

Formation of mineral N (NH_4^+ , NO_3^-) during mineralization of organic matter from coal refuse material and municipal sludge

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Summary – Zusammenfassung

In 1994, the 14 hectare plateau of a coal refuse bank in Landsweiler-Reden (Southwest-Germany) was covered with a mixture consisting of 80% (v/v) refuse materials, 10% (v/v) composted wood and 10% (v/v) of sewage sludge as part of a reclamation project. The amount of sludge dry matter applied was approximately 450 Mg ha⁻¹ to a depth of 2 m. The approximate amount of nitrogen (N) applied with the substrate was 20 Mg ha⁻¹ (total N). From April 1996 until November 1997, contents of mineral nitrogen and nitrogen mineralization were monitored down to a depth of 2 m. Nitrogen mineralization was monitored by means of a modified buried bag procedure using a retrievable cylindrical receptacle.

The contents of NH_4^+ - and NO_3^- -N were largest at the beginning of the observation period, reaching a peak value of 650 kg ha⁻¹ in May 1996. Then, mineral N stabilized in 1997 at a level of 200 kg N ha⁻¹, with the soil profile below 150 cm contributing about 75% to this amount. Net nitrogen mineralization was characterized by the same depth distribution. Other than in surface horizons, mineralization activity at the bottom of the profile continued into 1997 with the same intensity as in 1996. Variability among replicate buried bag incubations was high (CV > 100% on several occasions). Nitrogen loss through leaching was estimated at 630 kg N ha⁻¹ over the observation period, averaging at 360 kg N ha⁻¹ a⁻¹. The reclamation procedure used in this study may have the potential to contaminate ground water in hydrologically sensitive areas.

Key words: municipal sludge / coal refuse / ammonium / nitrate / mineralization

Stickstoffumsatz in einem Rekultivierungs-substrat aus Bergematerial, Holzkompost und Klärschlamm

1994 wurde das 14 Hektar große Plateau der Bergehalde Landsweiler-Reden im Zuge einer Rekultivierungsmaßnahme mit einer Mischung aus 80 Vol.-% Bergematerial, 10 Vol.-% Holzkompost und 10 Vol.-% Klärschlamm bedeckt. Dabei wurden etwa 450 Mg ha⁻¹ Klärschlamm bis in eine Tiefe von 2 m eingebaut. Die dabei ausgebrachte Gesamtstickstoffmenge betrug etwa 20 Mg ha⁻¹. In diesem Substrat wurden Mineralstickstoffgehalte und die Stickstoffmineralisation während des Zeitraumes von April 1996 bis November 1997 bis in eine Tiefe von 2 m beobachtet. Dies geschah mit Hilfe der „buried bag“-Methode unter Verwendung einer herausnehmbaren zylindrischen Brutkapsel.

Die Gehalte an NH_4^+ und NO_3^- waren zu Beginn der Beobachtungsphase am höchsten, der Spitzenwert betrug 650 kg N ha⁻¹ im Mai 1996. Der Mineralstickstoffgehalt stabilisierte sich während des Jahres 1997 bei 200 kg N ha⁻¹. Der Profildbereich tiefer als 150 cm hatte daran einen Anteil von 75%. Die Nettostickstoffmineralisation war durch einen gleichgerichteten Trend gekennzeichnet. Im Gegensatz zu den Oberflächenhorizonten war die Mineralisationsaktivität in den tiefen Bereichen des Profils im Jahr 1997 ebenso hoch wie im Jahr 1996. Die Variabilität zwischen den Einzelinkubaten war hoch (Variationskoeffizient an mehreren Terminen > 100%, n = 20 pro Tiefe). Stickstoffverluste mit dem Sickerwasser wurden für die gesamte Beobachtungsphase mit 630 kg N ha⁻¹ (entsprechend 360 kg ha⁻¹ a⁻¹) geschätzt. Das hier benutzte Rekultivierungsverfahren kann daher nicht zur Anwendung in hydrologisch sensiblen Bereichen empfohlen werden.

Introduction

Mining of all types has disturbed millions of hectares throughout the world. Disturbed sites are often characterized e.g. by dark coloured materials leading to high surface temperatures, low pH, low cation exchange capacity, low nutrient content and low water holding capacity (Sopper, 1993). In short, disturbed sites lack a functioning soil cover. The current methods used to revegetate steep coal waste banks involve topsoiling, i.e. transportation of large volumes of overburden material to the devastated site. A potential alternative seems to be the use of organic wastes

(i.e. sewage sludge) to substitute for soil organic matter, thereby creating an artificial or “new” soil. If successful, such a procedure would solve two problems at the same time: reclamation of devastated lands and sludge disposal.

