

# Differing N status and N retention processes of soils under old-growth lowland forest in Eastern Amazonia, Caxiuanã, Brazil

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## Abstract

Indirect evidence of the nitrogen (N) status of tropical forests strongly suggests that in heavily weathered soils under old-growth lowland tropical forests nitrogen is in relative excess. However, within the lowland forests of the Amazon basin, there is substantial evidence that soil texture influences soil  $\text{NH}_4^+$  and  $\text{NO}_3^-$  concentrations and hence possibly N availability and retention in the soil. Here, we evaluate the soil N status of two heavily weathered soils which contrast in texture (sandy versus clay Oxisol). Using  $^{15}\text{N}$  pool dilution, we quantified gross rates of soil N cycling and retention. We also measured the  $\delta^{15}\text{N}$  signatures from the litter layer down to 50-cm depth mineral soil and calculated the overall  $^{15}\text{N}$  enrichment factor ( $\epsilon$ ) for each soil type. The clay soil showed high gross N mineralization and nitrification rates and a high overall  $^{15}\text{N}$  enrichment factor, signifying high N losses. The sandy soil had low gross rates of N cycling and  $^{15}\text{N}$  enrichment factor, manifesting a conservative soil N cycling. Faster turnover rates of  $\text{NH}_4^+$  compared to  $\text{NO}_3^-$  indicated that  $\text{NH}_4^+$  cycles faster through microorganisms than  $\text{NO}_3^-$ , possibly contributing to better retention of  $\text{NH}_4^+$  than  $\text{NO}_3^-$ . However this was opposite to abiotic retention processes, which showed higher conversion of  $\text{NO}_3^-$  to the organic N pool than  $\text{NH}_4^+$ . Our combined results suggest that clay Oxisol in Amazonian forest have higher N availability than sandy Oxisol, which will have important consequences for changes in soil N cycling and losses when projected increase in anthropogenic N deposition will occur.

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## 1. Introduction

High nitrogen (N) deposition is an environmental problem recognized until recently mainly in the economically developed regions of Europe and North America (Matson et al., 1999). Projections are that rates of N deposition in the tropics will increase by several hundred percent due to demands for food and energy by a growing population with increasing per capita use of N (Galloway et al., 2002). Latin America has at present both high and low inputs of reactive N, and a rapid increase of N deposition is predicted for this region (Galloway et al., 2004) as a consequence of continued increase in fertilizer use, fossil fuel consumption (Matson et al., 1999),

and biomass burning (Cochrane, 2003; Fabian et al., 2005).

A study in a montane forest in Hawaii showed that N additions resulted in large and immediate losses of  $\text{N}_2\text{O}$  and NO from N-rich forest soil compared to small and delayed increases of N-oxide emissions from N-limited forest soil (Hall and Matson, 2003). From the studies in temperate forests, Aber et al. (1998) summarized the results into a conceptual model which showed that the rate at which a forest ecosystem reacts to elevated N input is regulated by two main factors: the rate of N input and the inherent N status of the system (the latter being determined by soil type, forest type, and land-use history). These studies made clear that the soil N status is the key to analyze effects of increasing anthropogenic N deposition on forest ecosystem processes. At present, our knowledge on the N status of tropical forests is based on indirect

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evidence, e.g., N contents in leaves and litter (Vitousek, 1984), extractable inorganic N, net rates of soil N cycling and related N oxide emissions (Davidson et al., 2000), and  $^{15}\text{N}$  isotope signatures in leaves and soils (Martinelli et al., 1999). While net rates of soil N cycling provide information on N availability for plants, they do not reveal the soil N retention processes which are important indicators of how a forest ecosystem reacts to changes in N input. An alternative way to assess the soil N status of tropical forest is by quantifying the gross rates of mineral N production (mineralization and nitrification) and comparing them against N retention rates. So far there are only few studies in tropical forest soils which quantified gross rates of soil N cycling, revealing rates of mineral N production and consumption (i.e. Puerto Rico, Silver et al., 2001; Hawaii, Hall and Matson, 2003; Costa Rica, Silver et al., 2005; Indonesia, Corre et al., 2006; and only one study in the Brazilian Amazon (Rondonia), Neill et al., 1999).

From indirect evidence of the N status of tropical forests, lowland forests have higher litterfall N contents, lower dry mass/N ratios (Vitousek, 1984), higher net mineralization rates and N trace gas emissions (Davidson et al., 2000), and higher  $\delta^{15}\text{N}$  signatures in leaves and soils (Martinelli et al., 1999) than montane forests, supporting the speculation that N in lowland forests is in relative excess. However, within the lowland forests of Amazon basin, there is substantial evidence that soil texture influences soil  $\text{NH}_4^+$  and  $\text{NO}_3^-$  concentrations, water and other nutrient ion availability, decomposition, soil C retention and net primary production, particularly in highly weathered soils (Silver et al., 2000; Luizão et al., 2004). For example, Silver et al. (2000) reported that Amazonian lowland forests on clay soils have higher  $\text{NO}_3^-$  and lower  $\text{NH}_4^+$  concentrations than those on sandy soils. Sites where  $\text{NO}_3^-$  dominates over  $\text{NH}_4^+$  have been characterized as having a relative excess of N or 'leaky' N cycle (i.e. high N losses through  $\text{NO}_3^-$  leaching or N trace gases emissions), while sites where  $\text{NH}_4^+$  dominates over  $\text{NO}_3^-$  have been interpreted as N-limited or having a 'conservative' N cycle (Davidson et al., 2000). We studied two highly weathered soils (Oxisol) in an eastern Amazon forest with contrasting textures (sandy and clay). We hypothesized that the sandy Oxisol has a conservative N cycle with low rates of N cycling and N loss, while the clay Oxisol depicts a leaky N cycle—rates of soil N cycling are lower than rates of N loss. Our objectives were: (1) to assess the soil N status of sandy and clay lowland forest soils by quantifying gross rates of N mineralization and nitrification, and (2) to evaluate their differences in soil N-retention processes by measuring the microbial N immobilization, dissimilatory  $\text{NO}_3^-$  reduction to  $\text{NH}_4^+$ , and abiotic N immobilization. Direct quantification of the inherent soil N status of tropical forests and processes of N retention will provide much-needed baseline information for tropical forests, which may hint how such ecosystems will react to predicted increase in N deposition.

