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**Slash and Char as Alternative to Slash and Burn**  
*soil charcoal amendments maintain soil fertility and establish a carbon sink*



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## EXTENDED SUMMARY

### Introduction

Tropical forests account for between 20 and 25% of the world terrestrial carbon (C) budget (Bernoux *et al.* 2001). Soils under tropical forest contain approximately the same amount of C as the lush vegetation above it. On average they contain 2.7% C in the surface horizons and 0.5% in the subsurface horizons to 100 cm depth (Sombroek *et al.* 2000).

The current rapid conversion of Amazonian forest to agricultural land makes disturbance of this C stock important to the global C balance and net greenhouse gas emissions. The net release of soil C was 8.5 Mg ha<sup>-1</sup>, or 11.7 Gg of C for the 1.38 million hectares cleared in 1990. C emissions from soil alone as a result of deforestation in the Amazon represent a quantity of C approximately 20% as large as Brazil's annual emission from fossil fuels (Fearnside and Barbosa 1998).

Changes in land use, particularly by clearing forests, reduce organic C by 20% to 50% in the upper soil layers (Sombroek *et al.* 1993). This reduction of soil organic matter (SOM) is responsible for soil degradation. A slash-and-burn site investigated by Tiessen *et al.* (1994) had lost 81% of its litter layer and 29% of its soil C to 0.15 m depth over 3 years. Tiessen *et al.* (1994) concluded that the accelerated SOM decay under agriculture will lead to mineralization of over half the nutrients in 2 years. Thus agriculture is not sustainable without nutrient inputs beyond 3 years of cultivation, although the release of remaining nutrients can provide for the re-establishment of secondary successions (Tiessen *et al.* 1994).

On soils with low nutrient retention capacity the strong tropical rains easily leach available and mobile nutrients, such as those supplied by inorganic nitrogen (N) fertilizers, rapidly into the subsoil where they are unavailable for most crops (Giardina *et al.* 2000; Hölscher *et al.* 1997; Renck and Lehmann 2004) limiting the efficiency of conventional fertilizers.

Therefore slash-and-burn agriculture is practiced by about 300 to 500 million people, affecting almost one third of the planet's 1500 million ha of arable land (Giardina *et al.* 2000; Goldammer 1993). This traditional agricultural practice is considered to be sustainable if adequate fallow periods (up to 20 years) follow a short time of cultivation (Kleinman *et al.* 1995). A growing population with changing socio-economic habits may not be able to practice slash-and-burn in a sustainable way. In most agricultural systems the tendency has been for population pressure to increase, leading to shorter fallow periods (Fearnside 1997).

Maintaining high levels of SOM in tropical soils would be a further step towards sustainability and fertility on tropical agricultural land, thus reducing the pressure on intact primary forests. Charcoal formation and deposition in soils seems to be a promising option to transfer an easily decomposable biomass into refractory SOM pools (Fearnside *et al.* 2001; Glaser *et al.* 1998, 2001b; Zech *et al.* 1990). However, charcoal formed through traditional slash-and-burn techniques represents just 1.7% of the pre-burn biomass (Fearnside *et al.* 2001).

The existence of an anthropogenic and C-enriched dark soil in different parts of the world and especially in Amazonia (Amazonian Dark Earths (ADE) or *Terra Preta de Índio*) proves that the predominant Ferralsols and Acrisols can be transformed into fertile soils. The ADE's fertility is most likely linked to an anthropogenic accumulation of phosphorus (P), calcium (Ca) associated with bone apatite (Lima *et al.* 2002, Zech *et al.* 1990), and black C as charcoal (Glaser *et al.* 2001a). Charcoal persists in the environment over centuries and is responsible for the stability of the ADE's SOM. Fertility persists to the present under continuous agriculture by contemporary and likely intensive cultivation by native populations.