However, the addition of organic waste can often increase nutrient loads as well as loads of trace metals, polychlorinated biphenyls and other toxic organic substances on the reclaimed site. The transfer of trace metals and other xenobiotics may be controlled by limiting their application rates. There is little danger of mobilization, if the chemical composition of the new substrate is adjusted properly in terms of pH and redox conditions. As to the potential contamination of the surrounding environment, nutrient loads have to be regarded as a more critical issue. Lack of

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plant-available N is a major problem in revegetation of spoil heaps created by deep coal mining in Germany (Berge, 1969). Sewage sludges contain 20 to 60 g N kg⁻¹ or more, much of which is in the organic form (Sommers et al., 1976). If organic N compounds are mineralized, soluble (NO₃⁻; NH₄⁺) and gaseous (N₂O) products may leave the soil profile and contaminate adjacent environmental compartments. Environmental problems related to such losses have been summarized by Keeney (1982). Public concern over these issues has caused improved management of organic wastes to receive a high priority of state and national regulatory agencies in Germany (Anonymous, 1996; 1998; Bannick, 1995).

The aim of this work was a) to attempt an assessment on the influence of substrate heterogeneity on the precision of mineralization measurements, b) to assess the influence of profile depth on the rates of ammonification and nitrification, c) to document the amount of N mineralized over a 18-month period, d) to correlate mineralization intensity with climate parameters and finally to e) allow an assessment of the amount of mineral N, that may be leached from the substrate.

Materials and methods

Site description

The deep coal mining refuse bank "Landsweiler-Reden" is located 5 km west to the city of Neunkirchen in Southwest Germany (Saarland). The refuse bank has steep, conical-shaped side slopes, covers a total area of 56 ha and rises to an elevation of 80 m above the surrounding terrain, where it levels off to a plateau of 14 ha. The refuse bank was abandoned in 1996, with reclamation activities on the plateau starting in 1994. The plateau of the refuse bank had been subsequently covered with a mixture consisting of 80% (v/v) refuse materials, 10% (v/v) composted wood and 10% (v/v) of sewage sludge. The amount of sludge dry matter applied was approximately 450 Mg ha⁻¹. The bank consists of carboniferous sedimentary rocks of widely different nature. Conglomerates, sandstones and dark shales as well as remnants of rock salt are mixed with 5–10% of anthracitic coal, which was not removed in the process of separating coal from accessory rock. Pyrite and markasite are present at about 0.35% of weight.

Substrate application and properties

Beginning in April 1994, 320 000 m³ of refuse materials, 35 000 m³ of municipal sewage sludge and 32 000 m³ of composted wood were mixed to an approximate volume of 8:1:1. The mixing process consisted of the materials being added to a conveyor belt system, which included several overthrow stations on its way to the plateau of the refuse bank. Mixing quality was assessed by visual inspection of the material as it arrived at its place of destination. The substrate was then applied to the plateau to a thickness of 2 m. Substrate properties are given in Tab. 1.

Observation site

The observations reported here started in April 1996 in that part of the plateau, which had been covered in the early phase of the project and was at that time two years old. Vegetation was dominated by several varieties of *Chenopodium*, *Bromus inermis* and *Cirsium vulgare*. Maximum rooting depth visually observed was 1.20 m. Numerous earthworms (*Eisenia foetida*) and earthworm casts were observed down to the bottom of the substrate (2 m). The observation site consisted of an open soil pit for sampling purposes, a weather station and associated suction lysimeters, tensiometers and buried bag receptacles.

Measurement of nitrogen mineralization/immobilization

Here the method initially suggested by Eno (1960) was employed, which involves a soil sample being enclosed in a plastic bag, that allows gases to permeate and prevents water from doing so. The amount of mineral nitrogen is measured at the begin and the end of the incubation period, and rates of net mineralization are derived by dividing the change in nitrogen concentration in soil samples by the duration of the incubation period. Polyethylene freezer bags (Haaf, Lohhof, Germany) were used as containers for the soil. The bags have a average standard thickness of 50 µm. Polyethylene film is impermeable to water, while it does not impede oxygen and carbon dioxide transmission (Gordon et al., 1987). After subsampling for inorganic N determination (2 subsamples per location), soil samples weighing about 100 g each and enclosed in PE-bags were incubated for 2 weeks (incubation time as suggested by Adams et al., 1989) in the same soil depth they had been taken from. The substrate was generally incubated as taken from the pit wall. There was no sieving. Gravel bigger than approximately 5 cm, earthworms and visible root fragments were sorted out prior to the enclosure of the sample. For inorganic N determination a 30 g subsample was taken for extraction with 500 ml of 0.0125 M CaCl₂. Inorganic N in the extract was determined according to Schlichting et al. (1995, p. 129–133).

Table 1: Selected properties of recultivation substrate at Landsweiler-Reden, Southwest-Germany.