## 2. Materials and methods

### 2.1. Site description

The experimental site was located in Caxiuanã National Forest, Pará, Brazil ( $1^\circ 43' 3.5''\text{S}$ ,  $51^\circ 27' 36''\text{W}$ ). The forest is an old-growth lowland *terra firme* rainforest and the most common species are: *Dinizia excelsa*, *Marmaroxylon racemosum*, *Couratari guianensis*, *Buchenavia grandis*, *Swartzia racemosa*, and *Dipteryx odorata*. Mean annual air temperature is  $25.7^\circ\text{C}$ . Mean annual rainfall is 2272 mm, with a pronounced dry season between July and December, when on average only 555 mm of rainfall occurs (Fisher et al., 2006). The plateau of the Caxiuanã region belongs to Alter do Chão Formation (Costa and Kern, 1999), which contains sedimentary rocks with texture varying from sand to clay. Soils in Caxiuanã are predominantly Oxisols (Brazilian classification Yellow Latosol; Kern, 1996). Our study was conducted on two Oxisols with contrasting texture: clay and sand (Table 1).

Table 1  
Main characteristics of the forest sites (mean  $\pm$  SE;  $n = 3$ ) in Caxiuanã, Brazil

	Sand		Clay	
	Mean	SE	Mean	SE
<b>Leaves<sup>a</sup></b>				
C ( $\text{mg C g}^{-1}$ )	47.0	ND	46.2	ND
N ( $\text{mg N g}^{-1}$ )	1.6	ND	1.4	ND
C/N ratio	29.2	ND	33.9	ND
$\delta^{15}\text{N}$ (‰)	4.3	ND	6.7	ND
<b>Litter</b>				
C ( $\text{mg C g}^{-1}$ )	46.8	1.7	47.5	1.0
N ( $\text{mg N g}^{-1}$ )	1.2	0.0	1.6	0.1
C/N ratio	38.2	1.5	30.3	1.2
$\delta^{15}\text{N}$ (‰)	5.0	0.5	6.4	0.4
<b>Decomposing litter</b>				
C ( $\text{mg C g}^{-1}$ )	44.6	0.7	41.8	1.8
N ( $\text{mg N g}^{-1}$ )	1.4	0.1	1.4	0.1
C/N ratio	30.9	4.9	29.6	2.4
$\delta^{15}\text{N}$ (‰)	5.5	0.2	6.8	0.5
<b>Mineral soil (0–5 cm depth)</b>				
Bulk density ( $\text{Mg m}^{-3}$ )	1.4	0.5	1.2	0.6
Sand (%)	78.3	ND	43.4	ND
Clay (%)	13.6	ND	42.2	ND
Silt (%)	8.1	ND	14.4	ND
pH-H <sub>2</sub> O	3.9	0.1	3.8	0.1
pH-KCl	3.3	0.1	3.4	0.0
Effective CEC ( $\text{mmol}(+)\text{kg}^{-1}$ )	31.1	7.0	54.6	5.7
Base saturation (%)	24.8	4.0	23.2	3.3
C ( $\text{mg C g}^{-1}$ )	20.1	4.1	35.8	4.6
N ( $\text{mg N g}^{-1}$ )	1.3	0.2	2.5	0.3
C/N ratio	15.5	0.5	14.0	0.2
$\delta^{15}\text{N}$ (‰)	7.7	0.1	9.7	0.3
Total P ( $\text{mg P g}^{-1}$ )	0.1	0.0	0.2	0.0

ND = no determination.

<sup>a</sup>Analysis from leaves was conducted on one composite sample ( $n = 1$ ) made from three samples.

Both soils have a broken laterite layer (0.3–0.4 m thick) at 3–4 m depth. Soil mineralogy is characterized by kaolinite in the clay fraction and quartz in the sand fraction (Ruivo and Cunha, 2003).

## 2.2. Sampling design

Soil samples were collected from the sand and clay sites during the dry (August 2004) and wet (April 2005) seasons. From a randomly selected central transect, five sampling points spaced 50 m apart were delineated. At each sampling point, five undisturbed samples of the topsoil (0–5 cm) were taken within a 0.30 m × 0.30 m area using stainless steel cores of 8 cm diameter. Soil cores were put in cases with built-in fittings to maintain their integrity and packed with blue ice during transport. They were transported from Caxiuanã, Brazil to the Buesgen Institute, Göttingen, Germany, where the N cycling measurements were conducted. Samples were kept cool during the whole duration of the transport. The duration between field sampling and the start of measurement was 2 weeks. Prior to measurements, the soil cores were acclimated in the laboratory for 2 days at 24 °C (average soil temperature of the site).

## 2.3. $^{15}\text{N}$ pool dilution for measurement of gross rates of N mineralization and nitrification

We followed the procedures described by Davidson et al. (1991) for  $^{15}\text{N}$  injection into intact cores and subsequent extraction. From four cores at each sampling point, two were injected with  $(^{15}\text{NH}_4)_2\text{SO}_4$  solution (for gross N mineralization) and two with  $\text{K}^{15}\text{NO}_3$  solution (for gross nitrification). Each core received five 1-mL injections, containing  $30 \mu\text{g N mL}^{-1}$  with 98%  $^{15}\text{N}$  enrichment. This was equivalent to  $1.3 \pm 0.1 \mu\text{g }^{15}\text{N g}^{-1}$  for the sand and  $1.8 \pm 0.1 \mu\text{g }^{15}\text{N g}^{-1}$  for the clay. One core of each labeled pair was immediately extracted with  $0.5 \text{ mol L}^{-1} \text{ K}_2\text{SO}_4$  (approx. 5:1 ratio of solution to dry mass soil). The time elapsed between injection and extraction was 10 min ( $T_0$ ). The  $T_0$  cores were used to correct for the reactions that occur immediately after injection of  $^{15}\text{NH}_4^+$  and  $^{15}\text{NO}_3^-$ . The other pair of labeled cores was incubated in the dark at 24 °C for 1 day for the  $^{15}\text{NH}_4^+$ -labeled cores and for 2 days for the  $^{15}\text{NO}_3^-$ -labeled cores ( $T_1$ ) and was extracted with  $\text{K}_2\text{SO}_4$ . Gross N mineralization and nitrification rates were estimated using the modified calculation procedure of Davidson et al. (1991) from the Kirkham and Bartholomew (1954) model.

## 2.4. N concentration and $^{15}\text{N}$ analyses

$\text{NH}_4^+$  and  $\text{NO}_3^-$  concentrations were analyzed following the same methods described by Corre and Lamersdorf (2004). The organic N content of extracts was determined by persulfate digestion, which involves oxidation of  $\text{NH}_4^+$  and organic N to  $\text{NO}_3^-$  while  $\text{NO}_3^-$  remains the same

(Cabrera and Beare, 1993; Stark and Hart, 1996). The N concentration of the persulfate digests was analyzed using continuous flow injection colorimetry (copper–cadmium reduction method; Cenco/Skalar Instruments, Breda, The Netherlands). Extractable organic N was calculated as the difference between persulfate-N and  $\text{NH}_4^+ + \text{NO}_3^-$  concentrations.

