

A Study of Sewage Sludge Composting, Utilization of Compost  
and Nitrogen Dynamics in Plant–Soil System

September 2017

NGUYEN THANH BINH

Graduate School of Environmental and Life Science

(Doctor course)

OKAYAMA UNIVERSITY, JAPAN

# A Study of Sewage Sludge Composting, Utilization of Compost and Nitrogen Dynamics in Plant–Soil System

A thesis submitted by NGUYEN THANH BINH  
in partial fulfillment of the requirements for degree of

Doctor of philosophy

Graduate School of Environmental and Life Science  
(Doctor course)

OKAYAMA UNIVERSITY, JAPAN

September 2017

## Declaration

It is hereby certified that the work described in this thesis has been carried out by the candidate NGUYEN THANH BINH at Graduate School of Environmental and Life Science, Okayama University, Japan. It has not, in whole or in part, been submitted for any other degree. This dissertation was accepted for the award of the Doctor of Philosophy at Okayama University, Japan.

.....

Professor KAZUTO SHIMA

Supervisor

Graduate School of Environmental and Life Science

Okayama University, Japan

## Preface

The work presented in this doctoral dissertation was conducted at Division of Environmental Science, Graduate School of Environmental and Life Science, Okayama University, Japan from April 2014 to July 2017. The preliminary experiments were done during master course time from 2011 to 2012.

This dissertation is based on the prepared reports:

1. Binh, N., Quynh, H. and Shima, K. (2015) Effect of composts combined with chemical N fertilizer on nitrogen uptake by Italian Ryegrass and N transformation in soil. *Journal of Agricultural Chemistry and Environment*, 4, 37-47. <http://dx.doi.org/10.4236/jacen.2015.42004>.
2. Nguyen Thanh Binh and Kazuto Shima (2017). Composting of sewage sludge with a simple aeration method and its utilization as a soil fertilizer. *Environmental Management* journal (under review).
3. Nguyen Thanh Binh and Kazuto Shima (2017). Nitrogen mineralization in soil amended with compost and urea as affected by plant residues supplements with controlled C/N ratios. *Journal of Advanced Agricultural Techniques* (acceptance).
4. Nguyen Thanh Binh and Kazuto Shima (2017). Nitrogen mineralization and N uptake by Komatsuna from N-fertilized soil as affected by bamboo stem powder addition with controlled C/N ratios. Manuscript under preparation.
5. Nguyen Thanh Binh and Kazuto Shima (2017). Effect of volumetric ratios of sludge and woodchips on physicochemical properties during composting process. Manuscript under preparation.
6. Nguyen Thanh Binh and Kazuto Shima (2017). Fate of  $^{15}\text{N}$  derived from sewage sludge compost under modeling plant–soil systems in a pot–culture experiment. Manuscript under preparation.

## Structure of the work

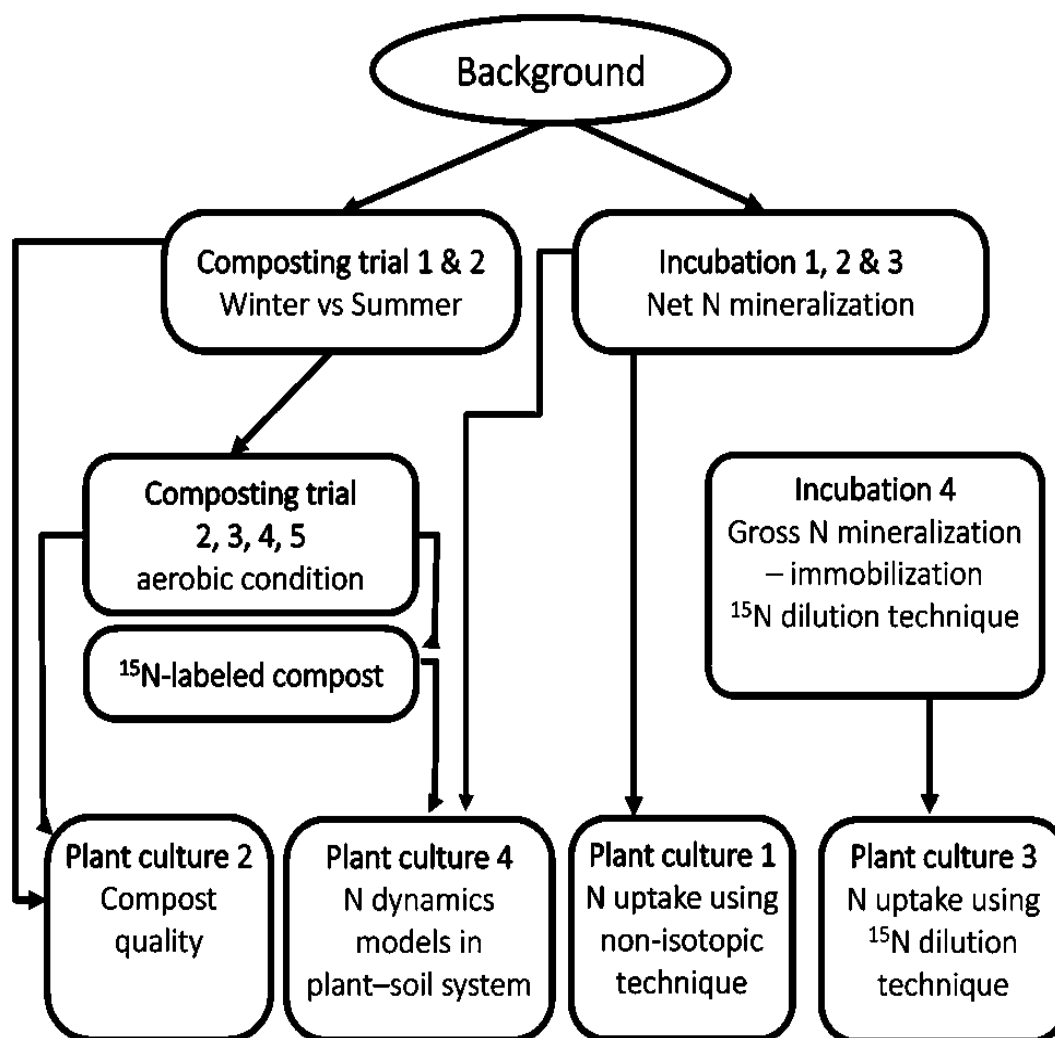


Fig. 1.0. The relationships of between experiments and their primary tasks

## Acknowledgements

First and foremost, I would like to express my gratitude to my supervisor, Professor Kazuto Shima, for accepting me as his student, helping and advising me during my studying in Japan. My appreciation also goes to my co-advisors, Professor Keiji Sakamoto and Professor Muneto Hirobe, for their expertise during my work.

Thanks to all my labmates for their help, feedback, and encouragement during long working hours in our laboratory.

The author is grateful to Japanese Ministry of Education, Culture, Sports, Science, and Technology (MEXT) for the financial support that made this research possible.

Sincere gratitude to my friends and colleagues, particularly in the Graduate School of Environmental and Life Science, Okayama University, Faculty of Agronomy, Nong Lam University in Ho Chi Minh city and the Okayama-Hue International Master's program for their kindness and support me throughout the graduate program.

Finally, this dissertation is devoted to my parents, my wife and my daughter, to whom I sincerely grateful for their continuous support, patience, and encouragement throughout my period of study.

.....

September 2017

Nguyen Thanh Binh

## Abstract

The disposal of sewage sludge (SS) in landfills has caused serious pollution problems in developing countries. This has increased the demand for recycling of SS into valuable products. Composting has been considered as an attractive option for effective reduction of waste volume to an organic soil fertiliser. However, the quality of compost and the effects of refuse compost application on the plant–soil system are not well understood in humid tropical regions. The main objectives of this study were to determine the effects of composting on physicochemical properties of SS and to utilise compost as an organic fertiliser, with special attention to nitrogen (N) dynamics in the plant–soil system.

Five small-scale composting trials were carried out to examine the feasibility of composting using SS and woodchips as bulking agents under different seasonal temperatures and aerobic conditions. SS from one of the five composting trials was labelled with  $(^{15}\text{NH}_4)_2\text{SO}_4$  (99.74 atom%) to produce artificially-enriched compost for use in further experiments. Physicochemical parameters, including temperature, moisture content, pH, carbon content, nutrient content and heavy metal content, were measured to determine the changes in SS during composting. Better results of composting were achieved in summer than in winter, with more woodchips being used, suggesting a strategy for controlling temperature and aeration. The compost temperatures were maintained above 55°C for three consecutive days when SS was mixed with woodchips at ratios of 1:1 and 1:0.5 (v/v). The overall decrease in free amino acid N (FAAN) at the end of the process corresponded to the stability of other physicochemical properties, which can be regarded as an indicator of compost maturity. However, the finished composts had high total N (TN) contents and low C/N ratios, predicting rapid N mineralisation.

Three 20-day soil incubations were conducted under laboratory conditions to study the availability of N from compost and determine the effect of addition of chemical N fertilisers (CF) and different carbon sources [powder of bamboo stem (BS), wood for building (WB) and rice straw (RS)] on the N availability. The changes in inorganic N ( $\text{NH}_4$  and  $\text{NO}_3$ ) concentrations with incubation were measured, and net N mineralisation rates were estimated. It should be noted that nitrification was predominant in soil amended with compost. The quality of added carbon may be an important factor which controls N mineralisation in soil. The compost with higher CF supplements resulted in an increase in soil N mineralisation, whereas that with BS supplements resulted in a significant decrease in soil N mineralisation due to temporary immobilisation of nitrate N.

To compare the results obtained from initial composting trials and further incubation studies, two pot-culture experiments were subsequently set up under greenhouse conditions. In the first experiment, Komatsuna (*Brassica rapa* var. perviridis) was treated under similar conditions as those used in previous incubation studies to correlate the net N mineralisation rates with plant N uptake. A significant positive relationship between net N mineralisation rate and total plant N uptake may explain the availability of N

from compost and CF. However, mineralisation and immobilisation turnovers can simultaneously occur, making it more difficult to estimate the contribution of N from each source. In the second experiment, the quality of two self-made composts produced during winter and summer were compared to that of raw materials and CF as control treatments on the yield of Komatsuna. The results showed that the effects of two composts and winter SS were comparable to that of CF but were significant higher plant yield than that of summer SS. Thus, decreasing ratios of FAAN/TN, C/N and  $\text{NH}_4/\text{NO}_3$  in composts compared with those in SS were found to correspond to the increase in plant yield, which may reduce the phytotoxicity of the raw material.

By using  $^{15}\text{N}$  isotope labelling techniques, the gross N mineralisation–immobilisation in soil was measured to distinguish between N contributions from compost and CF. In a 10-day incubation study,  $(^{15}\text{NH}_4)_2\text{SO}_4$  (13.172 atom%) was added to unlabelled compost using  $^{15}\text{N}$  pool dilution technique. Gross N immobilisation rates varied from 16.08 to 29.62 mg N  $\text{kg}^{-1}$  soil  $\text{day}^{-1}$ , while only 0.40–0.66 mg N  $\text{kg}^{-1}$  soil  $\text{day}^{-1}$  from the organic pool was mineralised to the inorganic pool. In another pot-culture experiment using the same approach, approximately one-third of the total plant N uptake was derived from compost when treated with compost and CF at a ratio of 1:1. However, this method may have led to problems associated with overestimation of gross N transformation due to a pool substitution effect or an added N interaction.

In another improved approach, the  $^{15}\text{N}$ -labelled compost (0.639 atom%) obtained from the aforementioned composting trial was used as a tracer, and three N dynamic models called control (CTRL), non-RS addition (NRSA) and RS addition (RSA) were developed to quantify plant N uptake, N retention in soil and N loss from different N sources. For CTRL, where either compost or CF were applied to the soil, N uptake from CF was 1.6-fold higher than that from compost, but half of the total applied N was lost by N leaching. For NRSA, a combination of compost and CF in the ratio of 1:1 reduced N leaching by 2.5-folds while maintaining similar N uptake from CF alone, in which 56% of the TN uptake was derived from compost. The incorporation of RS into compost and CF in the RSA model resulted in the highest retention of N in the surface soil.

These results suggest a simple aerobic composting technique for recycling of SS and assess the compost quality based on bioassays which evaluate the changes in temperature, N availability and plant growth. N mineralisation in composts can be controlled by combining them with CFs and/or bioavailable carbon sources, which can modify their C/N ratios. Labelling the compost with  $^{15}\text{N}$  permitted more accurate estimation of plant N uptake, N retention in soil and N leaching. However, further in situ experiments using  $^{15}\text{N}$ -labelled composts are required to evaluate the effectiveness of these models.