Tabelle 1: Eigenschaften des Rekultivierungssubstrates von Landsweiler-Reden, Südwestdeutschland.

| horizon ⁺ | lower depth | rock | | CEC | pH | | C _t | C _{carbonate} | C _{coal} | C _{org} | N _t | total pore | |
|----------------------|-------------|-----------|--------------------|------------------------------------|----------------------|----------------|----------------|------------------------|-------------------|------------------|----------------|------------|----------|
| | | fragments | BD | | (CaCl ₂) | C _t | | | | | | space | pyrite-S |
| | cm | % (v/v) | Mg m ⁻³ | cmol _c kg ⁻¹ | | % | % | % | % | % | % (v/v) | % | |
| Ah1 | 10 | 59 | 1.4 | 18.2 | 7.0 | 15.6 | 0.1 | 9.4 | 6.1 | 0.60 | 30 | 0.10 | |
| Ah2 | 30 | 67 | 1.4 | 28.6 | 7.4 | 17.1 | 0.3 | 8.5 | 8.3 | 0.75 | 21 | 0.14 | |
| Ah+yjCn | 50 | 52 | 1.5 | 19.0 | 7.5 | 17.4 | 0.2 | 8.2 | 9.0 | 0.86 | 38 | 0.17 | |
| yjCn1 | 110 | 51 | 1.7 | 9.4 | 7.6 | 16.2 | 0.1 | 10.8 | 5.3 | 0.62 | 38 | 0.17 | |
| yjCn2 | 160 | 58 | 1.5 | 13.4 | 7.6 | 16.9 | 0.1 | 9.8 | 7.0 | 0.65 | 36 | 0.17 | |
| yjCn3 | 190 | n.d. | 1.6 | 10.4 | 7.6 | 12.9 | 0.1 | 8.9 | 3.9 | 0.49 | n.d. | 0.11 | |

BD = bulk density; C_{carbonate} = CaCO₃-Carbon; CEC = cation exchange capacity; C_t = total carbon; N_t = total nitrogen; C_{coal} = anthracitic carbon (Wu et al. 1996);

C_{org} = Carbon in wood, sludge and humified plant detritus; n.d. = not determined. Properties determined according to Schlichting et al. (1995);

C_t - C_{coal} - C_{carbonate} = C_{org}.

⁺ Horizon designators according to AG Boden (1994)

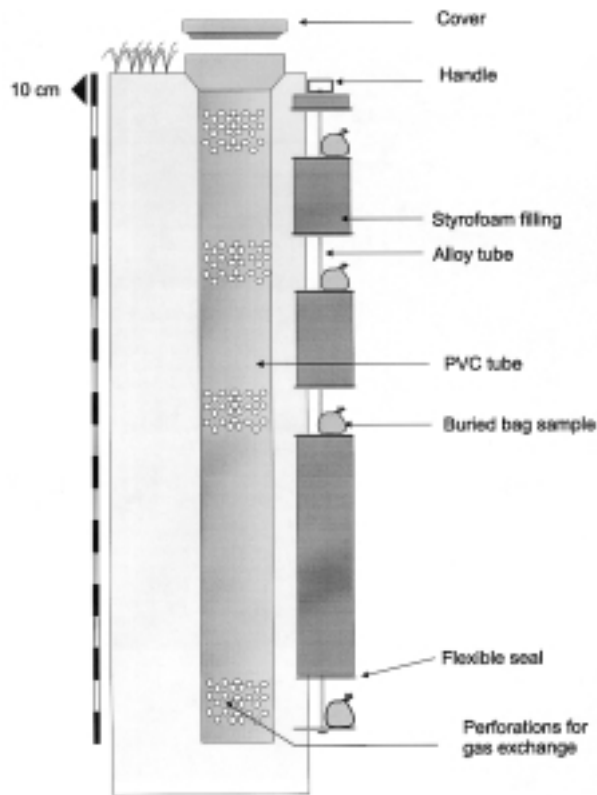


Figure 1: Receptacle for buried bag incubations.
Abbildung 1: Schemazeichnung eines Brutbehälters.

Nitrogen contents on a kg ha^{-1} basis were calculated by multiplying the concentrations in 10–30 cm, 50–70 cm, 90–110 cm and 180–200 cm with bulk densities for the profile regions 0–30 cm, 30–80 cm, 80–150 cm and 150–200 cm, respectively. To be able to incubate soil samples at the appropriate depth, a modified cylindrical receptacle as suggested by Schlichting et al. (1995; page 212) was inserted into the soil (Fig. 1).

The receptacle consists of an outer PVC-tube with aeration holes at each individual incubation depth. A movable inner mounting supports four replicate buried bag samples per sampling depth. Styrofoam disks provide vertical separation of samples together with thermal insulation, in order to ensure exposure to ambient soil temperature. To account for suspected variability introduced by the heterogeneity of the substrate, it was decided to use 5 buried bag receptacles, each capable to store 4 replicate bags per depth. This amounted to 20 buried bags in total per incubation period and depth, which was thought to be a sample population big enough to draw inference from.

The wall of the soil pit was protected by several layers of styrofoam plates, bags filled with Styrofoam chips and plastic covers. On sampling occasions, 20 cm of soil was taken off the profile wall, discarded, and samples then taken from the newly exposed surface. To determine the importance of horizontal substrate heterogeneity on net nitrogen mineralization, the profile wall was subdivided in five vertical sampling zones, which will be referred to as locations 1–5. Materials from individual locations were always incubated in the same receptacle. Spacing between receptacles was 1 m, slightly bigger than the spacing between sampling locations.