## Objectives

The sustained fertility in charcoal-containing ADE and the frequent use of charcoal as a soil conditioner (Steiner *et al.* 2004) in Brazil and other parts of the world (mainly Japan) (Ogawa 1994a) provided the incentive to study the effects of charcoal application to a highly weathered soil (Lehmann *et al.* 2003). The production of charcoal for soil amelioration purposes (slash and char) out of the aboveground biomass (secondary forest and crop residues) instead of converting it to carbon dioxide (CO<sub>2</sub>) through burning (slash-and-burn) could establish a significant C sink and could be an important step towards sustainability and SOM conservation in tropical agriculture.

Therefore, the aim of this dissertation is to examine the use of charcoal in agricultural practice and management of a highly weathered Xanthic Ferralsol on *terra firme* north of Manaus (Brazil). The first chapter describes indigenous soil fertility management by burning and use of organic amendments. A socio-economic study on charcoal producers collected information on household economic activity, demographic composition, and access to land, labour, and capital. Particular attention was given to charcoal production, wood procurement, labour input, charcoal output, and economic returns in comparison to their agricultural activities. Discussions and first-hand observations provided more general information about production techniques, risks and use of charcoal waste in agriculture. The second chapter (II) discusses the feasibility of slash and char with and without carbon trade mechanism for small farmers and the potential for carbon sequestration. Observations about present charcoal use are summarized in chapter III. The influence of charcoal and condensates from smoke (pyrolygneous acid, PA) on the microbial activity was assessed via measurements of basal respiration, substrate induced respiration, and exponential population increase after substrate addition (chapter IV). The effectiveness of charcoal as slow release nutrient carrier (N, P, and K) was studied in a greenhouse experiment. Rice seedlings were fertilized with mineral N, P, and K either based on charcoal or kaolin as a nutrient carrier in order to assess the availability of nutrients to plants and microorganisms (chapter V). Long term effects of different organic matter (OM) applications and mineral fertilization on soil fertility and crop production were assessed in the chapters VI, VII, VIII and IX. Chapter VII delineates the influence on the soil microbial population by measurement of soil respiration. Potential microbial growth after substrate (glucose) addition served as a soil fertility indicator for *Terra Preta* and Ferralsol soils. Fertilization with <sup>15</sup>N labelled nitrogen provided information about the retention of N on plots amended with charcoal or compost in comparison to only mineral fertilized plots (chapter VIII). Weed succession, pressure and species composition on these plots is described in chapter IX. Chapter X investigated organic and inorganic soil fertility management in two different perennial crops by comparing soil respiration, and soil chemical properties. Finally an experiment was established on an expanding banana plantation in order to test the suitability of charcoal application in the local farming context. Chapter XI deals with foliar and soil nutrient contents, nutrient leaching, bulk density and water retention in soils amended with charcoal (powder, pieces, and waste as available) in comparison to the normal agricultural practice.

## Material and Methods

This Dissertation comprises field (chapters VI, VII, VIII, IX, X, and XI), greenhouse (chapter V) and laboratory experiments (chapter IV). In addition, data and information were gathered at local charcoal production sites (chapters II and III) and indigenous soil fertility management (chapter I) was observed and described.

### *Study Locations*

Most experiments (chapters IV, V, VI, VII, VIII, IX, and X) were established 30 km north of Manaus, Amazonas, Brasil (3°8'S, 59°52'W, 40–50 m a.s.l.) at the Embrapa-Amazônia Ocidental (Empresa Brasileira de Pesquisa Agropecuária) experimental research station. The natural vegetation is evergreen tropical rainforest with a mean annual precipitation of 2530 mm (1971–1997) having its seasonal maximum between December and May, a mean annual temperature of 25.8 °C (1987–1997) and a relative humidity of 85 % (Correia and Lieberei 1998). The soil is classified as a highly weathered Xanthic Ferralsol (FAO 1990) derived from Tertiary sediments. The soil is fine textured with up to 80% clay. It is strongly aggregated and has medium contents of organic C (24 g kg<sup>-1</sup>), low pH values of 4.7 (in H<sub>2</sub>O), low CEC of 1.6 cmol<sub>c</sub> kg<sup>-1</sup> and low base saturation (BS) of 11.2 % (chapter VI; appendix, table II).

The data for the chapters I, II, III, and XI were collected outside the research station. An indigenous family group was visited several times over a time period of one year (chapter I). For 9 years they have been settled at the Ariau River about 40 km southwest from the city Manaus, between the Negro and Solimões rivers. The village location is close to the river and the forested part of it is influenced by seasonal flooding.