Contents	
Preface .....	i
Structure of the work.....	ii
Acknowledgements .....	iii
Abstract.....	iv
List of Tables .....	viii
List of Figures .....	ix
List of Abbreviations .....	xi
Chapter I: Introduction .....	1
1.1. General introduction .....	1
1.2. Objectives .....	3
1.3. Outline of the dissertation .....	4
References .....	5
Chapter 2: Literature review .....	7
2.1. Sewage sludge regeneration from waste water treatment plant .....	7
2.2. Sewage sludge treatment options.....	8
2.3. Status for use of sewage sludge in Vietnam and Japan .....	9
2.3.1. Vietnam .....	9
2.3.2. Japan .....	10
2.4. Nutrient and heavy metal contents of sewage sludge .....	10
2.5. Composting process.....	11
2.5.1. Concepts and purpose .....	11
2.5.2. Methods of composting .....	12
2.5.3. Composting process .....	13
2.5.4. Composting process management .....	14
2.6. Utilization of SS compost.....	15
2.6.1. Effects on crop production and soil properties.....	15
2.6.2. Effects on heavy metal accumulation .....	16
2.7. Dynamics of compost N in the plant–soil system and introduction of stable N isotope .....	17
2.7.1. N mineralisation and immobilisation.....	18
2.7.2. N uptake by plant .....	19
2.7.3. N retention in soil and N losses .....	20
References .....	21
Chapter 3: Composting process .....	25
3.1. Introduction .....	25
3.2. Materials and methods .....	26
3.2.1. Composting runs 1 and 2 .....	26
3.2.2. Follow-up composting runs 3, 4 and 5 .....	28

3.3. Results and discussion.....	31
3.3.1. Effect of seasonal temperature on composting performance .....	31
3.3.2. Effect of volumetric ratios of sludge and woodchips ratios .....	37
3.4. Conclusions .....	47
References .....	48
4.1. Introduction .....	51
4.2. Materials and methods .....	51
4.2.1. Preparation for incubation 1, 2 and 3.....	51
4.2.2. Soil analyses .....	52
4.2.3. Calculation and statistical analysis.....	52
4.3. Results and discussion.....	56
4.3.1. Effects of different carbon sources addition on soil N mineralisation–immobilisation.....	56
4.3.2. Effects of BS with controlled C/N ratios on soil N mineralisation- immobilisation .....	56
4.3.3. Effect of BS addition on soil N mineralisation when compost was combined with urea .....	62
4.4. Conclusions .....	66
References .....	67
Chapter 5: Effectiveness of compost as soil fertilizer .....	69
5.1. Introduction .....	69
5.2. Materials and methods .....	69
5.2.1. Preparation for pot-culture experiment 1 and 2 .....	69
5.2.2. Soil–plant analysis and calculation .....	70
5.2.3. Statistical tests.....	71
5.3. Results and discussion.....	72
5.3.1. Pot-culture experiment 1 .....	72
5.3.2. Pot-culture experiment 2 .....	74
References .....	81
Chapter 6: Nitrogen dynamics in plant-soil system .....	83
6.1. Introduction .....	83
6.2. Materials and methods .....	84
6.2.1. Initial experiments using $^{15}\text{N}$ labelled $(\text{NH}_4)_2\text{SO}_4$ .....	84
6.2.2. Later experiments on $^{15}\text{N}$ labelled compost .....	87
6.3. Results and discussion.....	89
6.3.1. The experiments using $^{15}\text{N}$ labelled $(\text{NH}_4)_2\text{SO}_4$ .....	89
6.3.2. The experiments using $^{15}\text{N}$ -labelled compost.....	92
6.4. Conclusions .....	102
References .....	103
Chapter 7. Conclusion.....	105
Appendix A.....	107

## List of Tables

Table 2.1. Average macronutrient content in sewage sludge of some countries

Table 2.2. Regulatory standards of heavy metals in sewage sludge of some countries

Table 3.1. Physicochemical properties of Japanese SS for composting runs 1–5 and the reference data

Table 3.2. Changes in physicochemical properties during composting in runs 1 and 2

Table 3.3. Changes in total phosphorus, total potassium, total calcium and magnesium as affected by sludge: woodchips recipes

Table 3.4. Changes in total heavy metals (Cu, Zn, Mn, Pb and Cd) as affected by sludge: woodchips recipes

Table 3.5. Correlation coefficients among physicochemical parameters of composts

Table 4.1. Chemical properties of materials used for incubations 1, 2 and 3

Table 4.2. Treatment combinations and weight of ingredients in incubations 1 and 2

Table 4.3. Treatment combinations and weight of ingredients in incubation 3

Table 4.4. Soil inorganic N as affected by compost and carbon sources addition at controlled C/N = 25

Table 4.5. Soil net Ammonification rate, net nitrification rate and net N mineralization rate as affected by compost and carbon sources addition at controlled C/N = 25

Table 4.6. Net ammonification, net nitrification and net N mineralisation rates of N-fertilized soil as affected by BS addition with controlled C/N=10 and C/N=25

Table 4.7. Net ammonification, net nitrification and net N mineralisation rates of N-fertilized soil as affected by BS addition with controlled C/N=25

Table 5.1. Physicochemical properties of composts and soil in the pot-culture experiment 2

Table 5.2. Treatment combinations and weight of ingredients in the pot-culture experiment 2

Table 6.1. Chemical properties of composts in incubation 4 and pot-culture experiment 3

Table 6.2. Treatment combinations and weight of ingredients in the incubation 4 and the pot-culture experiment 3

Table 6.3. Treatment combinations and weight of ingredients in pot-culture experiment 4

Table 6.4. Gross total N transformation and net N mineralisation rates as affected by temperature and inorganic-N addition to compost

Table 6.5. Total plant nitrogen uptake and partitioning of total plant N derived from compost, CF, and soil as affected by different treatments

Table 6.6. Leaf, root and total plant dry matter as affected by N sources and rice straw addition

Table 6.7. Leaf, root and total plant N uptake as affected by N sources and rice straw addition

Table 6.8. Leaf, root and total plant N uptake derived from compost and CF

Table 6.9. Total soil N retention in the surface soil derived from compost and CF

Table 6.10. Leaching/loss of nitrogen from the surface soil derived from compost and CF

## List of Figures

- Fig. 1.0. The relationships of between experiments and their primary tasks
- Fig. 1.1. Simplified compost-N dynamics in plant–soil system and research questions
- Fig. 2.1. Schematic representation of sludge transformation from wastewater to disposal
- Fig. 2.2. Overview of possible treatment and disposal systems for sewage sludge
- Fig. 2.3. Status of sludge management in Japan
- Fig. 2.4. Classification of composting systems
- Fig. 2.5. A basic schematic of an aerobic composting process
- Fig. 2.6. Simplified N dynamics in plant–soil system
- Fig. 2.7. Levels of nitrogen available to plants based on microbial decomposition
- Fig. 3.1. Schematic diagram of the composting run 1
- Fig. 3.2. Schematic diagram of the composting run 2, 3, 4 and 5
- Fig. 3.3. Temperature profiles at the top, middle, bottom of the compost and ambient for run 1 and run 2
- Fig. 3.4. Changes in nitrogen forms during composting process
- Fig. 3.5. Changes in temperature (A), moisture (B), pH (C), volatile solid (D), organic matter loss (E) and total nitrogen loss (F) as affected by sludge: woodchips recipes
- Fig. 3.6. Changes in total carbon (A), total nitrogen (B), C/N ratio (C), free amino acid-N (D),  $\text{NH}_4\text{-N}$  (E) and  $\text{NO}_3\text{-N}$  (F) as affected by sludge: woodchips recipes
- Fig. 4.1. Changes in inorganic nitrogen in paddy soil as affected by BS addition
- Fig. 4.2. The relationship between net ammonification, net nitrification, net N mineralisation rates and amount of BS addition
- Fig. 4.3. Changes in inorganic nitrogen as affected by BS addition with controlled C/N= 10 and C/N=25 when either compost or urea was applied to soil at rate of  $100 \text{ mg kg}^{-1}$  soil
- Fig. 4.4. Changes in inorganic N as affected by BS addition with controlled C/N=25 when compost was combined with urea and applied to soil at rate of  $50 \text{ mg kg}^{-1}$  soil
- Fig. 4.5. Changes in inorganic N as affected by BS addition with controlled C/N=25 when compost was combined with urea and applied to soil at rate of  $150 \text{ mg kg}^{-1}$  soil
- Fig. 5.1. Total plant N uptake when compost and urea were applied to soil as affected by BS addition with controlled C/N=25
- Fig. 5.2. Relationship between soil net ammonification rate, net nitrification rate, net N mineralisation rate and total plant N uptake as affected by BS addition with controlled C/N=25
- Fig. 5.3. Leaf length as affected by CF application rate
- Fig. 5.4. Plant dry matter as affected by CF application rate

Fig. 5.5. Leaf length as affected by sludges and composts application at rate of 600 mg N pot<sup>-1</sup>

Fig. 5.6. Plant dry matter as affected by sludges and composts application at rate of 600 mg N pot<sup>-1</sup>

Fig. 5.7. Relationship between total plant dry matter and FAAN/TN (A), NH<sub>4</sub>-N/NO<sub>3</sub>-N (B), C/N ratio (C) of amendments

Fig. 5.8. Concentrations of P, K in leaf (A) and plant P, K uptake (B) as affected by sludges and composts application at rate of 600 mg N pot<sup>-1</sup>

Fig. 5.9. Concentrations of Cu and Zn in leaf as affected by sludges and composts application at rate of 600 mg N pot<sup>-1</sup>

Fig. 6.1. <sup>15</sup>N atom percent in pools of total N and organic during composting process after labelling with (<sup>15</sup>NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> at the beginning of the experiment

Fig. 6.2. The distribution of applied N in plant–soil system as affected by N sources and rice straw addition

Fig. 6.3. N dynamics in plant–soil system as affected by the single application of CF and compost

Fig. 6.4. N dynamics in plant–soil system as affected by the combined application of CF and compost in the absence of RS

Fig. 6.5. N dynamics in plant–soil system as affected by the combined application of CF and compost in the presence of RS

Fig. A1. An overview of aerobic composting process

Fig. A2. View of composting systems in summer (left) and winter (right), from outside (above) and inside (below) of reactors

Fig. A3. View of an incubation study

Fig. A4. View of the pot-culture experiment

Fig. A5. Visible response of Komatsuna to composts and sludges

Fig. A6. Visible response of Komatsuna to compost and ammonium sulfate at 38 days after sowing

Fig. A7. Visible response of Komatsuna to compost and ammonium sulfate with and without rice straw addition

## List of Abbreviations

SS	Sewage sludge
BS	Bamboo stem
CF	Chemical N fertiliser
CTRL	Control model
DAS	Days after sowing
FAAN	free amino acid nitrogen
MIT	Mineralisation–Immobilisation turnover
MLIT	Japanese Ministry of Land, Infrastructure, Transport and Tourism
NRSA	Non-rice straw addition model
NUE	Nitrogen uptake efficiency
RS	Rice straw
RSA	Rice straw addition model
TNU	Total nitrogen uptake
WB	Wood for building
WWTP	Wastewater treatment plant

# Chapter I: Introduction

## 1.1. General introduction

SS (SS) or “bio-solids” is an inevitable by-product of urban wastewater treatment. The disposal of SS into landfills has been causing serious environmental pollution in developing countries. In Vietnam, wastewater treatment plants (WWTPs) were estimated to generate approximately 1.2 million dry Mg of SS annually (Karius, 2011). These numbers are predicted to increase dramatically in the coming years due to rapid urbanization and development of sewage networks. At present, the most commonly used sludge disposal system in Vietnam is dumping at sanitary landfills without recovery of the recyclable materials in spite of the increasing awareness of its environmental pollution. In contrast, Japan, before the great east earthquake, generated more than 2.2 million dry Mg of SS. Remarkably, nearly 80% of SS is reused and recycled as construction materials, compost, fuel, etc. (MLIT, 2011).

Composting of organic waste has been considered an attractive sludge management option for effective reduction of its volume to serve as a soil organic fertiliser. Composted sludge provides much needed organic matter and excellent plant nutrients like nitrogen, phosphorus, copper and zinc to plants, improving soil physical properties and water holding capacity (Warman and Termeer 2005; Pedra et al. 2007; Alvarenga et al. 2015). However, negative effects associated with the accumulation of heavy metals, toxicants and pathogens in plant-soil systems should be taken into consideration (Tan 2000).

The basic principles of the thermophilic composting processes are well understood. It is known to be an aerobic decomposition process of organic matter which changes with increasing and decreasing temperature to produce a finished compost. However, the complex composition of SS, of which the changes in physicochemical properties occurred during the composting process, make the assessment of compost quality a difficult task (Itävaara et al., 2002). In addition, free amino acid nitrogen (FAAN), an easily decomposable organic N, remains after composting which will be a very important N source for plants (Jones and Kielland, 2002). Although there have been several publications on the behavior of nitrogen from SS, the fate of FAAN during the composting process has received very little attention.

To study the availability of N release from compost, the simplest method involves incubation studies which unlabelled compost-N is measured as the difference in the inorganic N, and in that way, the net N mineralisation rates are estimated (Chalk et al., 2013). The same approach can be applied for pot-culture experiments where the N availability derived from compost could be estimated as the difference in total nitrogen uptake by plant between treatments in the presence and absence of added compost. Nevertheless, one of the most common problems associated with utilization of compost in advanced agricultural technologies is the risk of nitrate leaching into the groundwater through the application of fast-release N-fertilisers (Insam and Merschak, 1997; Łuczkiwicz and Quant, 2007). Chemical N

fertilisers are used as a supplementary N source for the plant in both traditional and modern agriculture due to the low N supplying potential of compost. As mentioned above, the non-isotopic studies have attempted to quantify the N availability from compost and chemical N fertiliser. Unfortunately, the mineralisation–immobilisation turnover (MIT) processes can occur simultaneously, making it more difficult to estimate the contribution of N from each source.

Alternately, the introduction of  $^{15}\text{N}$  isotope labeling has proven very advantageous in tracing the fate of applied  $^{15}\text{N}$  at several scales, from pot experiments to landscapes (Wolf et al., 1994; Bedard-Haughn et al., 2003; Luo et al., 2004). Yet one of the major problems associated with using  $^{15}\text{N}$  isotope techniques is the overestimation or underestimation of the true N availability derived from composted materials that caused by the effects of pool substitution or an added nitrogen interaction (ANI) (Hood et al., 1999). To overcome these limitations, several researchers have successfully applied artificially enriched tracer to studied the dynamics of compost N in the plant–soil system as reviewed by Chalk et al. (2013). However, the information on the soil nitrogen dynamics under humid tropical regions; particularly when compost, chemical N fertiliser and plant residues are simultaneously applied to soil, is very scarce. In addition, to our knowledge, the models of N uptake, N retention in soil and N losses derived from SS-based composts have not been specifically addressed in the literature.



## 1.2. Objectives

The main objectives of this study were to determine the effects of composting on physicochemical properties of SS and to utilise compost as an organic fertiliser with special attention to N dynamics in the plant–soil system.

The specific questions addressed were the following:

- ① How did the sludge properties change during composting?
- ② How did the compost and sludge affect the plant productivity?
- ③ How to regulate the N mineralisation from compost after soil application?
- ④ How did the compost-N dynamics in plant–soil system occur?

