NO<sub>3</sub><sup>-</sup> concentrations in environmental water sources overall are less clear because of the high background emissions of NO<sub>3</sub>-N from tillage soil.
- Gaseous losses of N may occur by NH<sub>3</sub> volatilization when biosolids are surface-applied or by denitrification for certain combinations of high risk soil and biosolids types, with subsequent potential for GHG emissions.
- Whilst current BMP land management controls place restrictions that minimize losses by leaching, runoff and volatilization they do not consider the effects of soil type or management practices on denitrification. Furthermore, applications of organic manures to fine-textured soils are encouraged as a measure to reduce NO<sub>3</sub><sup>-</sup> leaching, however, these soil types are more susceptible to denitrification of N in the presence of a labile organic matter source.
- Potentially mineralizable N values provided or implied through international fertilizer advice may underestimate the actual N value of biosolids in some cases. This may be explained because regional controls may not adequately account for local variations in wastewater and sludge treatment process operation, or the effects of soil environmental conditions and properties, that may influence the size of the mineralizable N pool or the extent of soil mineralization.
- Inconsistencies between fertilizer guidelines and actual PAN could result in oversupply and waste of supplementary mineral fertilizer N applications, representing an economic loss to the farmer and a potential impact on the environment from unused mineral N in the soil. This is particularly relevant to warm climates, where rates of mineralization may be rapid, and the extent of mineralization is potentially larger compared to cooler, temperate regions. Indeed, different PAN factors may apply to similar biosolids types across different climatic zones emphasizing the need for regional characterization of biosolids N availability to develop fertilizer recommendations that are appropriate and relevant to specific areas.
- Improving the efficiency of N utilization in biosolids-amended agricultural soils is therefore possible to deliver economic benefits, reduce mineral fertilizer inputs, maximize the resource recovery benefits, prevent environmental losses, minimize GHG emissions and contribute to global food security.

#### 9.2. Recommendations for further research

- The development of rapid chemical tests to determine potentially mineralizable N in biosolids would offer considerable savings and advantages and is therefore strongly recommended. This would improve the accuracy of routine N testing and fertilizer value advice available to increase the efficiency of recycling N resources in biosolids.
- It is important for researchers of biosolids N value to provide better descriptions of sludge properties and stability (e.g. VS content) and the nature of the wastewater treatment system that produced them to better understand the relationship between upstream treatment processes and N mineralization.
- The effects of upstream wastewater treatment processes, and the relative proportions of primary and activated sludge, on final treated biosolids stability, organic matter quality and N release in biosolids-amended soil are priority areas for further investigation of biosolids N value.

- Greater process understanding of N<sub>2</sub>O emissions from biosolidsamended soil is required to improve the accuracy of the default values used in C inventories to calculate potential denitrificationrelated N<sub>2</sub>O emissions (IPCC, 2006). This is necessary to ensure C footprint calculations are realistic, representative and properly inform decisions for optimal management of biosolids resources.
- Soil microbial community dynamics and mineralization-turnover require fundamental, process level investigation to determine the effects of soil temperature conditions on the overall extent of biosolids N mineralization in warm climates and to quantify the degradation rates of recalcitrant forms of biosolids organic N under these conditions compared to temperate soils.

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