Nitrogen leaching

A simple climatic water budget approach (Renger et al., 1974) was employed to estimate nitrogen losses from the substrate. NO_3^- and NH_4^+

concentrations in suction lysimeters at a depth of 2 m were then multiplied with the amount of percolate water:

$$\text{Loss} = \frac{\text{DW} \cdot \text{Conc}}{100} \quad (\text{Eq. 1})$$

where Loss (kg ha^{-1}) = amount of nitrogen lost in a given time interval; DW = water drained within time interval (L m^{-2}); and Conc (mg L^{-1}) = nitrogen concentration in suction lysimeter at depth 200 cm.

Data analysis

To calculate the contribution of depth, location and date to the variability of NO_3^- and NH_4^+ content and its change rates a multivariate analysis of variance (MANOVA) was carried out. The contribution of each factor was calculated as a percentual relation between the factor variance (sum of squares according to the factor) to the whole variance (total sum of squares). The interactions between the factors were added to the error variance.

The Tukey honest significance difference (HSD) with the level for critical ranges set at 0.05 was calculated to estimate the significance of soil depth on change rates (net mineralization) of NO_3^- and NH_4^+ .

Multiple forward stepwise linear regression was calculated to estimate the dependence of mineral NO_3^- and NH_4^+ content as well as its change rates (net mineralization) from the soil water content and soil temperature. If a F-value of 1 was exceeded, the influence of the factor was considered to be significant and it was included into the model.

Results

Mineral N in the soil

Two years after substrate application, there was no reason to believe that any soluble, mineral N originally associated with the sewage sludge should still be present. So all mineral N observed at that time was assumed to be a product of mineralization processes. Davies et al. (1995) reported rapid losses of nitrogen after restoring of topsoil, which had been stored in a stockpile for 12 years. In that study $2100 \text{ kg N ha}^{-1}$ of a total of $2450 \text{ kg N ha}^{-1}$ were lost in the first 5 months of a 2 year observation period. Therefore it was of particular interest, whether there would still be recognizable N dynamics in the soil developed on the coal refuse bank at Reden at a similar time after application. Analysis of mineral N throughout the observation period showed that N-dynamics at a depth of 190 cm were almost contrary to those at upper profile regions. Low NH_4^+ -contents were present down to 100 cm depth throughout the observation period, while NO_3^- contents declined from a moderate peak in spring 1996 to values near zero at the end of 1997 (Fig. 2). In contrast extraordinary high values of NH_4^+ were observed at 190 cm, while NO_3^- was low with occasional peaks.

Expression on a kg ha^{-1} basis facilitates a comparison of the mineral N content observed with the values ordinarily prevailing in native or agricultural soil, and with the amount necessary to sustain a plant cover. Fig. 3 shows levels of mineral N for the whole soil profile, which in late spring 1996 were well beyond any plant requirement (peak value at 650 kg N ha^{-1}). After this maximum, mineral N content

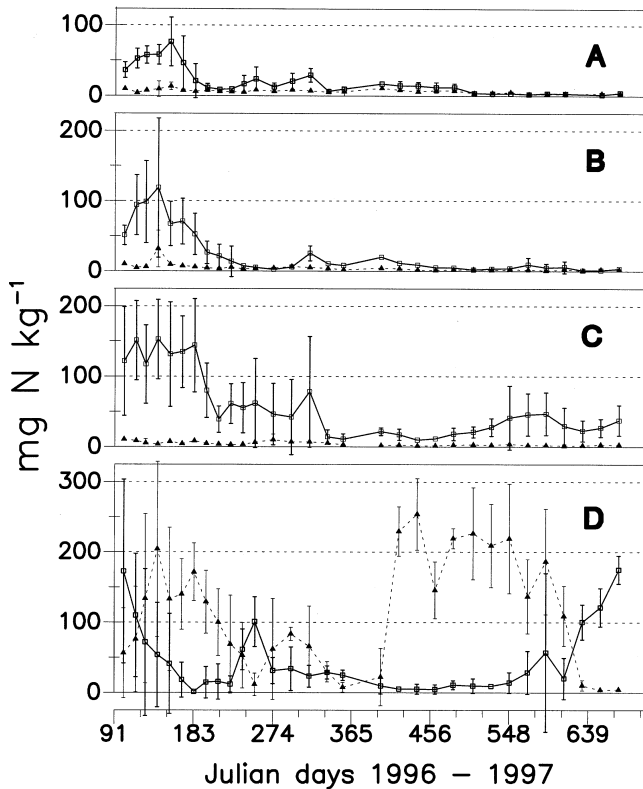


Figure 2: Content of ammonia (▲) and nitrate N (□) in separate soil horizons versus observation time. A = 10–30 cm; B = 50–70 cm; C = 90–110 cm; D = 180–200 cm. Error bars give Standard Deviation among the $n = 5$ receptacle mean values per depth.

Abbildung 2: Zeitlicher Verlauf von Ammonium- (▲) und Nitratstickstoff (□) in den einzelnen Inkubationstiefen. A = 10–30 cm; B = 50–70 cm; C = 90–110 cm; D = 180–200 cm. Fehlerbalken bezeichnen die Standardabweichung zwischen den Tiefenmittelwerten der $n = 5$ Brutgefäße.