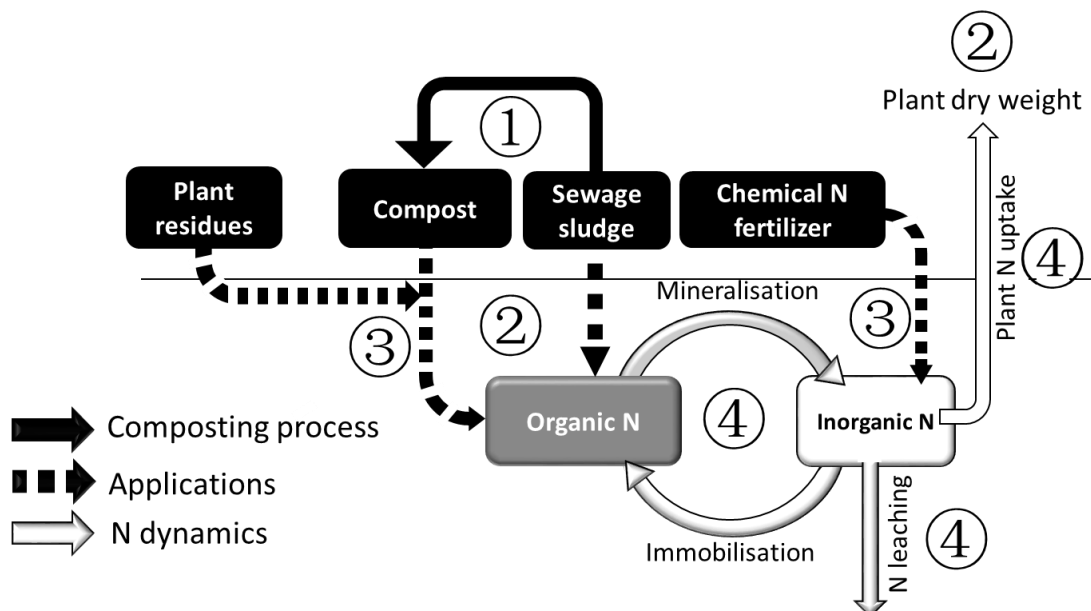


Fig. 1.1. Simplified compost-N dynamics in plant–soil system and research questions

### 1.3. Outline of the dissertation

This dissertation was presented semantically (i) a technique for recycling of SS as compost using a simple aeration method (ii) the utilization of compost as a soil fertiliser with special attention to dynamics of compost N in plant–soil system using pool dilution and  $^{15}\text{N}$  tracer approaches. Chapter 1 introduces the importance of composting in the reduction of waste volume, the benefit of compost application on soil with special attention to nitrogen availability. Status for use of SS in Vietnam and Japan is also addressed. This section also states the objective of our research and how it fits into the gap in knowledge. Chapter 2 provides a concise overview of the SS generation, sludge properties, composting process, methods used to evaluate the quality of compost. The nitrogen dynamics in plant–soil system were also intensively reviewed. Chapter 3 provides the results of five small-scale composting trials running in the winter and in the summer using different volumetric sludge/woodchips ratios. The measurement of amino acid nitrogen may add the novelty for checking the stability of the process. In Chapter 4, net ammonification, net nitrification and net N mineralisation were estimated by the difference in the inorganic N ( $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$ ) after 10 and 20 days of incubation under controlled laboratory conditions. The effects of different carbon sources and chemical N fertiliser as supplements to compost were also compared. The results from the incubation studies were consistent with those in the pot-culture experiment as reported in the first part of Chapter 5. In the second part of this chapter, the effectiveness of compost on plant was comparable to CF and higher than its raw material. In Chapter 6, gross N mineralisation, gross N immobilisation and proportion of plant nitrogen uptake from compost were initially estimated using pool dilution approach. In the last experiment,  $^{15}\text{N}$ -labelled compost was used as a tracer approach to calculate directly the N dynamics in plant–soil system. The final conclusions and some recommendations are summarized in Chapter 7.

## References

- Alvarenga, P., C. Mourinha, M. Farto, T. Santos, P. Palma, J. Sengo, M.C. Morais, and C. Cunha-Queda. 2015. Sewage sludge, compost and other representative organic wastes as agricultural soil amendments: Benefits versus limiting factors. *Waste Manag.* 40: 44–52.
- Bedard-Haughn, A., J.W. Van Groenigen, and C. Van Kessel. 2003. Tracing  $^{15}\text{N}$  through landscapes : potential uses and precautions. *J. Hydrol.* 272: 175–190.
- Chalk, P.M., A.M.T. Magalhães, and C.T. Inácio. 2013. Towards an understanding of the dynamics of compost N in the soil-plant-atmosphere system using  $^{15}\text{N}$  tracer. *Plant Soil* 362(1–2): 373–388.
- Hood, R.C., K.N. Goran, M. Aigner, and G. Hardarson. 1999. A comparison of direct and indirect  $^{15}\text{N}$  isotope techniques for estimating crop N uptake from organic residues. *Plant Soil* 208: 259–270.
- Insam, H., and P. Merschak. 1997. Nitrogen leaching from forest soil cores after amending organic recycling products and fertilizers. *Waste Manag. Res.* 15(3): 277–291.
- Itävaara, M., O. Venelampi, M. Vikman, and A. Kapanen. 2002. Compost Maturity — Problems Associated with Testing BT - Microbiology of Composting. p. 373–382. *In* Insam, H., Riddech, N., Klammer, S. (eds.), Springer Berlin Heidelberg, Berlin, Heidelberg.
- Jones, D.L., and K. Kielland. 2002. Soil amino acid turnover dominates the nitrogen flux in permafrost-dominated taiga forest soils. *Soil Biol. Biochem.* 34(2): 209–219.
- Karius, R. 2011. Developing an integrated concept for sewage sludge treatment and disposal from municipal wastewater treatment systems in (peri-) urban areas in Vietnam [Diploma thesis]. Technische Universität Dresden.
- Łuczkiwicz, a, and B. Quant. 2007. Soil and Groundwater Fecal Contamination as a Result of Sewage Sludge Land Application. *Polish J. Environ. Stud.* 16(4): 587–593.
- Luo, L., M. Kondo, and S. Itoh. 2004. Fate of  $^{15}\text{N}$  derived from composts and urea in soils under different long-term N management in pot experiments. *Compost Sci. Util.* 12(October 2014): 18–24
- MLIT. 2011. Status for the Use of Sewage Sludge in Japan. Japanese Minist. Land, Infrastructure, Transp. Tour.
- Pedra, F., A. Polo, A. Ribeiro, and H. Domingues. 2007. Effects of municipal solid waste compost and sewage sludge on mineralization of soil organic matter. *Soil Biol. Biochem.* 39(6): 1375–1382.
- Tan, K.H. 2000. Environmental Soil Science, Third Edition. CRC Press, UK.
- Walter, I., F. Martí'nez, and V. Cala. 2006. Heavy metal speciation and phytotoxic effects of three representative sewage sludges for agricultural uses. *Environ. Pollut.* 139(3): 507–514.
- Warman, P.R., and W.C. Termeer. 2005. Evaluation of sewage sludge, septic waste and sludge compost applications to corn and forage: Yields and N, P and K content of crops and soils. *Bioresour. Technol.* 96(8): 955–961.

Wolf, D.C., J.O. Legg, and T.W. Boutton. 1994. Isotopic Methods for the Study of Soil Organic Matter Dynamics. In: Weaver, R.W., Angle, S., Bottomly, P., Bezdicek, D., Smith, S., Tabatabai, A. and Wollum, A., Eds., Methods of Soil Analysis. Microbiological and Biochemical Properties, SSSA, Madison. Part 2: 865–906.

## Chapter 2: Literature review

### 2.1. Sewage sludge regeneration from waste water treatment plant

Wastewater treatment plants (WWTP) produce two output streams, a stream of treated wastewater to be discharged to watercourses and another stream of separated SS. Wastewater treatment is a complicated process. Basically, there are four stages of treatment: preliminary, primary, secondary and tertiary. In preliminary treatment, grit, grease and gross solids are removed (Fig.2.1 at position ①). Later in primary treatment, suspended solids, both organic and inorganic, from the influent wastewater were removed by sedimentation. Most of the solid settle to the bottom of the sedimentation tanks for a period of time, known as primary sludge or raw sludge (②). The remaining after settlement then flows to the secondary treatment where microorganism activities naturally occur to digest the organic residues in aerobic and anaerobic environments (③). Here, most of the soluble biodegradable organics are degraded and removed as activated sludge. The tertiary treatment can consist removal of nutrients (mainly nitrogen and phosphorus), toxic compounds, residual suspended matter, or microorganisms before water is declared environmental safe for discharge to a watercourse (④). Sludge from primary sedimentation, secondary sedimentation and tertiary treatment was finally going to sludge treatment process (⑤). Fig. 2.1 illustrates the schematic representation of sludge transformation from wastewater to disposal at five above points.

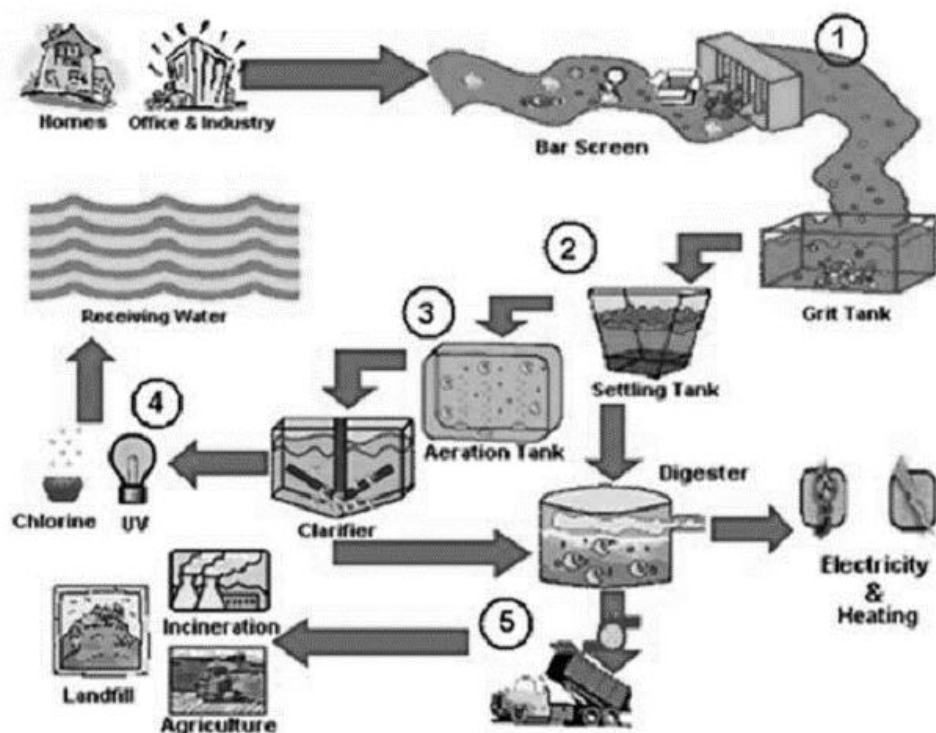


Fig. 2.1. Schematic representation of sludge transformation from wastewater to disposal (Adapted from Olajire, 2009)

## 2.2. Sewage sludge treatment options

There are many ways for treating SS to reduce its total volume including incineration; land application to agricultural land, rangeland, or forests; and landfill (but strictly regulated in developed countries). An overview of possible treatment and disposal system for SS is given in Fig. 2.2. Basically, raw sludge needs to undergo preliminary processes, i.e. thickening, stabilization, dewatering, drying, before going through different treatment methods.

Thickening is the process used prior to the subsequent dewatering process to increase the portion of solids and decrease the portion of liquid by common methods like gravity thickening, dissolved air flotation thickening and centrifugation. Sludge stabilization is especially required if the sludge will be utilized for agricultural purpose (Huybrechts, 2001). Dewatering is considered as the process of natural or mechanical removal of the water from sludge. Similarly, dewatering can be done by gravity method or by mechanical dewatering.

The thermal process involves a decrease of moisture from 90% to 5–10%. Thermally dried SS has interesting properties that allow its use as an alternative fuel. However, some consideration from the point of view of its safe operation to avoid explosive accidents caused by chemical reaction between fuel and oxygen consequent explosion (Fernandez-anez et al., 2014). In addition, the energy consumption needed for this process is extremely high (Wang et al., 2010).

Incineration, known as thermal oxidation, is a sludge disposal option that involves the firing of SS at high temperatures in an enclosed structure. Dewatered sludge will be ignited at temperatures of 420–500°C in the presence of oxygen, and further temperatures of 760–820°C are required for combustion completion of organic solids. As a result, this process reduces the sludge to a mass of ash that is less than 20% of total its original volume (Fericelli et al., 2011). Incineration process has several advantages such as the highest reduction of the total volume, complete destruction of pathogens, reduction of toxins and potential recovery of energy. However, such factors as the high operation costs, the highly skilled and experienced operating staffs, the problems associated with residual ash exceeding the maximum pollutant concentrations, and its discharges to the environment should be taken into consideration.

Landfilling is the disposal without any treatment option for SS. Landfilling can be practiced in mono-filling or co-disposal mode. Mono-filling is defined as sludge disposal practice in which only sludge is buried at a landfill site. In co-disposal, municipal solid wastes or alternatively soil, is mixed with sludge, before being buried at landfill site (Mostafa Kamal Tolba, 2001). Landfilling disposal of sludge is never considered as the best available techniques due to environmental pollution problems, irrespective of how the sludge has been pre-treated (Huybrechts, 2001).

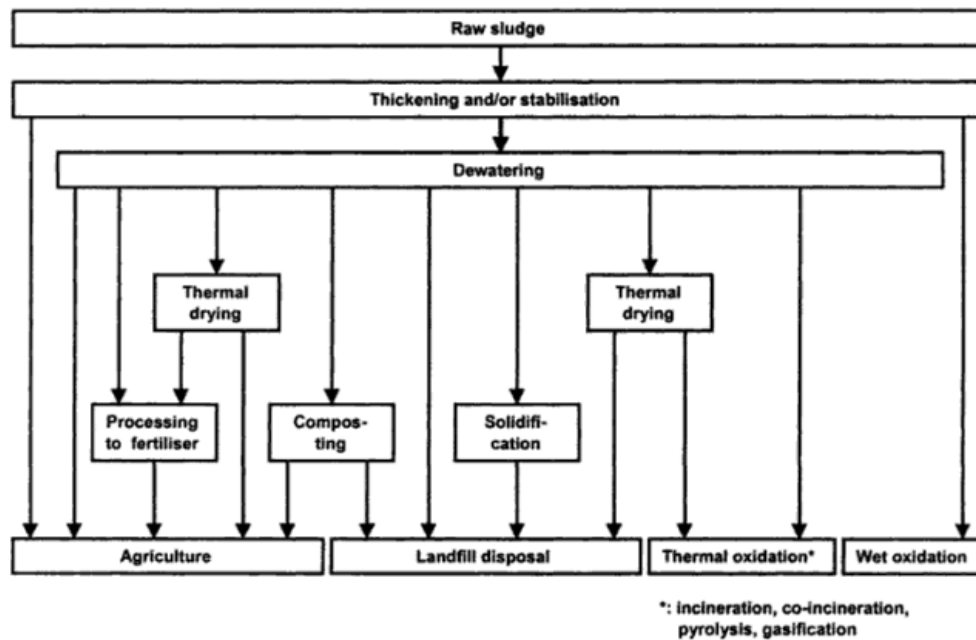


Fig. 2.2. Overview of possible treatment and disposal systems for sewage sludge (adapted from Huybrechts, 2001)

Composting of SS has long been considered as an attractive option for effective reduction of waste volume, and currently receiving increasing attention. This is a process of controlled biological decomposition of organic residue into partially humified material (Tan, 2000). The end-product is a stable material and can be used as soil organic fertiliser or soil conditioner. Details on the composting process will be described in more detail in the next section.