We identified the fates of added  $^{15}\text{N}$  at  $T_0$  by measuring  $^{15}\text{N}$  recoveries in  $\text{K}_2\text{SO}_4$ -extractable N and non-extractable (hereafter insoluble) organic N pools. For  $^{15}\text{N}$  analysis of the extracts, the same diffusion procedure and blank correction described by Corre et al. (2003) and Corre and Lamersdorf (2004) were followed. Part of the  $T_0$  extracts was used for serial diffusion of  $\text{NH}_4^+$  and  $\text{NO}_3^-$  and part was used for persulfate digestion for determination of  $^{15}\text{N}$  enrichment in extractable organic N pool (Corre et al., 2007).  $^{15}\text{N}$  enrichment in the extractable organic N pool was calculated based on isotope mixing equations using the difference in  $^{15}\text{N}$  enrichments and N concentrations between the persulfate-N and  $\text{NH}_4^+ + \text{NO}_3^-$  pools.  $^{15}\text{N}$  recovery in the insoluble organic N pool was calculated from the difference between  $^{15}\text{N}$  recoveries in the total N pool (analyzed from freeze-dried subsamples of the soil cores) and in  $\text{K}_2\text{SO}_4$ -extractable N pools. For the  $T_1$  extracts, only  $\text{NH}_4^+$  was diffused from the  $^{15}\text{NH}_4^+$ -labeled cores (for gross N mineralization), and  $\text{NH}_4^+$  and  $\text{NO}_3^-$  were sequentially diffused from the  $^{15}\text{NO}_3^-$ -labeled cores (for gross nitrification and dissimilatory  $\text{NO}_3^-$  reduction to  $\text{NH}_4^+$  (see below)). Part of the  $T_1$  extracts was reserved for the microbial N immobilization assay (see below).  $^{15}\text{N}$  was analyzed using isotope ratio mass spectrometry (IRMS) (Finigan MAT, Bremen, Germany).

## 2.5. Dissimilatory $\text{NO}_3^-$ reduction to $\text{NH}_4^+$ (DNRA)

We calculated rates of DNRA from the  $^{15}\text{NO}_3^-$ -injected cores as the difference in the  $^{15}\text{NH}_4^+$  atom% between  $T_0$  and  $T_1$ , multiplied by the mean  $\text{NH}_4^+$  pool size during the incubation period, and corrected for isotopic composition of  $^{15}\text{NO}_3^-$  source pool over the incubation interval (Silver et al., 2001, 2005). This was also corrected for any  $\text{NH}_4^+$  transported out of the  $\text{NH}_4^+$  pool (i.e. mean residence time (MRT) of  $\text{NH}_4^+$ ) over the incubation period. Our measured DNRA rates may be stimulated by the added  $^{15}\text{NO}_3^-$  as substrate of DNRA and/or possibly overestimated through microbial  $\text{NO}_3^-$  immobilization and remineralization during the 2-day incubation period.

## 2.6. $\text{NH}_4^+$ and $\text{NO}_3^-$ immobilization rates and microbial biomass C and N by chloroform fumigation

We used the  $T_1$   $^{15}\text{NH}_4^+$ - and  $^{15}\text{NO}_3^-$ -labeled cores to assess  $\text{NH}_4^+$  and  $\text{NO}_3^-$  immobilization rates, respectively. About 25 g of the  $T_1$   $^{15}\text{NH}_4^+$ - and  $^{15}\text{NO}_3^-$ -labeled samples were fumigated with  $\text{CHCl}_3$  for 5 days and extracted with  $0.5 \text{ mol L}^{-1} \text{ K}_2\text{SO}_4$  (approx. 5:1 ratio of solution to dry mass soil). From the fumigated  $T_1$  extracts and the

corresponding unfumigated  $T_1$  extracts, extractable organic N and  $^{15}\text{N}$  enrichment were determined using persulfate digestion and diffusion procedures described by Corre et al. (2007).  $\text{NH}_4^+$  and  $\text{NO}_3^-$  immobilization rates were calculated using the non-linear model described by Davidson et al. (1991).

Microbial biomass C and N were determined using the fumigation-extraction method (Brookes et al., 1985; Davidson et al., 1989) and following the same procedure described by Corre et al. (2007). Organic C from the extracts was analyzed by UV-enhanced persulfate oxidation using a Dohrmann DC-80 Carbon Analyzer with an infrared detector (Rosemount Analytical Division, CA, USA). Organic N was determined using persulfate digestion. Microbial biomass C and N were calculated as the difference in extractable organic C and persulfate-N between the fumigated and unfumigated soils divided by  $k_C = 0.45$  (Joergensen, 1996) and  $k_N = 0.68$  for 5-day fumigated samples (Shen et al., 1984; Brookes et al., 1985).

### 2.7. Calculation of MRT

The MRT indicates the average length of time an N atom resides in a given pool; a lower MRT indicates a faster pool turnover rate and hence a more dynamic pool. The calculation of MRT (N pool/flux rate; e.g. microbial  $N_{\text{MRT}} = \text{microbial N pool}/\text{total N immobilization rate}$ ) assumed that the  $\text{NH}_4^+$ ,  $\text{NO}_3^-$ , and microbial biomass N pools were at steady state and that the fluxes were equal to gross rates of N mineralization, nitrification, and total N ( $\text{NH}_4^+ + \text{NO}_3^-$ ) immobilization, respectively.

### 2.8. Other supporting parameters

Site characteristics (Table 1) were determined at the start of the study. Leaves from the trees were randomly collected to obtain a composite sample in each site. Three replicates of fresh litter, decomposing litter, and mineral soil (at 0–0.05, 0.05–0.10, 0.10–0.25, and 0.25–0.50 m depths) samples were also collected. From air-dried, ground samples, total organic C and N were measured using CNS Elemental Analyzer (Elementar Vario EL, Hanau, Germany) and  $\delta^{15}\text{N}$  using IRMS. From  $\delta^{15}\text{N}$  profile in each site, the  $^{15}\text{N}$  enrichment factor ( $\epsilon$ ) was calculated following similar procedure according to Mariotti et al. (1981) using a Rayleigh equation ( $\epsilon = \delta_s - \delta_{\text{so}}/\ln f$ ).  $\delta_{\text{so}}$  stands for the  $\delta^{15}\text{N}$  value of the input substrate, here the decomposing litter,  $\delta_s$  for the  $\delta^{15}\text{N}$  value at different depths in the soil profile, and  $f$  for the remaining fraction of total N (total N at certain depth divided by the total N of the decomposing litter). The  $\delta^{15}\text{N}$  and  $^{15}\text{N}$  enrichment factor in forests has been used as indirect indicator of N status (Martinelli et al., 1999), soil N cycling behavior (Nadelhoffer and Fry, 1988),  $\text{NO}_3^-$  losses (Vervaeke et al., 2002), and N gaseous emissions (Purbopuspito et al., 2006).