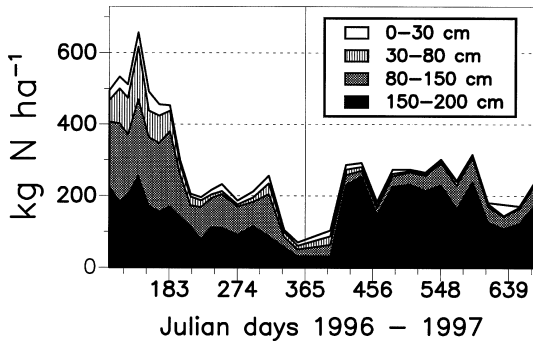


Figure 3: Content of mineral N in whole soil profile versus observation time, expressed on a kg N ha^{-1} basis.

Abbildung 3: Zeitlicher Verlauf der Mineralstickstoffgehalte bezogen auf das Gesamtprofil in kg N ha^{-1} .

declined to a moderate ($\approx 50 \text{ kg N ha}^{-1}$) level for the upper 150 cm of the soil profile. In the subsoil, where no plant roots were observed, a constant level of around 200 kg N ha^{-1} persisted throughout the whole year 1997.

Fig. 4 further indicates a symmetrical behaviour of NH_4^+ and NO_3^- at that depth, with NH_4^+ being high when NO_3^- was low and *vice versa*. Variability within depths ($n = 5$ locations) was rather high, as indicated by the large standard deviations shown in Fig. 2.

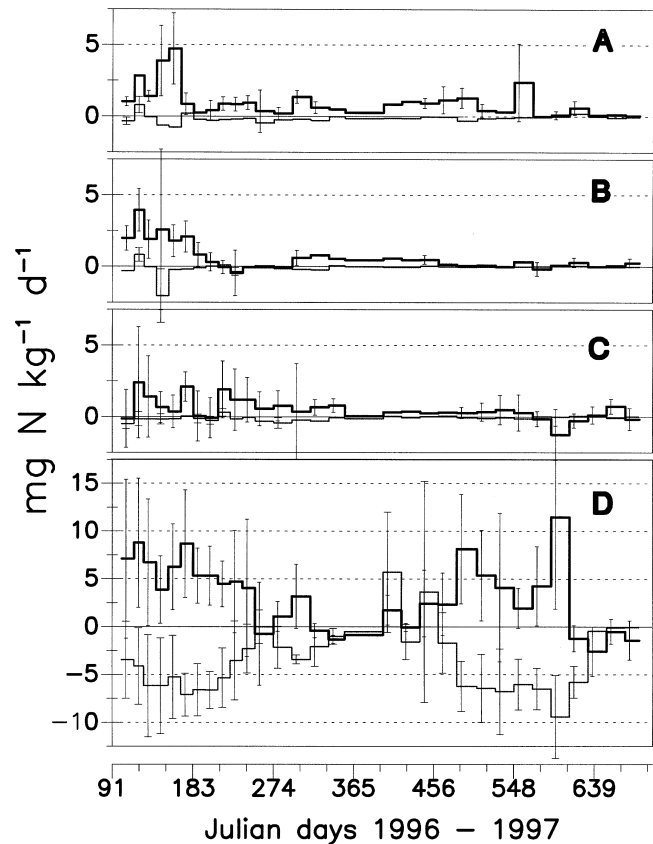


Figure 4: Net mineralization of ammonia (thin line) and nitrate N (bold printed line) versus time and depth. A = 10–30 cm; B = 50–70 cm; C = 90–110 cm; D = 180–200 cm. Error bars give Standard Deviation among the $n = 5$ receptacle mean values per depth.

Abbildung 4: Nettomineralisation von Ammonium-N (dünngedruckte Linie) und Nitrat-N (fettgedruckte Linie) pro Beobachtungszeitraum und Beobachtungstiefe. A = 10–30 cm; B = 50–70 cm; C = 90–110 cm; D = 180–200 cm. Fehlerbalken bezeichnen die Standardabweichung zwischen den Tiefenmittelwerten der $n = 5$ Brutgefäße. Negative Werte kennzeichnen Immobilisierung, positive Werte Mineralisierung.

Net nitrogen mineralization

Mineralization rates behaved differently in the upper (0–150 cm) and in the lower (< 150 cm) part of the profile (Fig. 5). In the upper portion, moderate mineralization activity compared to natural soils (Stahr et al., 1994) was observed in early summer 1996, to decline to near zero values for both NH_4^+ and NO_3^- in the later part of the observation period. Seasonal change in rates of mineralization was noted in the lower portion of the soil profile. The curves of NH_4^+ and NO_3^- exhibit a near perfect symmetry: negative change rates of NH_4^+ correspond with positive change rates for NO_3^- in time as well as in magnitude.

Mineralization versus depth of incorporation

It was of special interest, to determine whether differences in mineralization intensity would occur at different profile depths, and at which depth such mineralization intensity would be highest. For this reason, a Tukey-HSD test was performed including the data of the whole observation period.