Information about charcoal production and marketing were gathered in the vicinity of Manaus (chapters II and III). Primary research was carried out in the Taruma Mirim Settlement situated at km 21 on the BR 174 highway that links Manaus, Amazonas to Boa Vista, Roraima. It was created in 1992 as an agricultural settlement by INCRA (*Instituto de Nacionalizacao Colonizacao e Reforma Agraria* or the National Institute for Colonization and Agricultural Reform) and is situated between the streams of Taruma Acu and Taruma Mirim. Other areas in the region were also explored on an informal basis as a source of information on charcoal making activities near Manaus, including the banks of the BR-174 highway and the settlement Canoas/Rio-Pardo. The latter was created by INCRA for the same purposes as Taruma Mirim.

One trial was carried out in the local farming context (chapter XI) in order to provide cheap sustainable options to improve crop yields in the tropics. This experiment was established within an existing and expanding banana plantation north of Manaus on kilometre 99 along the road BR 174 leading to Boa Vista.

### *Experimental Setups and Designs*

Study IV (chapter IV) used fifty kg sieved topsoil (0–0.1 m) from an experimental bare soil area (see table 4-1, chapter IV, for chemical characteristics). Chicken manure was mixed with the soil to ensure a high microbial activity. The manure was dosed to apply about 65 Mg ha<sup>-1</sup> (65 g kg<sup>-1</sup>) in the first 10 cm of soil. The prepared soil was stored in a box for 10 weeks in the dark at a humidity of 28 %. To assess effects of charcoal portions of 40 g of soil (dry weight) were amended with 0 (treatment C), 2 (treatment CI), 4 (treatment CII), or 6 g (treatment CIII) charcoal powder (0, 50, 100, 150 g kg<sup>-1</sup> respectively) prior to measurement (chapter IV, table 4-2). The humidity of the charcoal powder was equalized to that of the soil. Each treatment was measured in 3 repetitions. The effect of pyroligneous acid (PA) on microbial respiration was tested in a factorial design (chapter IV, table 4-2). A volume of 0.5 ml of “biopirol” (Biocarbo, Itabirito, MG, Brazil) mixed with 2 g charcoal or kaolin was added per 40 g soil. Biopirol is the first fraction obtained during distillation of wood tar. Two ml H<sub>2</sub>O accounted for the factor “humidity” and 240 mg (6 g kg<sup>-1</sup>) glucose were applied as usual after measurement of the basal respiration.

Two kinds of mineral fertilizers were prepared for the experiment delineated in chapter V. One treatment was based on charcoal powder as a nutrient carrier and the other on kaolin. OSP, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, and KCl and were either mixed with kaolin or with charcoal powder prior to fertilization. The fertilizers were dosed to apply 3.6 g elemental N, K and P per kg of

charcoal or kaolin which corresponds to about 40 kg ha<sup>-1</sup> of these elements at a charcoal application of 11 Mg ha<sup>-1</sup>. The mineral fertilizers were dissolved in distilled water, charcoal or kaolin was stirred into the solution and left for drying at room temperature. Topsoil (0-0.1 m) was collected and sieved (<2mm). After adding water (humidity of 30%) the soil was amended with dried and milled elephant grass (*Penissetum purpurium*) (1 kg DW 50 kg<sup>-1</sup> of soil) and stored in a closed box for two months. Elephant grass was added to ensure a basic biodegradable C-stock for microbes. The soil was fertilized in a manner to create 5 treatments and divided into 5 different boxes. Treatment 1 contained unfertilized soil *Penissetum* mix (C); Treatment 2 (CI) was fertilized with charcoal based fertilizer (11 Mg ha<sup>-1</sup>, N, P, K 40 kg ha<sup>-1</sup>); in treatment 3 (KI) the same amount of NPK was applied but kaolin based (11 Mg ha<sup>-1</sup>); and, treatments 4 and 5 were fertilized with twice the amount of either charcoal (CII) or kaolin based fertilizer (KII), respectively (chapter V, table 5-1). Pots of 11.5 cm height and 12.5 cm diameter were filled with 840 g (DW) soil each. Twenty-five pots were filled to form 5 repetitions of each treatment. The pots were randomly distributed on a table in the centre of the greenhouse. In each pot 5 pre-germinated rice seedlings were planted (February 2<sup>nd</sup> 2003). Insect control (caterpillars) was done manually and every day if encountered. The rice plants were watered with 25 ml per pot for the first 3 days. No water occurred at the bottom of the pot at that watering level. From 10<sup>th</sup> February to 6<sup>th</sup> May 100 ml of water was applied daily and the leachate was collected for analyses of N, P, K, and pH. After 4 months the pots were emptied, the rice biomass was dried at 65°C and weighed separately as roots and aboveground biomass.