Bulk density was determined using the soil core method (Blake and Hartge, 1986). Particle size distribution was

analyzed using the pipette method with pyrophosphate as a dispersing agent (König and Fortmann, 1996). Soil pH was measured from a saturated paste mixture (1:1 ratio of soil to  $\text{H}_2\text{O}$  and soil to 1 M KCl). Base saturation was calculated as the percentage base cations of the effective cation exchange capacity (CEC); CEC was determined from air-dried, 2-mm sieved samples, percolated with unbuffered 1 M  $\text{NH}_4\text{Cl}$  (König and Fortmann, 1996), and the percolates analyzed for exchangeable cations using Flame-Atomic Absorption Spectrometer (Varian, Darmstadt, Germany). Total P was analyzed from air-dried, ground samples, digested under high pressure in concentrated  $\text{HNO}_3$  (König and Fortmann, 1996), and the digests were analyzed using Inductively Coupled Plasma-Atomic Emission Spectrometer (Spectro Analytical Instruments, Kleve, Germany).

$\text{N}_2\text{O}$  and  $\text{CO}_2$  emissions were measured from the  $T_1$  cores used for the measurement of gross rates of soil N cycling. The soil cores were placed for 30 min in a 1-L glass incubation vessels with a gas sampling port fitting on the lid, from which gas samples were drawn and analyzed for  $\text{N}_2\text{O}$  and  $\text{CO}_2$  using a gas chromatograph (GC 14A, Shimadzu, Duisburg, Germany) equipped with an electron capture detector (Loftfield et al., 1997). Fluxes were calculated from the increase in  $\text{N}_2\text{O}$  and  $\text{CO}_2$  concentrations during the incubation period minus the background  $\text{N}_2\text{O}$  and  $\text{CO}_2$  concentrations (incubation vessel without a soil core). The  $\text{CO}_2$  evolution was used to calculate total C utilized by microbes ([microbial C:N ratio  $\times$  total N immobilization rate] +  $\text{CO}_2$ -C evolution rate), which we used as an index of available C similar to that of Schimel (1988) and Hart et al. (1994). Soil moisture was determined from each soil cores by oven-drying the samples for 24 h at  $105^\circ\text{C}$ , and moisture content was expressed in terms of water-filled pore space (WFPS) using the measured bulk densities.

### 2.9. Statistical analyses

We first tested the spatial independence of our sampling points using the rank version of von Neumann's ratio test (Bartels, 1982). We found that our sampling points spaced at 50 m apart were spatially independent, and hence they were considered replicates in the succeeding analyses. An earlier study on gross rates of microbial N cycling in tropical ecosystem (Corre et al., 2006) also showed sampling points at 10 m apart to be spatially independent. Tests for normality using Kolmogorov–Smirnov  $D$  statistic and for equality of variance using Levene statistic (Sokal and Rohlf, 1981) were conducted for each parameter. Parameters that showed heterogeneous variance were log transformed. Analyses were carried out using two-way analysis of variance (soil types and sampling seasons as factors). Multiple comparisons of treatments were conducted using a Least Significant Difference test at  $P \leq 0.05$ . Means and standard errors were reported as measures of central tendency and dispersion, respectively.

### 3. Results

#### 3.1. $^{15}\text{N}$ recovery from intact cores 10 min ( $T_0$ ) after $^{15}\text{N}$ injection

On average  $88 \pm 5\%$  of the added  $^{15}\text{NH}_4^+$  was recovered in the form added when intact cores were extracted at  $T_0$  (Fig. 1A). We detected no difference in  $^{15}\text{NH}_4^+$  recoveries between soil types. Recoveries of  $^{15}\text{N}$  in the  $\text{NO}_3^-$  pool were negligible ( $1.0 \pm 0.2\%$ ) and recoveries in the extractable organic N pool were not detectable (Fig. 1A).  $^{15}\text{N}$  recovery in the insoluble (not extractable by  $\text{K}_2\text{SO}_4$ ) organic N was only  $10 \pm 3\%$  of the added  $^{15}\text{NH}_4^+$ , and no difference was detected between soil types.  $^{15}\text{N}$  recoveries in the total N pool showed complete recovery of the injected  $^{15}\text{NH}_4^+$  (Fig. 1A).

For the  $^{15}\text{NO}_3^-$ -injected cores, on average  $57 \pm 4\%$  was recovered in the  $\text{NO}_3^-$  pool at  $T_0$ , and there was no difference detected between soil types (Fig. 1B). We detected no  $^{15}\text{N}$  above background level in the  $\text{NH}_4^+$  pool, while the recovery of  $^{15}\text{N}$  in the extractable organic N pool was on average  $12 \pm 5\%$  and did not differ between soil types. Also,  $^{15}\text{N}$  recoveries in the insoluble organic N pool did not differ between soil types and was on average  $25 \pm 6\%$ . For the sandy soil, higher  $^{15}\text{N}$  recovery was measured in the insoluble organic N than in the extractable

organic N, while for the clay similar  $^{15}\text{N}$  recoveries were observed in these pools.  $^{15}\text{N}$  recoveries in the total N pool were not significantly different from 100% (Fig. 1B).

#### 3.2. Gross rates of $\text{NH}_4^+$ transformation, microbial biomass, and available C

High gross N mineralization rates were measured in the clay soil (seasonal average of  $13.5 \pm 3.1 \text{ mg N kg}^{-1} \text{ d}^{-1}$ ), but these did not significantly differ from the rates in the sandy soil (seasonal average of  $6.0 \pm 0.9 \text{ mg N kg}^{-1} \text{ d}^{-1}$ ) because of the high variation. The rates also did not differ between seasons for each soil (Fig. 2).  $\text{NH}_4^+$  immobilization rates were similar to gross N mineralization rates in the dry season, but were only about 50% (sandy soil) to 65% (clay soil) in the wet season (Fig. 2). In the wet season,  $\text{NH}_4^+$  immobilization rates were lower in the sand than in the clay soil. MRT of the  $\text{NH}_4^+$  pool was on average  $3.2 \pm 1$  days and there was no difference between soil types and seasons (Fig. 2). Also, no difference existed between soil types and seasons for  $\text{NH}_4^+$  pool.