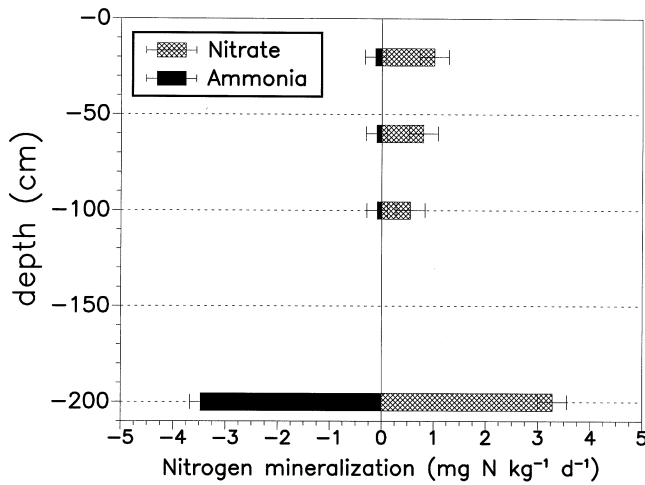


Figure 5: Mean mineralization of ammonia and nitrate versus depth of incorporation. Negative values indicate immobilization, positive values mineralization. Data represent the mean of the entire observation period.
Abbildung 5: Mittlere Ammonium- und Nitratmineralisation bezogen auf die Bebrütungstiefe. Negative Werte kennzeichnen Immobilisierung, positive Mineralisierung. Fehlerbalken = Vertrauensbereich für den Mittelwert ($\alpha = 0.05$). Darstellung bezogen auf den gesamten Beobachtungszeitraum.

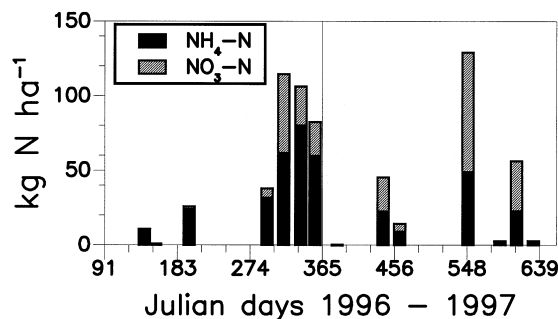


Figure 6: Estimates (Renger et al., 1974) of nitrogen leached from the substrate during observation period.
Abbildung 6: Geschätzte Stickstoffverluste mit dem Perkolationswasser während des Beobachtungszeitraumes (Renger et al., 1974).

There was no significant difference for NH_4^+ and NO_3^- transformations between the first three observation depths (Fig. 6). For NH_4^+ , there was hardly any activity at all, while NO_3^- was being produced regularly at moderate intensity. Although not significant, a tendency of decreasing NO_3^- mineralization with depth was visible down to a depth of 100 cm. At the profile base however, significantly higher negative change rates for NH_4^+ and higher positive change rates for NO_3^- were observed.

Variability of mineralization measurements

With an artificial substrate of heterogeneous nature, a high variability of N budget parameters was to be expected. The degree of variability encountered, however, was much higher than previously assumed. Even with 20 replicate buried bags per depth, coefficients of variation exceeded 100% on a regular basis. As an example, NH_4^+ and NO_3^- mineralization rates in August 1996 and at a depth of 100

cm are given in Tab. 2. It was therefore suspected, that spatial variability might exert an influence on mineralization rates.

This was tested by means of a multivariate analysis of variance (MANOVA, Tab. 3) including the factors depth, location and time. The influence of location on mineralization and N contents was significant but negligible.

Influence of soil temperature and soil moisture on N-budget parameters

To determine the influence of temperature and moisture on the N-budget, a forward stepwise multiple linear regression was calculated for individual depths and the whole soil profile (denoted "combined" in Tab. 4). It turned out to be impossible to explain more than 12.7% of mineralization rate variability by means of soil temperature and soil water content. Coefficients of determination were slightly higher for mineral N content, but remained below 22% with the single exception of the NO_3^- content at a depth of 60 cm. There, 61.4% of the variability could be attributed to the influence of moisture and temperature (Tab. 4).

Estimation of mineral N losses through leaching

A simple climatic water budget was calculated to estimate the amount of mineral N lost from the profile with percolate water. Leaching occurred during the last three months of 1996, and on single occasions in 1997 (Fig. 6). NH_4^+ constituted a significant part of the total amount of N lost through percolation. An estimated total of 375 kg ha⁻¹ of NH_4^+ -N and 254 kg ha⁻¹ of NO_3^- -N left the substrate throughout the 21-month observation period, to render an average annual loss estimate of 360 kg ha⁻¹ of mineral N (NH_4^+ -N + NO_3^- -N).