The studies delineated in the chapters VI, VII, VIII, and IX were part of a long term field experiment. Fifteen different amendment combinations (table 6-1) based on equal amounts of applied C in chicken manure, compost, charcoal and forest litter were tested during four cropping cycles with rice (*Oryza sativa* L.) and sorghum (*Sorghum bicolor* L.) in five repetitions. After clearing of about 3600 m<sup>2</sup> secondary forest, and removing the aboveground biomass, the treatments were applied on 4 m<sup>2</sup> plots (2x2 m). Charcoal derived from secondary forest wood, was bought from a local distributor. It was manually crushed to particle sizes smaller than 2 mm. The applied 11 Mg ha<sup>-1</sup> charcoal corresponded to the amount of charcoal-C which could be produced by a single slash-and-char event of a typical secondary forest on Xanthic Ferralsols in central Amazonia (Lehmann *et al.* 2002). The amount of C added with charcoal was chosen as a reference value for adding the compost, litter and chicken manure amendments. From the 12<sup>th</sup> to the 20<sup>th</sup> of February 2001 the fields were hoe-harrowed to 0.10 m depth and the organic amendments were mixed in with the soil. Mineral fertilizer (NPK and lime) was applied as ammonium sulphate [(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>], ordinary super phosphate (OSP), and potassium chlorite (KCl) as recommended by Embrapa (Fageria 1998). Organic materials were applied just once at the beginning of the experiment (February 3<sup>rd</sup> 2001). Mineral fertilizer was applied in March 2001 and after the second harvest in April 2002 (table 6-1, chapter VI). At the second fertilization the treatments L, and CCp+<sup>1</sup>/<sub>2</sub>CO+F additionally received micronutrients. Those treatments received mineral fertilization for the first time. As a first crop rice (*Oryza sativa* L.) was planted followed by three repeated sorghum (*Sorghum bicolor* L. Moench) crops. Rice was planted March 10<sup>th</sup> 2001 in a density of 200 seeds per m<sup>2</sup>, followed by sorghum planted on October 15<sup>th</sup> 2001 in a density of 12.5 plants per m<sup>2</sup>, the 3<sup>rd</sup> crop was established in a density of 25 plants per m<sup>2</sup> on April 18<sup>th</sup> 2002, the latter producing two harvests by ratooning.

Table 6-1. Treatments and applications of organic matter (in brackets Mg ha<sup>-1</sup>) and nutrients (in brackets kg ha<sup>-1</sup>), lime (2100 ~460 Ca, 270 Mg) 2800 ~613 Ca, 360 Mg), lime (430 ~94 Ca, 55 Mg)