Microbial biomass N in the clay soil was more than twice as high as in the sandy soil (Fig. 2), and in the sand, it was higher in the dry than in the wet season (Fig. 2). The MRT of microbial N pool was on average  $10.1 \pm 2.1$  days, and no difference was observed between soil types

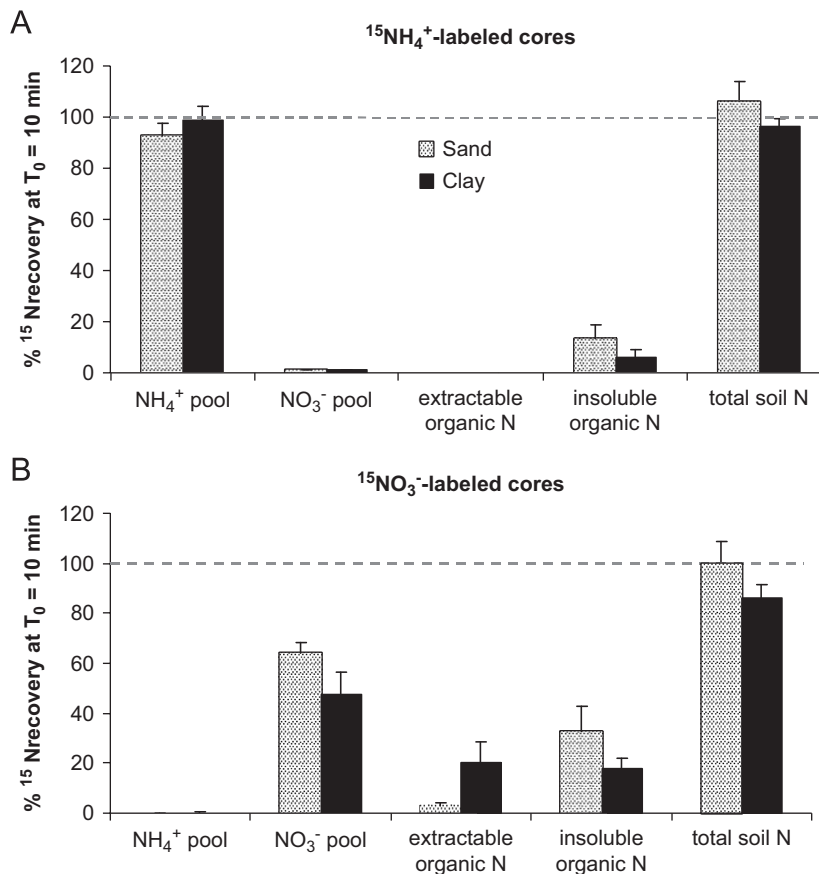


Fig. 1. Percent recovery (means  $\pm 1$  SE;  $n = 5$ ) of  $^{15}\text{N}$  in soil N pools after 10 min ( $T_0$ ) of  $^{15}\text{N}$  injection into the intact cores during the wet-season sampling (April 2005): (A)  $^{15}\text{NH}_4^+$ -labeled cores and (B)  $^{15}\text{NO}_3^-$ -labeled cores.

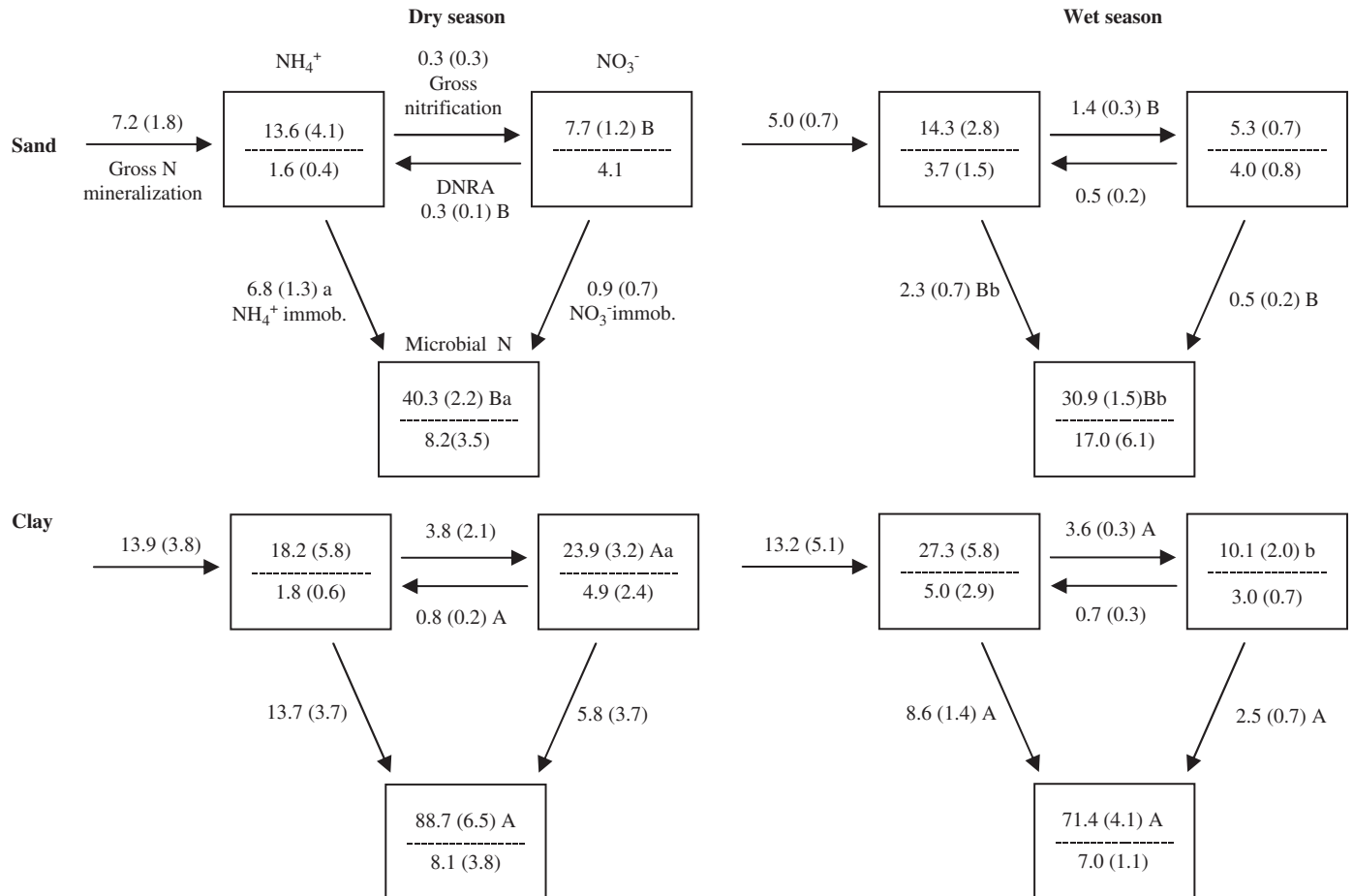


Fig. 2. Means  $\pm$  1 SE ( $n = 5$ ) of gross rates of microbial N cycling ( $\text{mg N kg}^{-1} \text{d}^{-1}$ ), of N pools ( $\text{mg N kg}^{-1}$ ; upper numbers in boxes), and of mean residence time (day; lower numbers in boxes). For each parameter, means with different letters indicate significant differences between soil types at each season (upper case) and between seasons at each soil type (lower case). Arrows in sandy soil, dry season correspond to the same cycling components in clay soil and both seasons.

and seasons. Microbial biomass C was higher in the clay than in the sandy soil and did not differ between seasons. Available C was higher in the clay than in the sandy soil and in the clay, it was higher in the wet than in the dry season (Table 2).