Discussion

Parameters affecting N mineralization

N mineralization is known as a parameter of rather variable nature. In the work of Kleber (1997), the coefficient of variation among replicate buried bag incubations was higher than 10% in more than half of the incubation periods, and higher than 50% in a quarter of the incubation periods. Springob and Mohnke (1995) found 10 replicates necessary to estimate net N mineralization for a 100 square meter plot on sandy soils. Raison et al. (1987) and Deboz and Vinther (1989) used up to 15 replicate soil cores, while Goncalves and Carlyle (1994) needed 8 replicates. Stenger (1996) found coefficients of variation between 20% and 50% with eight replicate incubations. In the incubation period and incubation depth presented in Tab. 3, which was chosen arbitrarily, the coefficient of variation (n = 20) encountered in the reclamation substrate was the same for NH_4^+ and for NO_3^- with 110% and 112%, respectively. Thus, the influence of substrate heterogeneity

Table 2: Variability of 20 replicate measurements of net nitrogen mineralization versus variability among 5 receptacles. Data from August 1996 and incubation depth of 100 cm. Roman numerals identify receptacles, arabian numerals identify replicate buried bag samples. Negative values indicate immobilization. \bar{x} = mean value, SD = standard deviation.

Table 2: Variabilität der Stickstoffnetto-mineralisation innerhalb der 20 Wiederholungen einer Inkubationstiefe verglichen mit der Variabilität zwischen den 5 Einzelbrutgefäßen. Werte vom August 1996 und aus einer Bodentiefe von 100 cm. Die römischen Zahlen stehen für das jeweilige Brutgefäß, die arabischen kennzeichnen die einzelnen Brutbeutel. Negative Zahlen kennzeichnen eine Immobilisation, \bar{x} = Mittelwert, SD = Standardabweichung.

| Nitrogen as | receptacle (location) Nr. | replicate bag Nr. | | | | \bar{x} (SD) |
|--------------------|---------------------------------------|---------------------------------------|-------|-------|-------|----------------|
| | | 1 | 2 | 3 | 4 | |
| | | mg N kg ⁻¹ d ⁻¹ | | | | |
| NO ₃ -N | I | 0.07 | 0.57 | -1.99 | -0.94 | -0.57 (1.14) |
| | II | 2.20 | 1.26 | 3.05 | 2.25 | 2.19 (0.73) |
| | III | 4.46 | 7.42 | 2.93 | 2.72 | 4.38 (2.17) |
| | IV | -0.04 | 5.77 | 2.87 | 3.90 | 3.13 (2.43) |
| | V | 0.34 | 1.29 | 2.31 | -1.88 | 0.52 (1.79) |
| | Variability among receptacles (n = 5) | | | | | 1.93 (1.98) |
| | Variability total (n = 20) | | | | | 1.93 (2.17) |
| NH ₄ -N | I | 0.13 | 0.00 | 0.06 | 0.21 | 0.10 (0.09) |
| | II | 0.56 | 0.20 | 1.25 | 0.41 | 0.61 (0.45) |
| | III | 0.77 | 0.10 | 0.53 | 0.25 | 0.57 (0.23) |
| | IV | -0.02 | 0.09 | -0.05 | 0.36 | 0.10 (0.19) |
| | V | 0.23 | -0.09 | 0.63 | 0.04 | 0.20 (0.31) |
| | Variability among receptacles (n = 5) | | | | | 0.31 (0.25) |
| | Variability total (n = 20) | | | | | 0.31 (0.35) |

seems to be dominating over the heterogeneities in N transformation processes.

The number of replicate buried bags required to estimate mineralization within 10% of its mean value ($\alpha = 0.05$, *Snedecor and Cochran, 1967*) would have been 519 (NO₃⁻) and 559 (NH₄⁺) in the example (Tab. 2) chosen. This variability must be considered as the most probable reason for the impossibility to correlate any one of the factors soil temperature, soil moisture, incubation depth, time of the year and receptacle location with N mineralization.

Suitability of deep incorporation

Mineral N content was high and net N mineralization intense at the beginning of the observation period, which itself started almost two years after the application of the substrate. As mineralization intensity and mineral N content declined to remain at near zero levels in soil compartments above 150 cm, it seems proper to assume that the mineralization of added organic matter terminated at the end of 1996, about two and a half years after substrate application. It may be inferred from this observation, that N dynamics proceeded at much higher levels in the period immediately after application. At below 150 cm, however, N turnover continued with high intensity. NH₄⁺ was consumed at rates symmetrical to rates of NO₃⁻ production. NO₃⁻ levels remained low despite high production rates, indicating that leaching or – less probable at that profile depth – volatilization occurred. This is supported by the estimation of N-losses from the soil profile by means of a

Table 3: Percentage of variability in nitrogen content and nitrogen mineralization as explained by time, depth and location. Contribution of each factor given as percentual relation between the factor variance (sum of squares according to the factor) to the whole variance (total sum of squares). Read: Over the whole observation period, 30.1% of the variability of NO₃ content were explained by the time of observation, 9.7% by incubation depth and 1.5% by receptacle location (= pit wall segment).