Treatment	Organic Matter [Mg ha <sup>-1</sup> ]	Nutrient contents of organic matter [kg ha <sup>-1</sup> ]	1 <sup>st</sup> fertilization [kg ha <sup>-1</sup> ]	2 <sup>nd</sup> fertilization [kg ha <sup>-1</sup> ]
C	control	-----	-----	-----
L	litter (13)	N (114), P (0.3), K (4.3), Ca (13.3), Mg (4.7)	-----	N (55), P (40), K (50), lime* (2800), Zn (7), B (1.4), Cu (0.6), Fe (2.3), Mn (1.6), Mo (0.08)
LB	burned litter (13 Mg litter burned on the plot)	N (??), P (0.3), K (4.3), Ca (13.3), Mg (4.7)	-----	N (55), P (40), K (50), lime* (2800)
F	mineral fertilizer	-----	N (30), P (35), K (50), lime*(2100)	N (55), P (40), K (50), lime* (430)
CM	chicken manure (47)	N (774), P (324), K (836), Ca (784), Mg (143)	-----	-----
CO	compost (67)	N (681), P (49), K (191), Ca (219), Mg (101)	-----	-----
CC	charcoal (11)	N (59), P (0.3), K (2.5), Ca (9.0), Mg (1.9)	-----	-----
CO+F	compost (67)	N (681), P (49), K (191), Ca (219), Mg (101)	N (30), P (35), K (50), lime*(2100)	N (55), P (40), K (50), lime* (430)
CC+F	charcoal (11)	N (59), P (0.3), K (2.5), Ca (9.0), Mg (1.9)	N (30), P (35), K (50), lime*(2100)	N (55), P (40), K (50), lime* (430)
CC <sup>1/2</sup> + <sup>1/2</sup> CO	charcoal (5,5), compost (33,5)	N (370), P (24.5), K (96.8), Ca (114), Mg (51.6)	-----	-----
CC <sup>1/2</sup> + <sup>1/2</sup> CO + F	charcoal (5,5), compost (33,5)	N (370), P (24.5), K (96.8), Ca (114), Mg (51.6)	N (30), P (35), K (50), lime*(2100)	N (55), P (40), K (50), lime* (430)
CC + <sup>1/2</sup> CO	charcoal (11), compost (33,5)	N (399), P (24.7), K (98.1), Ca (118.6), Mg (52.5)	-----	-----
CC + <sup>1/2</sup> CO + F	charcoal (11), compost (33,5)	N (399), P (24.7), K (98.1), Ca (118.6), Mg (52.5)	N (30), P (35), K (50), lime*(2100)	N (55), P (40), K (50), lime* (430)
CCp + <sup>1/2</sup> CO	charcoal pieces (11), compost (33,5)	N (399), P (24.7), K (98.1), Ca (118.6), Mg (52.5)	-----	N (55), P (40), K (50), lime* (2800), Zn (7), B (1.4), Cu (0.6), Fe (2.3), Mn (1.6), Mo (0.08)
CCp + <sup>1/2</sup> CO	charcoal pieces (11), compost (33,5)	N (399), P (24.7), K (98.1), Ca (118.6), Mg (52.5)	-----	N (55), P (40), K (50), lime* (2800)

\*lime (2100) ~460 Ca, 270 Mg, (2800) ~613 Ca, 360 Mg, (430) ~94 Ca, 55 Mg)

In order to study the influence of charcoal, N and P two field experiments were established with two different perennial crops (banana, *Musa sp.*; guarana, *Paullinia cupana*) in a confounded factorial design (chapter X). Each plantation tested three different factors in three different levels making up 27 (3<sup>3</sup>) treatment combinations. Whereas the banana plantation received mineral fertilization in addition to the charcoal applications the guarana was fertilized organically using chicken manure and bones meal as the corresponding factors. The banana plants (variety *Caipira*) were planted in planting holes (0.4 x 0.4 x 0.6 m) with a spacing of 3.0 x 2.0 m in May 2001. The planting holes were prepared 30 days before

planting and filled with a mixture of soil, charcoal (0, 8 and 16 litre), chicken manure (5 litre), OSP (200, 300 and 400 g) and lime (200 g). Urea (0, 200, 400 g) was fertilized on the soil surface. The application of urea, simple super phosphate and charcoal was repeated on the soil surface (chapter X, table 10-1). Furthermore the plants were fertilized with KCl (200 g in January 03 and March 2004), zinc sulphate ( $ZnSO_4$ , 50 g in October 2002) and FTE BR 12 (micronutrient mix, 50 g in February 2004). These combinations of three doses of charcoal, P and N form 27 treatments consisting of 162 plants (6 plants each). The guarana plantation covers 4 ha with 1604 plants. From October to December the area was cleared and the plants were planted in March 2003. As the intention of the experiment was to produce organic guarana, weed and pest control was done without pesticides. Fertilization was restricted to organic amendments only. In July 2003 ground charcoal was applied to the soil surface. Bone meal was applied in August 2003 and chicken manure in October 2003 (chapter X, table 10-1). Charcoal, bone meal and chicken manure were applied in three different doses allowing the formation of 27 different treatment combinations, each treatment consisting of 6 guarana plants. The entire experiment was established in five repetitions with five different varieties of guarana. For soil respiration curves only one variety (*Maués*) was chosen.