### 3.3. $\delta^{15}\text{N}$ signatures and $^{15}\text{N}$ enrichment factor ( $\epsilon$ )

In both soils,  $\delta^{15}\text{N}$  signatures increased from leaves to litter to soil and with soil depth (Fig. 3).  $\delta^{15}\text{N}$  values of leaves were on average 2.4‰ higher and  $\delta^{15}\text{N}$  values of fresh litter were 1.4‰ higher in the clay than in the sandy soil. We calculated the  $^{15}\text{N}$  enrichment factor, which considers the  $\delta^{15}\text{N}$  signatures from the decomposing litter down to 50-cm depth soil, to get an overall measure of the degree of  $^{15}\text{N}$  enrichment for each soil type. A more negative enrichment factor indicates a faster mineralization of isotopically depleted organic N in the upper soil layers and an enrichment of residual soil organic matter in  $^{15}\text{N}$ . Our calculated  $^{15}\text{N}$  enrichment factors were higher for the clay than for the sandy soil (Fig. 3).

### 3.4. Gross rates of $\text{NO}_3^-$ transformation, dissimilatory $\text{NO}_3^-$ reduction to $\text{NH}_4^+$ (DNRA), and $\text{N}_2\text{O}$ emissions

Gross rates of nitrification and  $\text{NO}_3^-$  immobilization were much lower than gross rates of N mineralization and  $\text{NH}_4^+$  immobilization in both soil types and seasons (Fig. 2). Both gross rates of nitrification and  $\text{NO}_3^-$  immobilization were higher in the clay than in the sandy soil; this was particularly detectable in the wet season when the variation was less (small standard errors; Fig. 2).  $\text{NO}_3^-$  immobilization rates were similar to gross nitrification rates in the dry season (with high variation), but accounted only about 35% (sandy soil) to 69% (clay soil) in the wet season. The  $\text{NO}_3^-$  pool was larger in the clay than in the sandy soil during the dry season; and in the clay soil, the  $\text{NO}_3^-$  pool was larger in the dry than in the wet season (Fig. 2). MRT of the  $^{15}\text{NO}_3^-$  pool was on average  $3.9 \pm 0.6$  days and no difference was detected between soil types or seasons (Fig. 2). Rates of DNRA were higher in the clay than in the sandy soil during the dry season, and did not differ between seasons for each soil type (Fig. 2). For the sandy soil, DNRA rates were similar to gross nitrification rates in the dry season but this

Table 2  
Microbial C, available C, water-filled pore space and rates of N<sub>2</sub>O emission for Oxisols with sand and clay texture at Caxiuana National Forest, Brazil

	Sand		Clay	
	Mean	SE	Mean	SE
Microbial C (mg C kg <sup>-1</sup> )				
Dry season	338 B	17	820 A	57
Wet season	329 B	5	837 A	42
Available C (mg C kg <sup>-1</sup> d <sup>-1</sup> ) <sup>a</sup>				
Dry season	97	46	218 b	40
Wet season	163 B	11	392 Aa	62
N <sub>2</sub> O emission (mg N kg <sup>-1</sup> d <sup>-1</sup> )				
Dry season	2.9 B	2.1	19.3 Aa	5.1
Wet season	0.1	0.0	6.6 b	1.8
Water-filled pore space (%)				
Dry season	51 B	5.4	71 Ab	1.7
Wet season	58 B	3.8	88 Aa	5.9

Means  $\pm$  1 SE ( $n = 5$ ) with different letters indicate significant differences between soil types at each season (upper case) and between seasons at each soil type (lower case) (two-way ANOVA, Least Significant Difference test at  $P \leq 0.05$ ). There was no significant difference in N<sub>2</sub>O emissions between the T<sub>1</sub> cores injected with <sup>15</sup>NH<sub>4</sub><sup>+</sup> and <sup>15</sup>NO<sub>3</sub><sup>-</sup>; data for each replicate were the average from the two T<sub>1</sub> cores.

<sup>a</sup>Calculated using a similar index to that of Schimel (1988) and Hart et al. (1994), where available C = [(microbial C:N ratio  $\times$  total N immobilization rate) + CO<sub>2</sub>-C evolution rate].

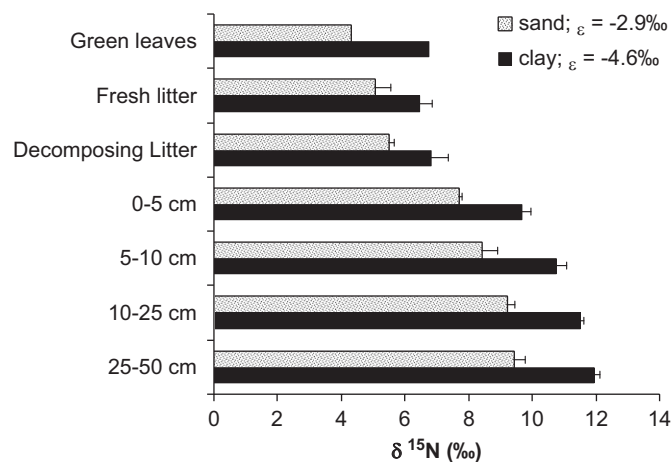


Fig. 3. The <sup>15</sup>N profiles (‰; means  $\pm$  1 SE,  $n = 3$ ) and <sup>15</sup>N enrichment factor ( $\epsilon$ ) of the sand and clay Oxisol.

reduced to only about 35% in the wet season. For the clay, DNRA rates were about 20% of the gross nitrification rates in both seasons (Fig. 2). When comparing their importance for NO<sub>3</sub><sup>-</sup> retention, rates of DNRA and NO<sub>3</sub><sup>-</sup> immobilization were similar in the sand, but in the clay DNRA rates were only 14–28% of NO<sub>3</sub><sup>-</sup> immobilization rates (Fig. 2). N<sub>2</sub>O emission rates were higher in the clay than in the sand (Table 2). For the sandy soil, we did not detect differences in N<sub>2</sub>O emission rates between sampling periods. For the

clay soil, N<sub>2</sub>O emissions were higher in the dry than in the wet period, possibly indicating further reduction of N<sub>2</sub>O to N<sub>2</sub> under a more O<sub>2</sub>-reduced condition (i.e. high WFPS, Table 2).