## 2.3. Status for use of sewage sludge in Vietnam and Japan

### 2.3.1. Vietnam

The disposal of SS into landfills has been causing serious environmental pollution in developing countries. An estimated calculation of the total amount of SS in Vietnam has been proposed by Karius (2011). The result shows that in Vietnam, approximately 2.5 million dry Mg of SS is estimated to be produced when all households are connected. Among them, WWTPs were estimated to generate approximately 1.2 million dry Mg of SS annually. These numbers are predicted to increase dramatically in the coming years due to rapid urbanization and development of sewage networks.

In urban areas, wastewater is discharged into the public sewerage system. However, barely a couple of the major Vietnamese cities have WWTPs. Most of the municipal wastewater is discharged into river, channel or lake. Only 4% of collected septage is treated (AECOM, 2010). Therefore, an estimated number 96% of wastewater in Vietnam runs of untreated (Karius, 2011). The sludge generated from sanitation structure are sucked, collected and transported to landfill sites (together with domestic waste) without recovery of the recyclable materials in spite of the increasing awareness of its environmental pollution.

### 2.3.2. Japan

The status for use of SS in Japan is shown in Fig. 2.3. Since 2001 to present, Japan generated more than 2 million dry Mg of SS annually. Remarkably, nearly 80% of SS is reused and recycled as construction materials, compost, fuel, etc. (MLIT, 2011). Since the 1990s, a tremendous process has been made in using treated sludge as construction material like blocks, tiles. The application of incineration treatment of sludge in Japan has advanced, mainly in the city areas where the utilization of inorganic substances in sludge has been progressing. However, the incineration treatment is limited from the viewpoint of transportation and it depends so much on the cement plant in some cities (Leblanc et al., 2008).

Of the total SS generated in Japan, about 10% is used for compost and other green farm purposes. This number has been increasing slightly year by year (Fig. 2.3). The proportion of compost is as high as 73% and that of dewatered sludge and dried sludge are 12% and 11%, respectively (Leblanc et al., 2008).

Landfill and other types of disposal increased in 2011 as a result of the Great East Earthquake in Japan. This explains why the recycling rate was dramatically decreased from 78% in 2010 to 55% in 2011 and then gradually increased to 63% by 2014.

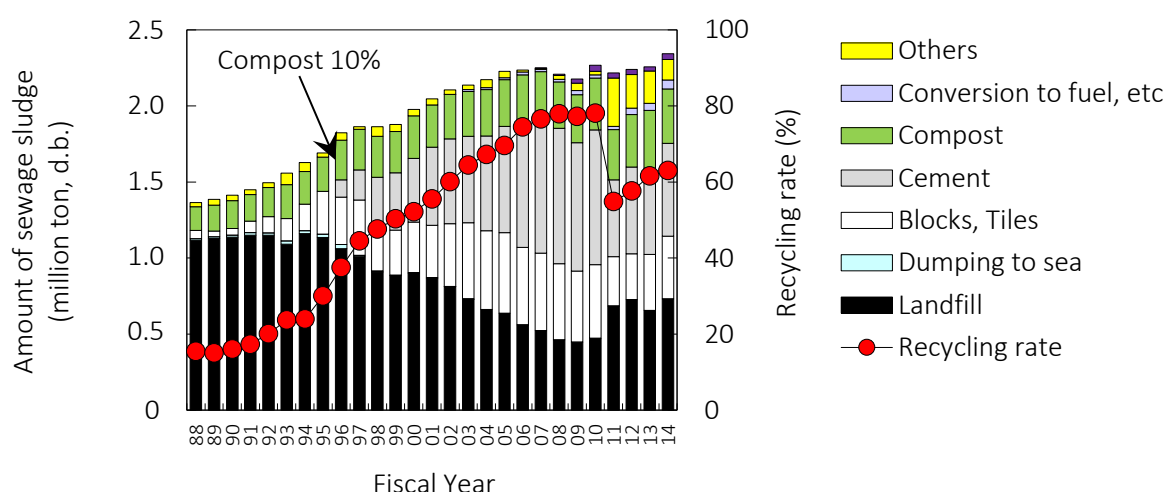


Fig. 2.3. Status of sludge management in Japan (adapted from MLIT, 2011)

### 2.4. Nutrient and heavy metal contents of sewage sludge

The average macronutrient composition of SS is given in Table 2.1. The data indicate that SS contains considerable amounts of nitrogen and phosphorus. The nutrient contents in SS varies considerably depending on the different sources. For example, SS from Spain, Australia, Japan, Brazil, USA and Italia is higher in N, P and K than that from Vietnam, Thailand, Malaysia, Canada, Turkey and England. The potassium concentration in SS is generally lower than N and P. Typically, SS has a total N of 12–76 g kg<sup>-1</sup>, total P of 1–25 g kg<sup>-1</sup> and total K of 1–8 g kg<sup>-1</sup> (Table 2.1).



Table 2.1. Average macronutrient content in sewage sludge of some countries

Country	Total N (g kg <sup>-1</sup> )	Total P (g kg <sup>-1</sup> )	Total K (g kg <sup>-1</sup> )
The standard sludge	35.0	15.3	1.7
Australia	75.0	7.9	8.1
Brazil	58.0	7.9	3.0
Canada	21.0	2.2	0.8
England	12.0	6.0	3.0
Italy	52.0	6.1	—
Japan	73.0	25.0	4.6
Malaysia	21.4	7.1	0.8
Spain	75.8	18.9	4.6
Thailand	34.3	1.1	0.9
Turkey	17.0	3.0	4.1
USA	58.0	19.0	3.6
Vietnam <sup>†</sup>	22.4	7.6	—

Sources: Asagi and Ueno, 2008; Banegas et al., 2007; Leblanc et al., 2008; Parkpain et al., 1998; Rosenani et al., 2004; Tan, 2000. <sup>†</sup>Data from current study

Other than macronutrients, SS also contains heavy metals. The important heavy metal contaminants in SS are Zn, Cu, Ni, As, Hg and Cd (Kumazawa, 1997). The heavy metals concentration in SS varies considerably depending on the contribution of industry. In general, municipal sludge is very high in Cu and Zn contents. The Pb, Ni and Cd contents are on the average relatively small, whereas Hg is hardly detected in SS. Table 2.2 summarizes some regulatory limits of heavy metals from some countries.

## 2.5. Composting process

### 2.5.1. Concepts and purpose

The idea of modern composting was first introduced by Sir Albert Howard in England in 1940. This is a process of controlled biological decomposition of organic residue into partially humified material (Tan, 2000). The exothermic process produces heat which results in an increase of the temperature in the mass, called self-heating. During composting, the organic residue is lost most of its water, carbon and some volatile solids. Particularly, composting helps to reduce the phytotoxins (intermediary metabolites, ammonia, etc.) in the raw starting materials that may harm the plant growth after soil application (Diaz et al., 2007). At the end of composting, the organic waste remains the plant nutrients, i.e., N, P, K and much more, called compost which, can be used as a soil organic fertiliser or soil conditioner (Tan, 2000).

As mentioned in previous section, the huge volume of SS being disposed to landfill sites has caused serious environmental problems. In theory, composting will not only produce valuable products for agricultural purposes but also reduce large amounts of waste into a smaller volume and makes it easier to handle.

Table 2.2. Regulatory standards of heavy metals in sewage sludge of some countries

Region/ countries	Zn	Cu	Pb	Ni	As	Cd	Hg
The standard sludge	1000	500	200	40	–	3	3
North America							
USA	2800	1500	300	420	41	39	17
Canada	4200	1700	1100	420	170	34	11
South & Central America							
Brazil	2800	1500	300	–	41	39	17
Mexico	2800	1500	300	420	41	39	17
European Union							
European directive 1986	2500–4000	1000–1750	750–1200	300–400	–	20–40	16–25
Italy	2500	1000	750	300	–	20	10
Germany	1500	700	120	60	–	2.5	1.6
Asia							
China	3000	1500	1000	200	75	20	15
Japan	–	–	100	300	50	5	2
Vietnam <sup>†</sup>	–	–	250	100	2	2.5	2

Source: Leblanc et al., 2008; Kumazawa, 1997. All data are expressed in mg kg<sup>-1</sup> dry weight.

<sup>†</sup> Vietnam has not issued specific regulations with regards to heavy metal limits in SS, therefore, the regulatory standards of heavy metals in term of fertilisers are used as a reference.

## 2.5.2. Methods of composting

The composting can be performed in several ways. In this literature, methods of composting are classified based on the system: nonreactor and reactor or vessels as described by (Schuchardt, 2005). Classification of composting systems is shown in Fig. 2.4.

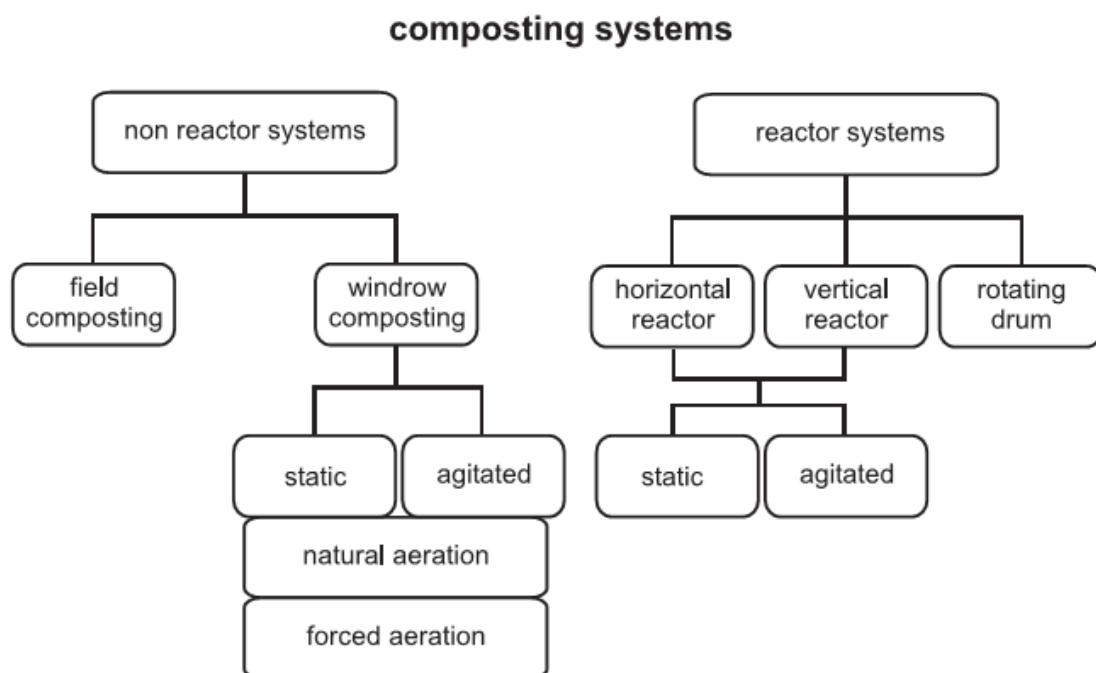


Fig. 2.4. Classification of composting systems (adapted from Schuchardt, 2005).

For windrow system with or without aeration, the aeration can be achieved by mechanical turning (agitated) and/or the delivery of air from the base of the windrow (static). Window systems have been the predominant form of composting in large scale, which are the oldest and least problematic input to process (Christensen, 2011).

Reactor or in-vessel composting involves the use of an enclosed space (e.g., container, box, bin, tunnel and so on) in which composting is controlled by mechanical or regulated aeration. The reactors are designed as horizontal or vertical flow or rotating-drum depending on the appropriate facilities. For the horizontal and vertical models, aeration is provided to the reactor by a blower fan which can work in a positive (blowing) and/or negative (sucking) mode. The air flow can be controlled accordingly the change of compost temperatures or the oxygen/carbon dioxide concentration in the air (Schuchardt, 2005). During composting process, the mixture can be turned (agitated) mechanically to increase the homogeneous conditions. In contrast, there is no turning/mixing in the static mode.

### 2.5.3. Composting process

In the composting process, microorganisms utilize the organic matter as their food source and generates heat, CO<sub>2</sub> and water. Microbes also require oxygen and water for their activities. The basic diagram of an aerobic composting process is illustrated in Fig. 2.5. Composting process can be divided into three phases based on the changes of temperatures of the compost mixture: the initial mesophilic phase, the second thermophilic phase and the final (second mesophilic phase). For the first one, easily degradable

organic substances like sugars and protein are degraded by microbes (Diaz et al., 2007). The temperatures of the compost pile gradually/rapidly rise from ambient air up to about 40°C. In the next phase, thermophilic microbes can adapt to the higher temperature at 35–65°C and almost replace the previous mesophilic microbes. More resistant parts of substance are decomposed and accelerates the increase of temperature to the peak points. These temperatures must be reached for a determined length of time to destroy pathogen organisms (Tan, 2000).

Maintaining a minimum temperature of 55°C for at least three consecutive days is required for acceptable composts (Burge et al. 1978). However, for windrow composting, the temperatures over 55°C should be maintained for at least 15 days (Larney and Olson, 2006). Moisture and oxygen are especially important during this stage to maintain the high population of thermophilic microbes. When the compost fails to reheat after turning, the composting slows down and enters the final phase before becoming stable at the end of the process.