Four different treatments were applied in the local farming context (chapter XI) and designated as: (a) Normal agricultural practice (NAP); (b) NAP + 6.5 litre (~2.2 kg) of charcoal powder; (c) NAP + 6.5 litre charcoal in small pieces (sieved to obtain a size between 0.2 and 1 cm); and, (d) NAP + 13 litre of charcoal mix (available as charcoal production waste from local charcoal producers). The experiment was designed as completely randomized with four repetitions of each treatment. The NAP is to plant bananas in planting holes 0.3 m deep and 0.45 m wide. The holes are filled with a mixture of 10 litre fresh chicken manure, 500g powdered lime, 50g micronutrient-mix (4.5 g Zn, 0.9 g B, 0.4 g Cu, 1.5 g Fe, 1.0 g Mn, 0.05 g Mo), 300g OSP (23.22 g P) and soil. The other treatments received an additional charcoal amendment. The holes were established in lines with a distance of 2.5 m between the holes and 3.5 m between the lines. Until harvest (February 26<sup>th</sup> to March 7<sup>th</sup> 2003) two further fertilizations were applied on the soil surface. In April 2002 60 g magnesium sulphate ( $MgSO_4$ ), 80 g KCl, 39 g  $ZnSO_4$ , 30g FTE BR12 (micronutrients), 30 g Borax, 90 g  $(NH_4)_2SO_4$  and 60 g OSP were applied. In July 2002 270 g KCl and 135  $NH_4SO_4$  were applied. On 22<sup>nd</sup> of April 2002 the bananas were planted as small clones (0.1 to 0.2 m plant size) of the variety *Caipira*. Six banana plants were planted per treatment in 4 repetitions.

### *Soil Samples*

In order to study indigenous soil management (chapter I) soil samples were taken at seven different locations (0 – 0.2 m depth) with a small shovel and placed in plastic bags. The locations were chosen according to the soil management (chapter I, table 1-1). The various forms included burned soil termed *Terra Queimada* (TQ), *Terra Queimada* with burned organic amendments (TQ + bOM), soil called *Terra Preta* or *Terra Preta Nova* (TPn), newly burned soil termed *Terra Cheirosa* (TC), and unmanaged background soil was taken in proximity of the football ground (BS). The samples from the field experiment (0 – 0.1, 0.1 – 0.3 and 0.3 – 0.6 m depth) were taken after the 4 consecutive harvests (chapters VI, VII, and VIII). Top soil (0 – 0.1 m) samples were taken on April 8<sup>th</sup> 2004 in the banana plantation and April 23<sup>rd</sup> 2004 in the guarana plantation (chapter X). One composite sample was formed out of 4 sub-samples taken in the fertilized area. The first and sixth plant was not sampled to minimize the influence of adjacent treatments. In the banana plantation (chapter XI) samples were taken from the planting holes (0 – 0.2, and 0.2 – 0.4 m depth). Immediately after sampling the soil was treated and analyzed. Samples from the pot experiment (chapter V) were analyzed after harvesting the plant biomass.