#### 4. Discussion

##### 4.1. Implications of the rapid reaction of injected <sup>15</sup>NH<sub>4</sub><sup>+</sup> and <sup>15</sup>NO<sub>3</sub><sup>-</sup> to organic N

Our <sup>15</sup>NH<sub>4</sub><sup>+</sup> recoveries at T<sub>0</sub> were higher than those reported for mineral soil of N-limited, tropical montane forests in Indonesia (Corre et al., 2006) and in Hawaii (Hall and Matson, 1999), but comparable with N-rich montane forest in Hawaii (Hall and Matson, 1999). The fast reaction of added <sup>15</sup>NH<sub>4</sub><sup>+</sup> to organic N pool is usually attributed to abiotic NH<sub>4</sub><sup>+</sup> immobilization (e.g. physical condensation reactions with phenolic compounds (Nömmik 1970; Nömmik and Vahtras, 1982; Johnson et al., 2000), and fixation on clay minerals (Davidson et al., 1991)). The higher <sup>15</sup>NH<sub>4</sub><sup>+</sup> recoveries at our sites compared to N-limited, tropical montane forests may indicate lower NH<sub>4</sub><sup>+</sup>-retention capacity in these soils through rapid, abiotic NH<sub>4</sub><sup>+</sup> reactions.

Our <sup>15</sup>NO<sub>3</sub><sup>-</sup> recoveries at T<sub>0</sub> from both soil types were much higher than those reported for mineral soil of N-limited, tropical montane forest in Indonesia (Corre et al., 2006). There are no other values reported for tropical forests to which we can compare our results. However, recent studies in temperate forests reported fast reaction of added <sup>15</sup>NO<sub>3</sub><sup>-</sup> to organic N (Dail et al., 2001; Fitzhugh et al., 2003; Corre and Lamersdorf, 2004; Corre et al., 2007). Using soil sterilization methods, this fast reaction of NO<sub>3</sub><sup>-</sup> to organic N has been attributed to abiotic immobilization (Dail et al., 2001; Fitzhugh et al., 2003), and is considered as a possible additional mechanism of N retention in temperate ecosystems (Davidson et al., 2003). The significance of this process to N retention in tropical forests, especially when changes in N deposition occur, deserves more attention.

##### 4.2. Gross rates of NH<sub>4</sub><sup>+</sup> transformation, and $\delta^{15}N$ values and overall <sup>15</sup>N enrichment factor ( $\epsilon$ ) indicate high gaseous losses in clay soil

Gross N mineralization rates in the sandy soil were comparable to the rates measured from a lowland forest soil in Rondonia, Southern Amazon (with 23–35% clay; Neill et al., 1999), and from an N-limited, montane forest soils in Hawaii (Hall and Matson, 2003) and in Indonesia (Corre et al., 2006). In the clay soil, gross N mineralization rates were comparable to the rates from a long-term (11 yr) N-fertilized (formerly N-limited), montane forest in Hawaii (Hall and Matson, 2003) and from lowland forest soils (with 68–76% clay; Veldkamp et al., 2003) in Costa Rica (Silver et al., 2005). These rates were only about half of the

rates measured from an N-rich, montane forest soil in Hawaii (Hall and Matson, 2003).

The high gross N mineralization rates observed in the clay soil, although not statistically significant, are coherent with the low C:N ratios in litter and soil, high total soil N (Table 1), high microbial N (Fig. 2), microbial C, and available C (Table 2). Gross N mineralization reflects both the microbial biomass size (presumably active in mineralization, e.g. chemoheterotrophs) and the quantity (e.g. high total soil N and available C) and quality (e.g. low C:N ratio) of available substrate. The positive correlations of gross N mineralization to  $\text{NH}_4^+$  immobilization ( $r = 0.67$ ;  $P < 0.05$ ), and  $\text{NH}_4^+$  immobilization to microbial biomass N ( $r = 0.59$ ;  $P < 0.05$ ) attest that the high gross N mineralization rates in the clay soil provided high available N for microbial assimilation and hence supported a high microbial biomass. On the other hand, the low gross N mineralization rates observed in the sandy soil are probably due to lower litter quality (high C:N ratio; Table 1) which was also reflected in the lower available C and microbial biomass (Table 2; Fig. 2). Low litter quality (Silver et al., 2000) and nutrient and water limitations for decomposition (Cuevas and Medina, 1986) have been reported for sandy Amazonian forest soils.

Our  $\delta^{15}\text{N}$  values for leaves and soil were within the range reported for Amazonian forest soils (2–8‰ for leaf litter (Piccolo et al., 1994) and 8–23‰ for soil (Piccolo et al., 1996)). The  $\delta^{15}\text{N}$  signatures and the  $^{15}\text{N}$  enrichment factor reflect the long-term behavior of the soil N cycle of an ecosystem. N-rich forest ecosystems with a leaky N cycle have high  $\delta^{15}\text{N}$  signatures because isotopically light N is lost from the ecosystem owing to fractionation during nitrification and denitrification, leaving isotopically enriched N behind. In our study, the clay soil that had high gross N mineralization and nitrification rates also had higher  $\delta^{15}\text{N}$  in leaves, litter and soil compared to the sandy soil (Fig. 3). The  $\delta^{15}\text{N}$  values in leaves have been used as an indicator of changing N status across successional forest chronosequence in Amazon basin (Davidson et al., 2007). The  $\delta^{15}\text{N}$  signatures of litter and soil were also directly correlated with gaseous N losses along a toposequence of montane forest soils in Indonesia (Purbopuspito et al., 2006). In Amazonian forest soils, the discrimination factor (which has similar meaning as the  $^{15}\text{N}$  enrichment factor but only has an opposite sign) generally increased with increasing clay content of the soil (Piccolo et al., 1996). Our study showed that the clay soil that had high N cycling rates also had a higher  $^{15}\text{N}$  enrichment factor (Fig. 3) and higher gaseous losses (i.e.  $\text{N}_2\text{O}$  emissions; Table 2) than the sandy soil.

#### 4.3. Gross rates of $\text{NO}_3^-$ transformation and the importance of $\text{NO}_3^-$ immobilization over dissimilatory $\text{NO}_3^-$ reduction to $\text{NH}_4^+$ (DNRA) for retention of $\text{NO}_3^-$

Gross rates of nitrification in the sandy soil were comparable to those reported for N-limited, montane

forests in Hawaii (Hall and Matson, 2003) and in Puerto Rico (Silver et al., 2001). These rates were lower than those measured from a lowland forest soil in Rondonia, Southern Amazon (with 23–35% clay; Neill et al., 1999). However, gross nitrification rates in the clay soil were higher than this Rondonia site and were comparable to the rates from a lowland forest with heavy clay soil in Costa Rica, characterized by high N availability (Silver et al., 2005) and large N losses (gaseous N losses, Keller et al., 1993; Keller and Reiners, 1994; Veldkamp et al., 1999; and leaching losses, Schwendenmann and Veldkamp, 2005).