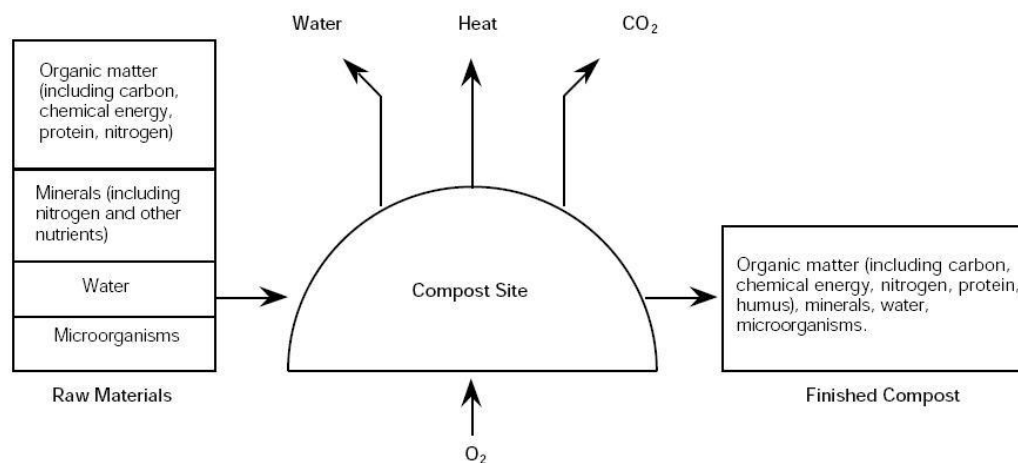


Fig. 2.5. A basic schematic of an aerobic composting process (adapted from Rynk, 1992)

#### 2.5.4. Composting process management

To produce high-quality compost, several variables must be controlled to provide the desirable conditions for composting microbes. The most important variables are feedstock, aeration conditions, moisture and temperature. In this literature, only temperature and aeration conditions are focused because SS by itself has high values of moisture content and no water was added throughout the composting process.

Although the compost temperatures are mainly governed by the self-heating due to microorganism activities, the temperature of compost can be regulated by improving the aeration condition manual/mechanical turning or forced aeration systems. These works may help to cool down the compost or re-heat the pile if the substrate has not been stable yet. Several studies used the temperature feedback with an adjustable set point at predefined temperature to control the on-demand of

aeration/ventilation conditions, by which the air was introduced intermittently (Lopez-Real and Baptista, 1996; Paredes et al., 2000) or continuously (Tiquia and Tam, 2000). For example, when the compost temperature exceeds the set point, the air was continuously introduced to cool down the compost pile. In contrast, when the compost temperature is below the set point, the air was supplied intermittently based on pre-setup schedule using the time-delay relay (Das et al., 1998).

Furthermore, the compost temperature is affected by ambient temperature. Controlling the ambient temperature is possible in case of the small scale of composting. The air inside the reactor can be heated using an electric heater and agitated with a fan for the isothermal reactor (Bach et al., 1984) or by submerging the air pipes in the water baths for small laboratory pilot (Lashermes et al., 2012). In many cases, the insulation surrounding the composting reactor is necessary to reduce the heat loss. For windrow composting where ambient temperature cannot be controlled especially in the winter due to an environmental factor, the volume and the mass of compost play a very important role to attain the higher thermophilic temperature. Tiquia and Tam (2000) proposed that increasing the volume of compost to 2.5 to 3.0 m<sup>3</sup> may help to promote greater efficiency of composting during winter.

During composting of the wet substrates, the air can find it difficult to penetrate through the compost piles in spite of diffusion or forced aeration. To solve this problem, bulking agents such as woodchips, wheat straw, peat, grass hay, garden trimmings, sawdust and so on are often mixed with the compost materials to provide texture support; absorb the high moisture content; improve the porosity for adequate aeration and allow easier movement of air across the mixture (Haug, 1993; Banegas et al., 2007). To determine the critical bulking agent requirement for municipal biosolids composting, Eftoda and McCartney (2004) tested four volumetric ratios of municipal biosolids and woodchips ranging from 1:1 to 1:4 (biosolids : woodchips). The results indicated that critical bulking agent requirements, to achieve a free air space more than 20% and a minimum pore space oxygen content of more than 5%, were estimated at recipes of 1:2.5 and 1:2.8 (biosolids : woodchips, v/v).

## 2.6. Utilization of SS compost

### 2.6.1. Effects on crop production and soil properties

Land application of SS is generally considered as the best option for disposal because it offers many benefits to plant, soil and recycling of nutrients in the environment. Application of SS compost to agricultural and forestry soil needs to have a balance between maximizing agronomic benefits while protecting soil and water quality because composting does not remove heavy metal from the raw materials, e.g., Pb, Cd, Hg and As (Tan, 2000).

The main determinant for efficient agronomic use is nitrogen availability from compost. The nitrogen use efficiency from chemical N fertilisers has been well understood, whereas that from compost requires further investigation (Gutser et al., 2005). Composted sludge can be used as a soil amendment to supply

nutrients to plants, improve soil physical properties, and increase the percentage of organic matter (Warman and Termeer 2005; Pedra et al. 2007), although negative effects associated with heavy metal accumulation and phytotoxicity phenomena in plant-soil systems should be taken into consideration (Tan 2000; Alvarenga et al., 2015).

Many studies have shown that the application of compost has promoted positive effects on a wide variety of crop. The yield of sorghum, corn, potatoes, tobacco, tomatoes, beet and ryegrass was increased as affected by compost as reviewed by Gallardo-Lara and Nogales (1987). Recently, in a two-year experiment using digested SS compost applied to soil for grass forage and feed corn, Warman and Termeer (2005) demonstrated that effectiveness of compost on corn yield was similar to that of chemical N fertiliser.

Furthermore, compost application to agricultural soil has been reported to improve the physical, chemical and biological and properties of soil. The improvement of total porosity caused by sludges and composts was comparable to that of manure in a two-year experiment on Italian sandy loam soil (Pagliai et al., 1981). Likewise, Mondal et al. (2015) observed the changes in soil quality in response to short-term application of municipal SS in a typical haplustept under cowpea-wheat cropping system. The results showed that the addition of compost at application rate of 15 tons ha<sup>-1</sup> to the upper layer of a soil favours the amount of soil macro aggregate and porosity.

With regard to the effects of SS compost on the chemical soil properties, Scherer et al. (1962) indicated that total organic carbon and total soil nitrogen content in soil applied with SS at high rate of 7.44 tons ha were significantly higher than those with mineral fertiliser in a long-term 45-year experiment following a cereal-root crop sequence. The addition of compost to the soil improved not only physicochemical properties but also positively affected microbial population and their enzymatic activities as reviewed by Carmen et al. (2008).

The application of compost to agricultural soil is shown to reduce the number of parasitic nematodes, and increase both the numbers of micro-arthropods and earthworms (Leroy et al., 2007). In addition, Melero et al. (2007) showed a clear increase of microbial biomass and enzymatic activities at the fourth year of compost application to clay soils.

#### 2.6.2. Effects on heavy metal accumulation

The greatest concern in sludge utilization is with respect to heavy metals because they may be present in relatively high concentrations in compost and accumulate in soil with continuous application (Kumazawa, 1997). Several studies have been carried out on the accumulation of heavy metals in plant-soil systems after application of SS compost. Gallardo-Lara and Nogales (1987) noted that maximum contamination of heavy metals takes place in the top layer soils. However, through shrink-swell cracks,

heavy metals can penetrate into the subsoil layers and consequently contaminate the surface groundwater (Behbahania et al., 2009).

With SS, Stevenson's study showed that an annual application of 20 tons ha<sup>-1</sup> of SS for 20 years would result in an increase of 8 mg kg<sup>-1</sup> of Co, 180 mg kg<sup>-1</sup> of Cu, 270 mg kg<sup>-1</sup> of Pb, and 890 mg kg of Zn (as cited in Tan, 2000). In order to avoid the heavy metal pollution in soil, in Japan, when SS is applied to agricultural land, it has to meet the standards defined by the Japanese fertiliser law (Kumazawa, 1997). For example, the upper limits for As, Cd and Hg in sludge are 50, 5 and 2 mg kg<sup>-1</sup> (Table 2.2). After applying to soil, the heavy metal accumulation in farm soils will be controlled by the reference to Zn content with the upper permissible level of 120 mg kg<sup>-1</sup> soil.

## 2.7. Dynamics of compost N in the plant–soil system and introduction of stable N isotope

N dynamics in plant–soil system lie at the heart of some of the questions being asked of researchers by farmers, environmentalists and policy makers (Stockdale et al., 1997). As mentioned in the previous section, SS compost contains a high value of N, which is present in both inorganic and organic forms. That is the reason why studies on dynamics of compost N have been performed to determine the N availability from composts as well as their impacts on the environment. The nitrogen dynamics in plant–soil system are very complex and involve many interactions between plant and soil factors (Luce et al., 2011). These dynamics include N mineralisation, nitrification, immobilisation, N uptake by plant and potential N losses to the environment via leaching and denitrification. Fig. 2.6 illustrates simplified N dynamics in plant–soil system.

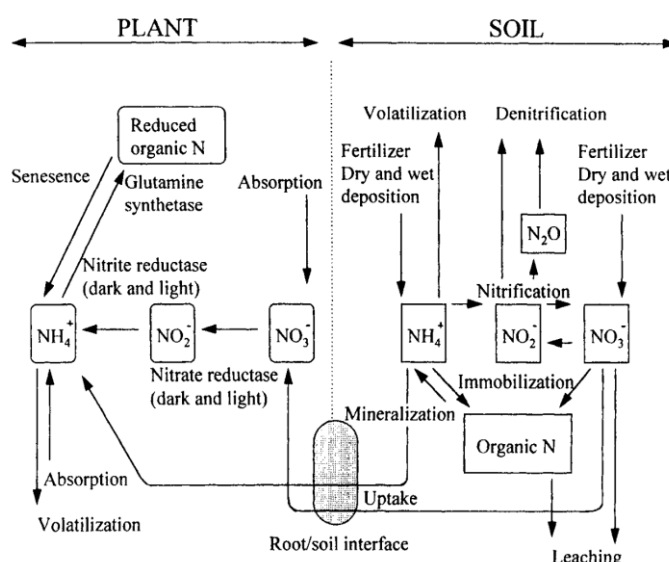


Fig. 2.6. Simplified N dynamics in plant–soil system (adapted from Stockdale et al., 1997)

Nitrogen is composed of two stable isotopes with atomic masses of 14 and 15. The majority of N in the atmosphere is composed of <sup>15</sup>N (99.6337%) and the remainder is composed of <sup>14</sup>N (0.3663%) (as

cited in Bedard-Haughn et al., 2003). Natural  $^{15}\text{N}$  abundance is expressed as delta ( $\delta$ )  $^{15}\text{N}$  in per mill (‰)  $^{15}\text{N}$  excess over a standard:

$$\delta^{15}\text{N} (\text{‰}) = 1000 \times (R_{\text{sample}} - R_{\text{standard}}) / R_{\text{standard}}$$

Where,  $\delta^{15}\text{N}$  (‰) is the isotope ratio of the sample relative to the atmosphere air standard and  $R_{\text{sample}}$  and  $R_{\text{standard}}$  are the molar ratios of  $^{15}\text{N}$  and  $^{14}\text{N}$ , respectively. Based on the principles of tracer data (using heavy or radioactive atoms), N transformations in soil and fate of N in plant–soil system are estimated. For example, when using a  $^{15}\text{N}$ -enriched source, e.g.  $(^{15}\text{NH}_4)_2\text{SO}_4$  or  $^{15}\text{N}$ -labelled compost, the percentage of N derived from the enriched N source ( $\%N_{\text{enrich source}}$ ) in a particular N pool is calculated as described by Bedard-Haughn et al. (2003):

$$\%N_{\text{enrich source}} = (\text{atom}\%^{15}\text{N}_{\text{excess in sample}} / \text{atom}\%^{15}\text{N}_{\text{excess in enriched source}}) \times 100.$$

### 2.7.1. N mineralisation and immobilisation

The N mineralisation process involves the conversion of organic N forms to  $\text{NH}_3$  or  $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$ . In the first step, complex organic N forms are broken down into simpler forms (e.g. amino acids and amino sugars). These small organic N compounds are further converted to  $\text{NH}_4\text{-N}$  via ammonification by heterotrophic microorganisms. The subsequent conversion from  $\text{NH}_4\text{-N}$  to  $\text{NO}_3\text{-N}$  is called nitrification and it is carried out in two steps mainly through two groups of autotrophic bacteria (*Nitrosomonas* and *Nitrobacter*). The  $\text{NH}_4\text{-N}$  is first converted to nitrite  $\text{NO}_2\text{-N}$  then  $\text{NO}_2\text{-N}$  is further oxidized to  $\text{NO}_3\text{-N}$ . The process of nitrification is generally fast and any  $\text{NH}_3$  produced due to ammonification is rapidly nitrified to  $\text{NO}_3$  (Benbi and Nieder, 2003).

Immobilisation is the process occurs in the reverse direction (Fig. 2.6), in which inorganic N forms ( $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$ ) are assimilated by microbial biomass and transformed them into organic forms in their cells and tissues (Benbi and Nieder, 2003). The relative magnitudes of N mineralisation and immobilisation determine whether the overall effect is net mineralisation or net immobilisation.

With regards to compost N, the processes of mineralisation and immobilisation may depend primarily on the C/N ratio of the amendments and the maturity of compost. The application of organic fertilisers having low N content with high C/N ratio  $>15$  can result in limiting the N availability due to the immobilisation (Gutser et al., 2005). In contrast, the application of organic fertilisers having C/N ratio  $< 12$  can result in net N mineralisation (Iglesias-Jimenez and Alvarez, 1993). Mature compost caused net N mineralisation in soil after its application, while a compost which has undergone a bio-oxidative phase of composting but not maturation may cause a slight N immobilisation immediately after its addition to soil (Bernal et al., 1998).