For the extraction of exchangeable nutrients the Mehlich-3 (Mehlich 1984; chapters V, and VI) or Mehlich 1 extraction (EMBRAPA standard, Claessen *et al.* 1997); chapters I, X and XI) were used. We analyzed the soil samples for N, P, K, sodium (Na), Ca, magnesium (Mg), aluminium (Al), acidity (Al+H), iron (Fe), zinc (Zn), manganese (Mn) and copper (Cu). Plant available P, K, Na, Zn, Mn, and Cu was analyzed in Mehlich 1 or 3 extracts and Mg, Ca, and Al in KCl extracts. The micro-nutrients (Fe, Zn, Mn and Cu), Ca and Mg were determined by atomic absorption spectrometry (GBL Avanta  $\Sigma$  Analitica, Australia, AA-1475 or Varian Associates, Inc., Palo Alto, CA). Exchangeable acidity and exchangeable Al were determined by titration (McLean 1965) after extraction with 1 N KCl and P was measured using a photometer (Helios  $\beta$ , Thermo Spectronic, Cambridge, UK) with the molybdene blue method (Olsen and Sommers 1982). Potassium was analyzed with a flame photometer (Micronal B 262, Sao Paulo, Brazil). pH was determined in water and 1 N KCl (1:5 w/v) using an electronic pH meter with a glass electrode (WTW pH 330, WTW, Weilheim, Germany) and conductivity was measured (HI 8733, HANNA Instruments). Total C and N were analyzed by dry combustion with an automatic C/N- Analyzer (Elementar, Hanau, Germany). Plant- available  $\text{NH}_4$  and  $\text{NO}_3$  (chapter VI) were determined photometrically in soil extracts (in KCl) using a rapid flow analyzer (Scan Plus analyzer, Skalar Analytical B.V., Breda, The Netherlands). Cation exchange capacity (CEC) was calculated as the sum of ammonium acetate-exchangeable cations and acidity (Claessen *et al.* 1997).

Before the total elemental determination (chapter I) large inclusions (organics, stones, ceramics, etc.) were removed from each sample by hand picking. The samples were then ground with a blender and materials larger than 2.0 mm screened out. The extraction was with concentrated  $\text{HNO}_3$  and 6.0 M HCl. The elements in the resulting aliquot were determined by ICP (IAP Solid State Spectrograph, Thermo Electron Corporation).

#### *Plant Samples*

For the determination of foliar nutrient contents a digestion with a mixture of  $\text{H}_2\text{SO}_4$ , salicylic acid,  $\text{H}_2\text{O}_2$  and selenium was used according to Walinga (1995). N was analyzed using the method of Kjeldahl and titration or by dry combustion with an automatic C/N- Analyzer (Elementar, Hanau, Germany). The elements P, K, Ca, Mg, sulphur (S), boron (B), Cu, Fe, Mn, and Zn (chapter XI) were analyzed according to (Malavolta *et al.* 1997).

#### *Soil Solution*

The treatments NAP and NAP+charcoal-mix (chapter XI) were established with 6 suction cups each. The suction cups were installed in an angle to be situated in 0.5 m depth (~0.2 m beneath the planting hole) for soil solution collection. Soil solution was taken on May 3<sup>rd</sup>, June 22<sup>nd</sup> and July 3<sup>rd</sup> 2002 for analysis of pH, conductivity, Mg, Ca and K. Ca and Mg were determined by atomic absorption spectrometry (GBL Avanta  $\Sigma$  Analitica, Australia) and K was analyzed with a flame photometer (Micronal B 262, Sao Paulo, Brazil). The pH (WTW pH 330, WTW, Weilheim, Germany) and conductivity (HI 8733, HANNA Instruments) were determined in the solution. Leached N contents were measured by the Kjeldahl technique (chapter V).

#### *$^{15}\text{N}$ Tracer Application*

Five treatments were chosen for  $^{15}\text{N}$  isotope enrichment using  $^{15}\text{N}$  labelled  $(\text{NH}_4)_2\text{SO}_4$  with 10 atom %  $^{15}\text{N}$  excess (chapter VIII). The tracer was mixed in a ratio 1:1 with conventional  $(\text{NH}_4)_2\text{SO}_4$  and applied at a rate of 55 kg N ha<sup>-1</sup> in April 2002 (chapter VIII, table 8-1). Soil and plant samples were taken at each harvest and analyzed for  $\delta^{15}\text{N}$ . Only the top 0.1 m of soil was sampled, this was also the depth into which the organic amendments were mixed. Two soil samples were taken per plot to form one composite sample. The labelled  $^{15}\text{N}$  remaining in soil or found in plant biomass was calculated after equation (1).