Table 3: Prozentanteile der Variabilitäten von Stickstoffgehalt und Stickstoffmineralisation, die durch Zeit, Tiefe und Zuordnung zu einem bestimmten Brutgefäß erklärt werden können. Der Anteil des jeweiligen Faktors ist angegeben als Prozentanteil der Varianz des Faktors bezogen auf die Gesamtvarianz. Lesebeispiel: Über den gesamten Beobachtungszeitraum gesehen konnten 30.1% der Variabilität des NO₃-Gehaltes durch den Beobachtungszeitpunkt, 9.7 durch die Bebrütungstiefe und 1.5 durch Zuordnung zu einem bestimmten Brutgefäß erklärt werden.

| | | Time | Depth | Location |
|---------------------|-----------------|------|-------|----------|
| | | % | | |
| Nitrogen content | NO ₃ | 30.1 | 9.7 | 1.5 |
| | NH ₄ | 8.6 | 49.8 | 0.3 |
| Mineralization rate | NO ₃ | 9.7 | 7.4 | 1.0 |
| | NH ₄ | 7.8 | 22.3 | 0.3 |

simple water-budget approach. Calculations of N-losses also indicated that large amounts of NH₄⁺ left the profile, which would not have been the case in soils with sufficient cation exchange capacity. Low clay contents in the substrate and high NH₄⁺ concentrations in the soil solution at a depth of 2 m indicate, that the substrate was unable to

Table 4: Dependence of mineral NO_3^- and NH_4^+ content and their respective change rates on soil water content and soil temperature as determined by multiple forward stepwise linear regression. Factors were considered to be significant and included in the model, once a F-value of 1 was exceeded. Combined = all depths together, R^2 = Coefficient of determination.

Tabelle 4: Abhängigkeit der Mineralstickstoffgehalte (NO_3^- und NH_4^+) und der zugehörigen Veränderungsrate von Bodenwassergehalt und Bodentemperatur (Multiple lineare Regression). Die Faktoren wurden als signifikant angesehen und im Modell berücksichtigt, wenn der jeweilige F-Test einen Wert von > 1 ergab. In der Zeile „combined“ sind alle Tiefen zusammengefasst, R^2 = Bestimmtheitsmaß.

| Variable | depth | constant | water content | soil temperature | R^2 |
|--|----------|----------|---------------|------------------|-------|
| | cm | | % (v/v) | °C | |
| NO_3^- -content | combined | | 1.08 | | 16.1 |
| | 20 | 7.76 | 0.61 | | 13.8 |
| | 60 | -56.7 | 1.62 | 0.0068 | 61.4 |
| | 100 | | 1.48 | | 18.9 |
| | 200 | | | | |
| NH_4^+ -content | combined | | 0.779 | | 4.2 |
| | 20 | 3.97 | 0.101 | | 12.9 |
| | 60 | | 0.167 | | 13.0 |
| | 100 | | 0.092 | | 22.2 |
| | 200 | | | | |
| $\text{NO}_3^- + \text{NH}_4^+$ -cont. | combined | 14.3 | 1.6 | | 21.6 |
| | 20 | 11.7 | 0.71 | | 15.8 |
| | 60 | -61.6 | 1.80 | 0.0077 | 56.6 |
| | 100 | 19.0 | 1.26 | | 20.5 |
| | 200 | 222.5 | | -0.0111 | 5.7 |
| NO_3^- -rate | combined | 3.65 | 0.018 | -0.00044 | 4.4 |
| | 20 | | 0.038 | 0.00006 | 10.7 |
| | 60 | | 0.039 | | 3.5 |
| | 100 | | 0.015 | | 3.5 |
| | 200 | 10.9 | | -0.0012 | 12.7 |
| NH_4^+ -rate | combined | -2.25 | | 0.0002 | 1.1 |
| | 20 | 0.29 | -0.005 | -0.00005 | 2.4 |
| | 60 | 0.62 | -0.008 | -0.00009 | 6.2 |
| | 100 | -0.23 | | 0.00002 | 1.3 |
| | 200 | -7.55 | 0.063 | | 4.1 |
| $\text{NO}_3^- + \text{NH}_4^+$ -rate | combined | 1.82 | 0.012 | -0.00027 | 2.1 |
| | 20 | | 0.041 | | 7.7 |
| | 60 | | 0.034 | | 2.5 |
| | 100 | | 0.014 | | 3.0 |
| | 200 | 2.93 | 0.041 | -0.00093 | 9.4 |

fix the surplus NH_4^+ produced by organic matter mineralization.

Conclusions

Two years after application and deep incorporation of large amounts of sewage sludge, contents of mineral and thus plant available N are very high and exceed the needs of the established vegetation. Results render it very probable, that N mineralization from the decomposition of sludge organic matter will arrive at a peak value following application, and consecutively declines rather rapid with the consumption of readily decomposable organic matter.

Losses through percolation and/or volatilization seem inevitable within this period, whose length depends on substrate, climate and the depth of incorporation.

Since no monitoring of mineral N was done below the mixed substrate it is difficult to assess the potential of the system to pollute groundwater. In addition, NH_4^+ was found at the deepest depth suggesting water stagnation may have played a significant role in remediating potential NO_3^- movement past the introduced substrate. While results are not completely conclusive, they still support the view of other authors (King, 1973; Johnson and Williamson, 1994), that municipal sewage sludge incorporated to greater depths may have a long term potential to contaminate ground water.

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