The N transformations in soil can be affected by the supplemental N and C inputs. Han et al. (2004) demonstrated that a combined application of compost with chemical N fertiliser could improve the



compost use efficiency by increasing mineralisation of compost N, especially in soils with low mineral N content. However, the combined incorporation of SS and oat straw to soil can increase the microbial nitrogen immobilisation (Giacomini et al., 2015). Likewise, Yadvinder-Singh and Bijay-Singh (1994) indicated that microorganism populations increase greatly as evident by increased release of CO<sub>2</sub> when plant residue with a wide C/N ratio (>25) is incorporated into the soil (Fig. 2.7).

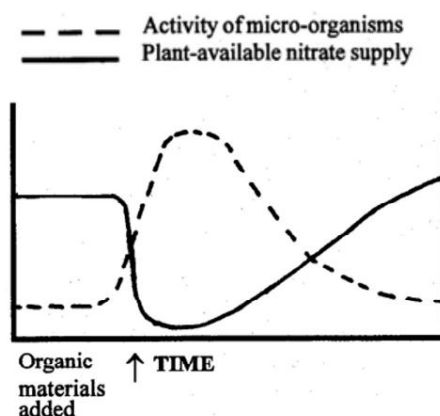


Fig. 2.7. Levels of nitrogen available to plants based on microbial decomposition (adapted from Yadvinder-Singh and Bijay-Singh, 1994)

### 2.7.2. N uptake by plant

There are three major forms of N in soil: organic N, ammonium N and nitrate N. Organic N forms make up for the highest proportion (>90%) of the total N in the soil. Only a small part is in inorganic N (NH<sub>4</sub>-N and NO<sub>3</sub>-N). Therefore, the mineralisation of organic N compounds into inorganic N is considered as the major source of plant-available N. It is well known that plant can use NH<sub>4</sub>-N and NO<sub>3</sub>-N in soil solution. However, recent studies indicated that plant can also take up organic N in form of free amino acid at a biologically important rate (Jones and Kielland, 2002; Leduc and Rothstein, 2010). Compost provides a supplemental N source for plant growth in both organic and inorganic form, however, the N mineralisation rate among different composts can vary significantly.

Total plant N uptake accounts for more than 50% of the N that leaves the soil system. Total N uptake (from soil and fertiliser sources) by cereals ranges from 100 to 180 kg N ha<sup>-1</sup> depending on the grain yield potential.

With regards to compost-N, several non-isotopic studies have attempted to quantify plant N uptake from organic residue, also for compost source based on the difference between  $\pm$ compost treatments (Chalk et al., 2013). However, these studies may have led to ambiguous conclusions due to the complexities of the mineralisation–immobilisation turnover processes (Hood et al., 1999). The use of <sup>15</sup>N technique allows the behavior of these added materials to estimate the plant N uptake from each source (Bedard-Haughn et al., 2003; Asagi and Ueno, 2008; Barbanti et al., 2011; Binh et al., 2015). In a study

on corn uptake and microbial immobilisation of  $^{15}\text{N}$ -labelled urea-N in soil as affected by composted pig manure, Choi et al. (2001) indicated that the uptake of urea N was reduced in the presence of compost due to the retention of N in soil through microbial immobilisation. These results suggest that there is a close relationship between N transformation in soil and total plant N uptake in plant–soil system.

### 2.7.3. N retention in soil and N losses

The N can be taken up by plant, immobilized by the soil or lost from the soil system. The aforementioned immobilisation process will temporary tie up the inorganic N by soil microorganisms, therefore, reduce the risk of N loss to the environment. However, the immobilized N in soil will not available for plant uptake until it is re-mineralized to inorganic forms.

The amount of N lost to the environments can become a serious pollutant and a conservation concern. The N losses can be leaching of nitrate to lower layer soil or to ground water, ammonia volatilization and denitrification in the atmosphere. In addition, soil N can also be lost by soil erosion and surface runoff.

N losses from compost-amended soil can be determined indirectly by  $^{15}\text{N}$  mass balance using  $^{15}\text{N}$  labelled compost or by direct measurement of gaseous N emissions or nitrate leaching in paired  $\pm$  compost treatments (Chalk et al., 2013). The indirect method will be discussed more in the last section of this dissertation.

## References

- AECOM. 2010. A Rapid Assessment of Septage Management in Asia: Policies and Practices in India, Indonesia, Malaysia, the Philippines, Sri Lanka, Thailand, and Vietnam. Environmental Cooperation-Asia.
- Alvarenga, P., C. Mourinha, M. Farto, T. Santos, P. Palma, J. Sengo, M.C. Morais, and C. Cunha-Queda. 2015. Sewage sludge, compost and other representative organic wastes as agricultural soil amendments: Benefits versus limiting factors. *Waste Manag.* 40: 44–52.
- Asagi, N., and H. Ueno. 2008. Determination of application effects of Sewage sludge on growth , soil properties , and N uptake in Komatsuna by using the indirect  $^{15}\text{N}$  isotope method. *Commun. Soil Sci. Plant Anal.* 39: 1928–1942.
- Bach, P.D., M. Shoda, and H. Kubota. 1984. Rate of composting of dewatered sewage sludge in continuously mixed isothermal reactor. *J. Ferment. Technol.* 62(3): 285–292.
- Banegas, V., J.L. Moreno, J.I. Moreno, C. García, G. León, and T. Hernández. 2007. Composting anaerobic and aerobic sewage sludges using two proportions of sawdust. *Waste Manag.* 27(10): 1317–1327.
- Barbanti, L., M. Grigatti, and C. Ciavatta. 2011. Nitrogen release from a  $^{15}\text{N}$ -labeled compost in a sorghum growth experiment. *J. Plant Nutr. Soil Sci.* 174(2): 240–248.
- Bedard-Haughn, A., J.W. Van Groenigen, and C. Van Kessel. 2003. Tracing  $^{15}\text{N}$  through landscapes : potential uses and precautions. *J. Hydrol.* 272: 175–190.
- Behbahani, A., S.A. Mirbagheri, N. Khorasani, J. Nouri, and A.H. Javid. 2009. Heavy metal contamination of municipal effluent in soil and plants. *J. Food, Agric. Environ.* 7(3 & 4): 851–856.
- Benbi, D.K., and R. Nieder. 2003. Handbook of processes and modeling in the soil-plant system. The Haworth Reference Press.
- Bernal, P.M., A.F. Navarro, M.A. Sánchez-Monedero, A. Roig, and J. Cegarra. 1998. Influence of sewage sludge compost stability and maturity on carbon and nitrogen mineralization in soil. *Soil Biol. Biochem.* 30(3): 305–313.
- Binh, N.T., H.T. Quynh, and K. Shima. 2015. Effect of Composts Combined with Chemical N Fertilizer on Nitrogen Uptake by Italian Ryegrass and N Transformation in Soil. *J. Agric. Chem. Environ.* 4(2): 37–47.
- Burge, W.D., W.N. Cramer, and E. Epstein. 1978. Destruction of Pathogens in Sewage Sludge by Composting. *Am. Soc. Agric. Biol. Eng.* 21(3): 0510–0514.
- Carmen, M., V. García, F. Suárez, E. M<sup>a</sup>, J. López, and J. Moreno. 2008. Influence of Compost Amendment on Soil Biological Properties and Plants. *Dyn. Soil, Dyn. Plant* 2(1): 1–9.
- Chalk, P.M., A.M.T. Magalhães, and C.T. Inácio. 2013. Towards an understanding of the dynamics of compost N in the soil-plant-atmosphere system using  $^{15}\text{N}$  tracer. *Plant Soil* 362(1–2): 373–388.

- Choi, W.J., S.A. Jin, S.M. Lee, H.M. Ro, and S.H. Yoo. 2001. Corn uptake and microbial immobilization of <sup>15</sup>N-labeled urea-N in soil as affected by composted pig manure. *Plant Soil* 235(1): 1–9.
- Christensen, T.H. 2011. *Solid Waste Technology and Management*. Blackwell Publishing Ltd.
- Das, K., E. Tollner, and T. Tornabene. 1998. Composting pulp and paper industry solid wastes: process design and product evaluation. *Proceedings of the Composting in the Southeast-conference and Expo Athens, GA*. p. 9–11.
- Diaz, L.F., M. De Bertoldi, W. Bidlinmaier, and E. Stentiford. 2007. *Compost Science and Technology*. Waste management series 8. Elsevier.
- Eftoda, G., and D. McCartney. 2004. Determining the Critical Bulking Agent Requirement For Municipal Biosolids Composting. *Compost Sci. Util.* 12(3): 208–218.
- Fericelli, P.D., C. Florida, U. States, S.L.C. Omposition, and C. Haracteristics. 2011. Comparison of Sludge Treatment by Gasification vs. Incineration. Ninth LACCEI Latin American and Caribbean Conference (LACCEI'2011), Engineering for a Smart Planet, Innovation, Information Technology and Computational Tools for Sustainable Development. pp 1–10.
- Fernandez-anez, N., J. Garcia-torrent, and L. Medic-pejic. 2014. Flammability properties of thermally dried sewage sludge. *Fuel* 134: 636–643.
- Gallardo-Lara, F., and R. Nogales. 1987. Effect of the Application of Town Refuse Compost on the Soil-Plant System : A Review. 19(I 987): 35–62.
- Giacomini, S.J., V.L.G. Simon, C. Aita, L.M. Bastos, D.A. Weiler, and M. Redin. 2015. Carbon and Nitrogen Mineralization in Soil Combining Sewage Sludge and Straw . *Rev. Bras. Ciência do Solo* 39: 1428–1435.
- Gutser, R., T. Ebertseder, A. Weber, M. Schraml, and U. Schmidhalter. 2005. Short-term and residual availability of nitrogen after long-term application of organic fertilizers on arable land. *J. Plant Nutr. Soil Sci.* 168(4): 439–446.
- Han, K.-H., W.-J. Choi, G.-H. Han, S.-I. Yun, S.-H. Yoo, and H.-M. Ro. 2004. Urea-nitrogen transformation and compost-nitrogen mineralization in three different soils as affected by the interaction between both nitrogen inputs. *Biol. Fertil. Soils* 39(3): 193–199.
- Haug, R.T. 1993. *The Practical Handbook of Compost Engineering*. Lewis Publishers, Boca Raton, Fl.
- Hood, R.C., K.N. Goran, M. Aigner, and G. Hardarson. 1999. A comparison of direct and indirect <sup>15</sup>N isotope techniques for estimating crop N uptake from organic residues. *Plant Soil* 208: 259–270.
- Huybrechts. 2001. Best available techniques for the treatment and disposal of sewage sludge in Flanders. In: Ravindra K.D, Mukesh C.L., and Michael J.M, editors. *Recycling and reuse of sewage sludge*. Thomas Telford.
- Iglesias-Jimenez, E., and C.E. Alvarez. 1993. Apparent availability of nitrogen in composted municipal refuse. *Biol. Fertil. Soils* 16(4): 313–318.

- Jones, D.L., and K. Kielland. 2002. Soil amino acid turnover dominates the nitrogen flux in permafrost-dominated taiga forest soils. *Soil Biol. Biochem.* 34(2): 209–219.
- Karius, R. 2011. Developing an integrated concept for sewage sludge treatment and disposal from municipal wastewater treatment systems in (peri-) urban areas in Vietnam [Diploma thesis]. Technische Universität Dresden.
- Kumazawa, K. 1997. Use of sewage sludge for agriculture in Japan. Proceedings of consultants meetings on sewage sludge and wastewater for use in agriculture. Oct. 1997. 199 pp. 111-127. Joint FAO/IAEA Div. of Nuclear Techniques in Food and Agriculture, Vienna Austria, Intern.
- Larney, F.J., and A.F. Olson. 2006. Windrow temperatures and chemical properties during active and passive aeration composting of beef cattle feedlot manure. *Can. J. Soil Sci.*: 783–797.
- Lashermes, G., E. Barriuso, M. Le Villio-Poitrenaud, and S. Houot. 2012. Composting in small laboratory pilots: Performance and reproducibility. *Waste Manag.* 32(2): 271–277.
- Leblanc, R.J., P. M., and R. P.R. 2008. Global atlas of excreta, wastewater sludge and biosolids management: moving forward the sustainable and welcome uses of a global resource. United Nations Human Settlements Programme (UN-HABITAT).
- Leduc, S.D., and D.E. Rothstein. 2010. Plant-available organic and mineral nitrogen shift in dominance with forest stand age Plant-available and mineral shift organic nitrogen in dominance with forest stand age. *Ecology* 91(3): 708–720.
- Leroy, B.L.M.M., L. Bommele, D. Reheul, M. Moens, and S. De Neve. 2007. The application of vegetable , fruit and garden waste ( VFG ) compost in addition to cattle slurry in a silage maize monoculture : Effects on soil fauna and yield. *Eur. J. Soil Biol.* 43: 91–100.
- Lopez-Real, J., and M. Baptista. 1996. A preliminary comparative study of three manure composting systems and their influence on process parameters and methane emissions. *Compost Sci. Util.* 4(3): 71–82.
- Luce, M.S., J.K. Whalen, N. Ziadi, and B.J. Zebarth. 2011. Nitrogen Dynamics and Indices to Predict Soil Nitrogen Supply in Humid Temperate Soils. 1st ed. Elsevier Inc.
- Melero, S., E. Madejón, J.F. Herencia, and J.C. Ruiz. 2007. Biochemical properties of two different textured soils (loam and clay) after the addition of two different composts during conversion to organic farming. *Spanish J. Agric. Res.* 5(4): 593–604.
- MLIT. 2011. Status for the Use of Sewage Sludge in Japan. Japanese Minist. Land, Infrastructure, Transp. Tour.
- Mondal, S., R.D. Singh, A.K. Patra, and B.S. Dwivedi. 2015. Environmental Nanotechnology , Monitoring & Management Changes in soil quality in response to short-term application of municipal sewage sludge in a typic haplustept under cowpea-wheat cropping system. *Environ. Nanotechnology, Monit. & Manag.* 4: 37–41.