Despite the high WFPS in the clay soil during the wet season (Table 2), which may limit  $\text{O}_2$  diffusion and decrease nitrification (Paul and Clark, 1996), higher gross nitrification rates were observed in the clay compared to the sandy soil. This suggests that substrate availability ( $\text{NH}_4^+$  for autotrophic nitrifiers and organic N for heterotrophic nitrifiers) limits nitrification activity rather than aeration. In the absence of root uptake, mineralized N was largely assimilated by microorganisms rather than nitrified (Fig. 2). Low gross N mineralization rates in the sandy soil may have imposed strong competition for  $\text{NH}_4^+$ , resulting in low gross nitrification rates.

The low DNRA rates in the sandy soil were comparable to the rates in lowland forest in Costa Rica (Silver et al., 2005), while the high DNRA rates in the clay soil were comparable to the montane forest in Puerto Rico (Silver et al., 2001). As opposed to the values reported by Silver et al. (2001) in which DNRA rates were stimulated by the added  $^{15}\text{NO}_3^-$  and exceeded gross nitrification, our measured DNRA rates did not exceed gross nitrification rates (Fig. 2). Contrary to what Silver et al. (2005) found, we did not detect correlations between DNRA rates and total C: $\text{NO}_3^-$  ratios or to any indicators of  $\text{NO}_3^-$  availability (e.g. gross nitrification or  $\text{NO}_3^-$  pool). Instead we observed a marginal relationship between DNRA and available C ( $r = 0.62$ ,  $P = 0.06$ ), indicating that this  $\text{NO}_3^-$  retention process may be in part driven by the availability of C to microorganisms. High DNRA rates in the clay soil also signified that this process is favored under a more  $\text{O}_2$ -reduced condition (i.e. high WFPS, Table 2). In terms of its significance for  $\text{NO}_3^-$  retention, only in the sandy soil, where production of  $\text{NO}_3^-$  was low, DNRA was as important as  $\text{NO}_3^-$  immobilization. In the clay soil, where gross nitrification rates and  $\text{NO}_3^-$  levels were high, DNRA was 3–7 times lower than  $\text{NO}_3^-$  immobilization.  $\text{N}_2\text{O}$  emission rates in the clay soil (Table 2) were also higher than the combined rates of DNRA and  $\text{NO}_3^-$  immobilization (Fig. 2), while in the sandy soil  $\text{N}_2\text{O}$  losses were lower than the sum of the rates of these  $\text{NO}_3^-$  retention processes. The low  $\text{N}_2\text{O}$  emissions in the sandy soil reflect the overall low rates of soil N cycling in this soil as well as its low WFPS and C availability. In contrast, the clay soil typified a system with leaky N cycle—rates of internal soil N cycling were lower than rates of N loss (e.g.  $\text{N}_2\text{O}$  emission).

#### 4.4. MRT and implication for N losses

MRT of  $\text{NH}_4^+$  during the dry season was similar to the values reported for Rondonia lowland forest soil (Neill et al., 1999) and for Indonesian montane forest soil (Corre et al., 2006). Hart et al. (1994) have shown that turnover rates of N pools responded to changes in C availability that drives the internal soil N cycle. We did not observe differences in turnover rates of  $\text{NH}_4^+$  and microbial N pools (Fig. 2), although differences in available C between soils existed (Table 2). The MRT of  $\text{NO}_3^-$  were comparable to the values reported for Rondonia lowland forest soil (Neill et al., 1999), but higher than those observed from tropical montane forest soils where  $\text{NO}_3^-$  was sometimes even undetectable (Silver et al., 2001; Hall and Matson, 2003; Corre et al., 2006). A long MRT of  $\text{NO}_3^-$  signals high potential for N losses in times of high soil water content. Leaching losses (e.g. Schwendenmann and Veldkamp, 2005; Klinge et al., 2004; Dechert et al., 2005) and N-oxide emissions (e.g. Verchot et al., 1999; Davidson et al., 2000; Purbopuspito et al., 2006) from tropical lowland forests were reported to be higher than tropical montane forests. Furthermore, we observed somewhat comparable  $\text{NO}_3^-$  immobilization rates and gross nitrification rates in the dry season while  $\text{NO}_3^-$  immobilization rates tended to be lower than gross nitrification in the wet season. When the sink of mineral N pool by microbial immobilization is low,  $\text{NO}_3^-$  could be more exposed to losses. We speculated that the overall gaseous N losses from the clay soil during the wet season could be higher than the measured  $\text{N}_2\text{O}$  emissions alone as  $\text{N}_2\text{O}$  can be further reduced to  $\text{N}_2$  under a more  $\text{O}_2$ -reduced condition (i.e. high WFPS during the wet season in the clay, Table 2), and this may have resulted to the lower  $\text{NO}_3^-$  levels during the wet than dry season in the clay soil.

#### 5. Conclusions

This study shows that large differences exist in N cycling and N retention between heavily weathered tropical forest soils that mainly differ in texture. While the clay Oxisol in our study had high gross rates of N mineralization and nitrification, and hence high potential for N losses, the sandy Oxisol had low gross rates of N cycling and reacted more like an N-limited soil. The  $\delta^{15}\text{N}$  signatures and the  $^{15}\text{N}$  enrichment factor, which reflect the long-term behavior of soil N cycling of an ecosystem, were also higher in the clay than in the sandy soil. Our combined results suggest that the N-limited sandy Oxisol may react positively to increased anthropogenic N deposition, at least within a short-term range, if other edaphic factors will favor for increased N uptake by plants, improved litter quality, increased decomposition and microbial biomass, and ultimately increased N retention. The clay Oxisol with a leaky N cycle may show immediate N loss under elevated N deposition if N accumulation by plants will be unaffected and if microbial biomass, microbial N immo-

bilization, and abiotic N retention would decrease, as observed in N-rich temperate forest soils. Furthermore, our findings of faster turnover rates of  $\text{NH}_4^+$  compared to  $\text{NO}_3^-$  signified that  $\text{NH}_4^+$  cycles faster through microorganisms than  $\text{NO}_3^-$ , possibly contributing to better retention of  $\text{NH}_4^+$  than  $\text{NO}_3^-$ . This was opposite to abiotic retention, which showed higher conversion of  $\text{NO}_3^-$  than  $\text{NH}_4^+$  to the organic N pool. How this will affect long-term N retention is presently unknown and cannot be deduced from the present study, which illustrates that the importance of biotic and abiotic N retention needs further attention.

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