- Mostafa Kamal Tolba. 2001. *Our Fragile World: Challenges and Opportunities for Sustainable Development*. EOLSS Publishers.
- Olajire, O.A. 2009. Optimisation of sewage sludge logistics in wastewater treatment plants [Master thesis]. Imperial College London.
- Pagliai, M., G. Guidi, M. La Marca, M. Giachetti, and G. Lucamante. 1981. Effects of Sewage Sludges and Composts on Soil Porosity and Aggregation. *J. Environ. Qual.* 10: 556–561.
- Paredes, C., A. Roig, M.P. Bernal, M.A. Sanchez-Monedero, and J. Cagarra. 2000. Evolution of organic matter and nitrogen during co-composting of olive mill wastewater with solid organic wastes. *Biol. Fertil. soils* 32: 222–227.
- Parkpain, P., S. Sirisukhodom, and A.A. Carbonell-Barrachina. 1998. Heavy metals and nutrients chemistry in sewage sludge amended Thai soils. *J. Environ. Sci. Heal. Part A* 33(4): 573–597.
- Pedra, F., A. Polo, A. Ribeiro, and H. Domingues. 2007. Effects of municipal solid waste compost and sewage sludge on mineralization of soil organic matter. *Soil Biol. Biochem.* 39(6): 1375–1382.
- Rynk, R. 1992. *On-farm composting handbook*. Northeast Regional Agricultural Engineering Service. Cooperative Extension Ithaca, NY.
- Scherer, H.W., D.J. Metker, and G. Welp. 1962. Effect of long-term organic amendments on chemical and microbial properties of a luvisol. *Plant Soil Environ.* 57(11): 513–518.
- Schuchardt, F. 2005. Composting of organic Waste. In: H.-J. Jördening and J. Winter, editors. *Environmental biotechnology. Concepts and applications*. WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim.
- Stockdale, E.A., J.L. Gaunt, and J. Vos. 1997. Soil–plant nitrogen dynamics: what concepts are required? *Dev. Crop Sci.* 25: 201–215.
- Tan, K.H. 2000. *Environmental Soil Science*, Third Edition. CRC Press, UK.
- Tiquia, S.M., and N.F.Y. Tam. 2000. Co-composting of spent pig litter and sludge with forced-aeration. *Bioresour. Technol.* 72(1): 1–7.
- Walter, I., F. Martínez, and V. Cala. 2006. Heavy metal speciation and phytotoxic effects of three representative sewage sludges for agricultural uses. *Environ. Pollut.* 139(3): 507–514.
- Wang, W., Y. Luo, and W. Qiao. 2010. Possible solutions for sludge dewatering in China. *Front. Environ. Sci. Eng.* 4(1): 102–107.
- Warman, P.R., and W.C. Termeer. 2005. Evaluation of sewage sludge, septic waste and sludge compost applications to corn and forage: Yields and N, P and K content of crops and soils. *Bioresour. Technol.* 96(8): 955–961.
- Yadvinder-Singh and Bijay-Singh. 1994. Soil and Fertilizer Nitrogen. In: Leeper, G.W; Uren, N.C; Bryant, Ray B. *Soil science: an introduction*. Melbourne University Press, Melbourne, Australia.

## Chapter 3: Composting process

### 3.1. Introduction

Aerobic composting could be performed in small to large scales, i.e. in laboratory (Malińska et al., 2014), pilot (Lu et al., 2008) and industrial composting facilities (Kuter et al. 1985) depending on the purpose of producers. For a small-scale study, composting was performed in order to evaluate the feasibility in an attempt to produce a small compost sample for trial purposes prior to performance in a full-scale project.

However, the challenge of composting raised under field condition when the ambient temperature cannot be controlled as perfectly as the reactor was placed in an incubator and the heat was supplied externally (Morisaki et al., 1989). Thus, the temperature within the compost reflected the seasonal ambient temperature (Tiquia et al., 1998). These authors reported that the composting carried out in the winter was slow and the finished compost was biologically unstable and immature because the composting process was not well controlled.

Several studies on composting have focused on the changes in compost physicochemical properties (Brewer & Sullivan 2003; El Fels et al., 2014) as indicators of compost stability and maturity, but information on the active fractions of organic nitrogen like amino acids and amino sugars during composting process is very scarce. Free amino acids are thought to be an easily decomposable organic N and become an important N source for plants (Jones and Kielland, 2002), but also the factor limiting N availability for plant uptake due to N source for heterotrophic microorganisms in the soil system.

During composting of the wet substrates like SS alone, the air can find it difficult to penetrate through the compost piles in spite of diffusion or forced aeration. To solve this problem, bulking agents such as woodchips, wheat straw, peat, grass hay, garden trimmings, sawdust, etc. are often mixed with the compost materials to provide texture support; absorb the high moisture content; improve the porosity for adequate aeration and allow easier movement of air across the mixture (Haug, 1993; Banegas et al., 2007).

However, one of the problems associating with the selection of suitable bulking agents is the ratio of bulking agent to the compostable material. That is because using more agent may provide more air space, but would also result in less material being composted in a given volume and sieving compost can be more tricky (McGuckin et al., 1999). In addition, the insufficient ratio would result in limitation of aeration condition and then reduce the compost quality (Malińska and Zabochnicka-Świątek, 2013).

Therefore, the main objective of this chapter was two-fold: first, to examine the feasibility of SS composting using a simple aeration method under two seasonal temperatures, providing information about the changes in physicochemical properties of sludge during the composting process with

particular attention to the temperature and the nitrogen availability; and second, to investigate the influence of volumetric ratios of sludge and woodchips as bulking agent on the effectiveness of composting process.

### 3.2. Materials and methods

#### 3.2.1. Composting runs 1 and 2

##### 3.2.1.1. SS and bulking material

In 2016, two Japanese sludges (called sludge 1 and sludge 2) were collected from the Green Yuki composting plant in Okayama prefecture, Japan and transported to the experimental site just before each run of the composting trials. In the preliminary survey, a Vietnamese sludge (V-sludge) sample was collected from a WWTP in Ho Chi Minh City and carried to Japan for laboratory analysis. However, this sludge was not used in subsequent composting experiments due to the feedstock limitation. The physicochemical characteristics of the sludges are presented in Table 3.1.

Woodchips of Japanese cedar (*Cryptomeria japonica*) with 5-7 cm length were used as the bulking agent to improve aerobic condition during composting. SS were mixed with woodchips at a volumetric ratio of 1:1 (36 kg of sludge: 5 kg of woodchips, wet basis).

##### 3.2.1.2. Composting facilities

Two aerated composting trials in sequence, run 1 using sludge 1 and run 2 using sludge 2, were performed in small scale at Okayama University, Okayama prefecture, Japan. Run 1 began on 15 February 2016 (winter) and run 2 began on 19 May 2016 (summer). Composting was monitored for 63 and 57 days for run 1 and run 2, respectively. Two plywood compost bins ( $0.45 \times 0.45 \times 0.45 \text{ m}^3$ ) were covered by a 50-mm thick styrofoam insulator to reduce heat loss. Air was forced up through small holes perforated on two open-ended parallel PVC pipes ( $\varnothing 16 \text{ mm}$ , 0.25 m apart) laid at the base. The pipes were covered by woodchips to prevent blockage of the holes, and connected to the air pumps with 0.04 and  $6 \text{ m}^3 \text{ min}^{-1}$  capacity for run 1 and run 2, respectively.

For run 1, because of the low ambient temperature in the winter, the inlet air temperature was maintained in the range of 20–35°C by a resistance wire surrounding a 2-m long aluminum pipe connected to a temperature controller. For run 2, the air was intermittently supplied with a preset cycle of 1 min aeration and 59 min pause using an automatic time-delay relay. A schematic diagram of the composting system run 1 is presented in Fig. 3.1.

##### 3.2.1.3. Temperature recording

The temperature within the compost piles was continuously recorded every 5 min by thermocouples



which were horizontally placed at the top (0.40 m from the base), middle (0.25 m from the base), and bottom (0.15 m from the base) of the composting bins. Another thermocouple was placed outside to measure the ambient temperature. All temperature data were averaged to get mean daily values. Several data on day 17 and from day 55 to 63 in run 1 were not available due to the failure of the data logger.

#### 3.2.1.4. Sampling procedures

Compost mixtures were turned and samples were taken on days 0, 11, 28, 46 and 63 for run 1, and days 0, 4, 11, 21, 32, 46 and 56 for run 2. After mixing all materials well, about 100 g of homogeneous compost samples (excluding woodchips) were collected at five symmetrical locations for pH, NH<sub>4</sub>-N, NO<sub>3</sub>-N and FAAN measurements in fresh condition. The remaining mixtures were then immediately shifted back to each bin for continuous composting. Dried samples were then ground to a very fine powder for other measurements, i.e. total carbon, total nitrogen, total phosphorus and heavy metals. The sludge and finished compost samples were taken in triplicates while the others were taken singly.

#### 3.3.1.5. Sample analyses

The sludges and composts were analysed for the following parameters: moisture content (105°C for 24 h); pH (1:5 of fresh sample: water, w/v) using pH electrode; total C and total N were measured by C-N analyser (CORDER MT-700; Yanaco, Japan); volatile solids were determined by loss on ignition (550°C for 3 h); loss of organic matter (OM) and loss of TN were computed using the equations of Paredes et al. (2000):  $OM\ loss\ (\%) = 100 - 100 \frac{X_1(100-X_2)}{X_2(100-X_1)}$ ;  $TN\ loss = 100 - 100 \frac{X_1 N_2}{X_2 N_1}$ ; where,  $X_1$  and  $X_2$  represent the initial ash content and the ash contents at a time  $t$ , respectively;  $N_1$  and  $N_2$  represent the initial TN content and the TN content at a time  $t$ , respectively.

FAAN was extracted by shaking 5 g of fresh sample with 50 mL of deionized water, followed by centrifugation at  $8256 \times g$  (10000 rpm for 5 min) and storage of supernatant solution at -30°C to await analysis. The analytical procedure followed that of Carter et al. (2007). Briefly, 2mL of dissolved organic matter sample and 1.25 mL of ninhydrin reagent were added in sequence to test tubes. The tubes were capped, shaken and kept in the water bath at 95°C for 25 min. After cooling, 4.5 mL of ethanol (50% v/v) were added to the tubes and absorbance read at 570 nm on the spectrophotometer (UV-1200, Shimadzu, Japan). A standard curve was prepared from six different concentrations of L-Leucine-N (from 1 to 16 mg N L<sup>-1</sup>) and the results were expressed in terms of L-Leucine-N equivalents. Inorganic nitrogen was extracted by 2.0 M KCl (w: v = 1:5); NH<sub>4</sub>-N was measured by the salicylate-hypochlorite method (Tan, 1996) and NO<sub>3</sub>-N was measured by vanadium (III) chloride reduction method (Doane and Horwath, 2003).

Organic-N was calculated as the difference between TN and inorganic N (sum of  $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$ ). Total phosphorus and available P (Trough-P) were determined by molybdenum blue method; total cations (K, Ca, Mg) and total heavy metals (Zn, Cu, Mn, Pd, Cd) were determined using atomic absorption spectrometry (AA-6800, Shimadzu, Japan) after dry ashing samples and dissolving in 10 ml 50% v/v HCl (Faithfull, 2002).

### 3.2.2. Follow-up composting runs 3, 4 and 5

Three follow-up composting runs were prepared in pilot scale using different volumetric recipes of sludge: woodchips as bulking agent: 1:1 (aforementioned run 2), 1:0.5 (run 3), 1:0.2 (run 4) and 1:0 (run 5). Compressed air was also provided aeration through small holes perforated on two open-ended parallel PVC pipes ( $\varnothing$  16 mm, 0.25 m far apart) laid at the base and connected to an air pump (Yodogawa BN3  $\varnothing$  75 mm, JAPAN) with a supplying capacity of  $6 \text{ m}^3 \text{ min}^{-1}$  for all four composting bins. The details of schematic diagram of the composting runs 2, 3, 4 and 5 are illustrated in Fig. 3.2.

The performance of composting process was evaluated during 56 days of composting by measuring temperature, moisture, total volatile solid, pH, total carbon, nitrogen, C/N values, FAAN, inorganic nitrogen ( $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$ ), total P, total K, total Ca, total Mg and total heavy metals (Cu, Zn, Mn, Pb, Cd). The procedures for recording temperature and sampling compost were treated under similar conditions as those used in run 1. The matrix correlation between physicochemical parameters was analyzed using EXCEL<sup>®</sup> macro add-ins DAAASTAT version 1.512 (Onofri and Pannacci, 2014).

Table 3.1. Physicochemical properties of Japanese SS for composting runs 1–5 and the reference data

Parameters	Sludge 1	Sludge 2 <sup>†</sup>	V-sludge <sup>††</sup>
Moisture content (% d.b.)	473.4 (22.7)	337.7 (17.7)	178.2 (16.7)
pH (1:5, w/v)	7.5 (0.03)	7.3 (0.06)	6.9 (0.3)
Volatile solids (%)	77.6 (0.4)	82.0 (0.2)	66.6 (4.0)
Total C (g kg <sup>-1</sup> )	379.2 (3.4)	447.4 (20.1)	169.3 (0.7)
Total N (g kg <sup>-1</sup> )	58.7 (4.0)	48.9 (2.2)	22.4 (0.1)
C/N ratio	6.5	9.2	7.6
FAAN (g kg <sup>-1</sup> )	1.45 (0.08)	2.53 (0.35)	–
NH <sub>4</sub> -N (g kg <sup>-1</sup> )	2.39 (0.15)	3.57 (0.41)	4.24 (1.20)
NO <sub>3</sub> -N (g kg <sup>-1</sup> )	0.02 (0.01)	0.01 (0.003)	3.79 (0.62)
Total P (g kg <sup>-1</sup> )	14.3 (0.13)	10.8 (1.25)	7.6 (0.3)
Troug-P (g kg <sup>-1</sup> )	0.9 (0.07)	1.7 (0.17)	0.4 (0.03)
C/P ratio	26.5	41.4	22.3
Total cations (g kg <sup>-1</sup> )			
K	3.9 (0.1)	4.1 (0.1)	–
Ca	1.2 (0.1)	2.8 (0.1)	–
Mg	1.9 (0.1)	1.6 (0.5)	–
Total heavy metal (mg kg <sup>-1</sup> )			
Zn	757.2 (14.2)	973.8 (8.9)	209.6 (34.5)
Cu	380.5 (7.4)	226.2 (1.4)	45.7 (3.1)
Mn	185.0 (2.9)	152.5 (1.2)	372.1 (22.1)
Pb	3.5 (4.7)	41.0 (2.6)	nd
Cd	nd	1.7 (0.1)	nd

Values in parentheses are standard deviation (n = 3); nd, not detected

<sup>†</sup>Sludge 2 was also used for runs 2, 3, 4 and 5

<sup>††</sup>V-sludge: Vietnamese sludge was not used in subsequent composting experiments due to the feedstock limitation

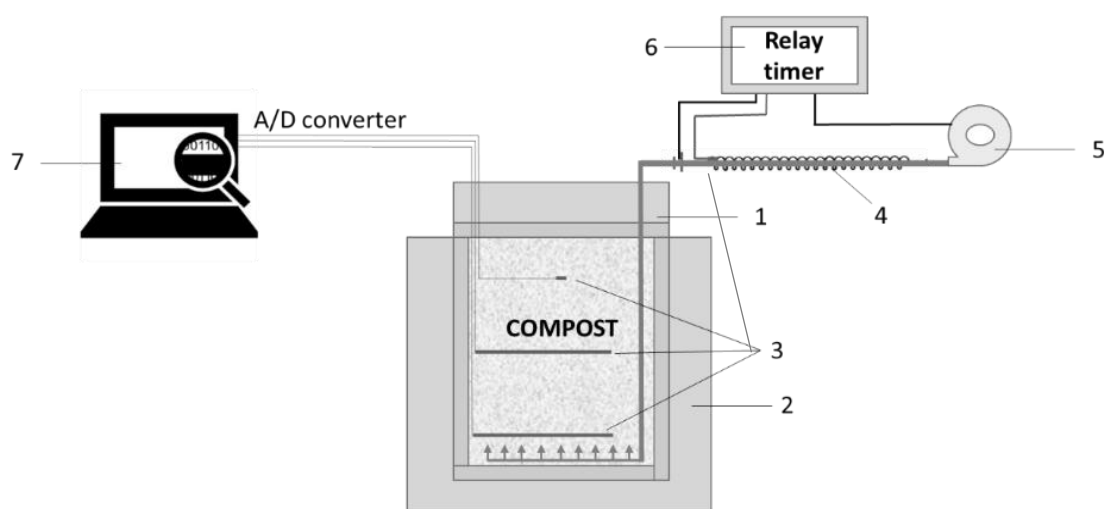


Fig. 3.1. Schematic diagram of the composting run 1 (in winter)

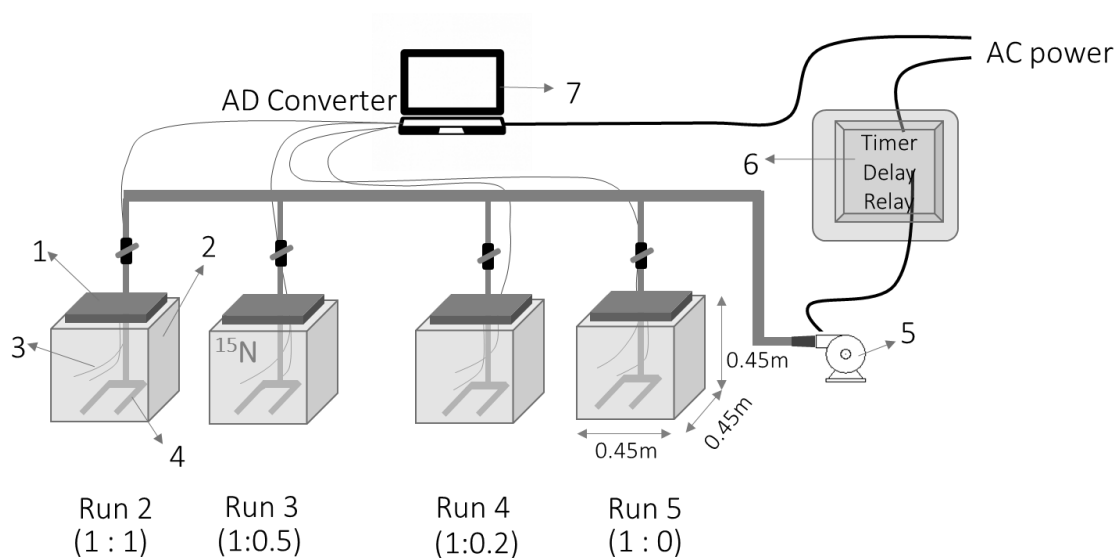


Fig. 3.2. Schematic diagram of the composting run 2, 3, 4 and 5 (in summer)

Note: 1- bin lid, 2- styrofoam insulator, 3- thermocouples, 4- parallel PVC  $\varnothing = 16\text{mm}$  with perforation of  $\varnothing = 3\text{mm}$ , 5- air pump, 6- timer delay relay, 7- PC notebook & data logger

### 3.3. Results and discussion

#### 3.3.1. Effect of seasonal temperature on composting performance

##### 3.3.1.1. Compost temperature

Compost temperature is a very important parameter indicating the success or failure of a composting process. The ambient temperatures ranged from 4.3°C to 18.2°C in run 1 and from 25.0°C to 33.4°C in run 2 (Fig. 3.3). In run 1, the temperatures immediately increased after composting and reached 47.4°C, 39.1°C, and 32.7°C on day 2 in the top, middle and bottom locations, respectively. In run 2, peak temperatures occurred later on day 3 and reached higher values of 43.8°C, 74.8°C and 55.7°C in the top, middle and bottom locations, respectively. After peaking, the temperature gradually cooled down and somewhat returned to the ambient temperature. At some locations, i.e. run 1-bottom or run 2-middle, compost temperatures were found to increase again. At the end of the process, the compost temperatures ranged from 9.4°C to 19.1°C in run 1 and from 19.7°C to 55.3°C in run 2 (Fig. 3.3).

##### 3.3.1.2. Moisture content

It should be noted that our moisture data are expressed on a dry weight basis which emphasizes the changes of mass of water compared to their initial dry mass of solids. The sludge used in these experiments had high moisture contents (473.4% in sludge 1, and 337.7% in sludge 2). During composting, the moisture contents gradually decreased to 118.8% in run 1 and 74.0% in run 2 as a consequence of common evaporation of water. Despite having a higher initial moisture content, the loss of moisture in run 1 was greater than run 2 (Table 3.2).

##### 3.3.1.3. pH

The composts had an initial pH value of 7.5 (run 1) and 6.6 (run 2), which rapidly increased to maximum values of 8.6 for run 1 and 9.0 for run 2 within 4-11 days (Table 3.2). After that, the pH gradually decreased until the end of the composting process. The difference in the pH values between two finished composts was about 1-unit pH (5.7 in compost 1 and 6.8 in compost 2). Both sludges were expected to be compatible with Komatsuna plant in the later pot experiment.

##### 3.3.1.4. Total volatile solids, organic matter loss and total nitrogen loss

Total volatile solids, OM and TN loss indicated the degradation of organic matter. The total volatile solids substantially decreased from 75.2 to 69.2% in run 1 and from 83.9 to 69.2% in run 2. Most of TN losses occurred during the first 11 days, while OM loss extended longer in later periods as found in run 2. At the end of composting, the percent of OM losses were 25.7% and 50.2% in run 1 and run 2, respectively. Similarly, after composting, TN losses were 32.8% in run 1 and 44.4% in run 2 (Table 3.2).

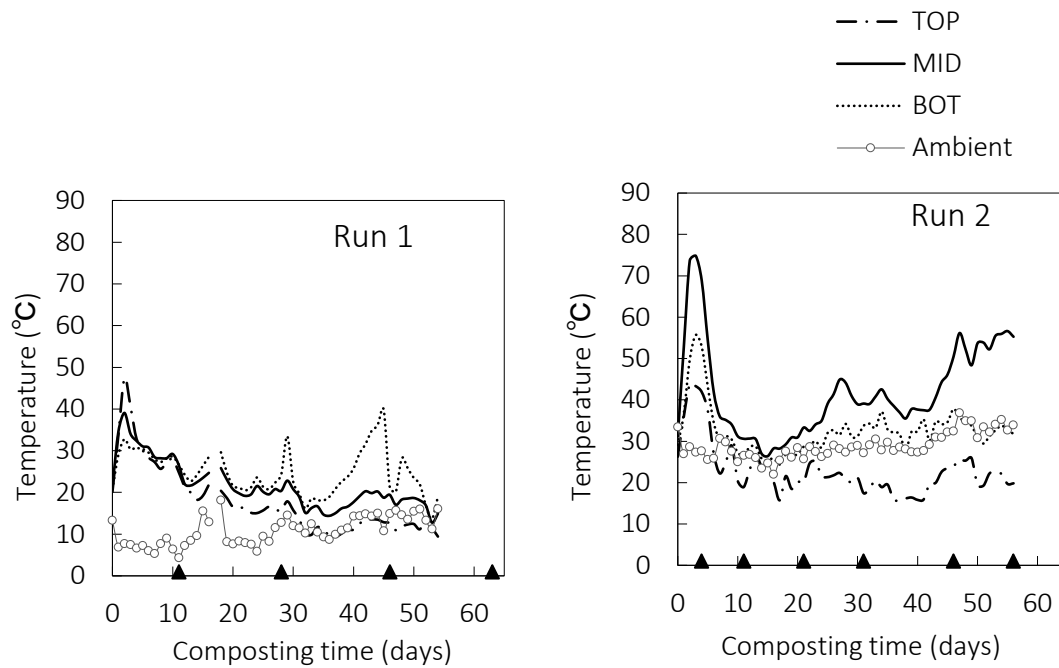


Fig. 3.3. Temperature profiles at the top, middle, bottom of the compost and ambient for run 1 and run 2. Black triangles (▲) on the x-axis indicate times for turning/sampling events

#### 3.3.1.5. Total carbon, total nitrogen and C/N ratios

Microorganisms use carbon as a source of energy and nitrogen for their population growth. In both sludges TC and TN changed as a result of OM and TN losses during composting. The total C decreased from initial values of 380.8 to 329.3 g kg<sup>-1</sup> in run 1, and from 414.2 to 331.7 g kg<sup>-1</sup> in run 2 at the end of the process (Table 3.2). Similarly, TN decreased from 60.8 to 50.6 g kg<sup>-1</sup> in run 1 and from 51.6 to 46.2 g kg<sup>-1</sup> in run 2 (Table 3.2 and Fig. 3.4). Consequently, C/N ratios of the finished composts, which were 6.5 in run 1 and 7.2 in run 2, showed little change during the whole process. Nevertheless, the slight increase in C/N ratio from 8.0 to 10.8 found in run 2 during the first 11 days indicated that the loss of nitrogen exceeded the loss of carbon during the same period of time.

#### 3.3.1.6. Inorganic nitrogen (NH<sub>4</sub>-N and NO<sub>3</sub>-N)

The initial NH<sub>4</sub>-N contents in run 1 and run 2 were 3.63 and 10.73 g kg<sup>-1</sup>, respectively. For run 1, the NH<sub>4</sub>-N content increased dramatically to 18.9 g kg<sup>-1</sup> by day 11, thereafter, dropped to 6.2 g kg<sup>-1</sup> by day 28 and fluctuated between 7.8 and 8.7 g kg<sup>-1</sup> from day 46 to day 63. For run 2, the NH<sub>4</sub>-N also increased rapidly to 24.3 g kg<sup>-1</sup> by day 4, but then gradually decreased to 5.8 g kg<sup>-1</sup> at the end of process (Fig. 3.4). In contrast to the trend of changes in NH<sub>4</sub>-N, NO<sub>3</sub>-N contents showed a gradual increase in the latter stages when temperatures have been reduced to ambient levels. It should be noted that the increase of NO<sub>3</sub>-N in run 1 was much greater than run 2. As a result, the final amounts of NO<sub>3</sub>-N were also clearly

different in the two runs. By the end of the process, the  $\text{NO}_3\text{-N}$  concentration in compost 1 was 13-fold higher than that in compost 2 (Fig. 3.4). In addition, the  $\text{NH}_4\text{-N}/\text{NO}_3\text{-N}$  ranged from 254.3 to 1.0 in run 1 and from 923.3 to 8.1 in run 2 (Table 3.2).

Table 3.2. Changes in physicochemical properties during composting in runs 1 and 2

Composting time (days)	Moisture content (%, d.b.)	pH	Volatile solids (%)	OM loss (%)	TN loss (%)	Total C (g kg <sup>-1</sup> )	Total N (g kg <sup>-1</sup> )	C/N ratio	$\text{NH}_4/\text{NO}_3$ ratio
Run 1									
0	470.8	7.5	75.2	0.0	0.0	380.8	60.8	6.3	254.3
11	331.1	8.5	70.2	22.0	30.9	347.2	50.3	6.9	179.8
28	265.2	6.8	—	—	—	—	—	—	6.4
46	175.6	6.3	69.5	24.5	30.4	337.4	52.1	6.5	1.6
63	118.8	5.7	69.2	25.7	32.8	329.3	50.6	6.5	1.0
Run 2									
0	299.9	6.6	83.8	0.0	0.0	414.2	51.6	8.0	923.3
4	249.6	9.0	76.9	26.3	34.6	384.7	40.7	9.5	1239.2
11	203.1	8.9	77.2	25.1	42.9	381.7	35.2	10.8	1390.6
21	222.2	8.5	77.7	23.1	38.7	362.1	37.0	9.8	4602.1
32	168.5	7.5	72.8	40.7	39.1	350.0	44.7	7.8	282.5
46	102.0	6.7	70.0	48.3	44.0	335.2	45.3	7.4	13.8
56	74.0	6.8	69.2	50.2	44.4	331.7	46.2	7.2	8.1

### 3.3.1.7. Free amino acid-N

Amino acids are nitrogen-containing organic compounds that contribute significantly to organic matter. In this study, the changes in FAAN were measured to assess biodegradation during the composting process. In run 1, the amount of FAAN markedly decreased from an initial value of 1.45 to 0.61 g kg<sup>-1</sup> during the first 11 days, and then maintained a steady level until the end of composting. In run 2, the amount of FAAN was found to increase sharply during the first 4 days, from 2.43 to 3.77 g kg<sup>-1</sup>, then gradually decreased until the end of the composting process. The finished composts had the FAAN contents in the range of 0.57–0.65 g kg<sup>-1</sup> (Fig. 3.4).

In short, except for  $\text{NO}_3\text{-N}$ , most of the physicochemical changes occurred during the first 30 days of composting. During this period, the results indicated that the degradation of organic matter into amino acids and the transformation from amino acids to  $\text{NH}_4\text{-N}$  via synthesis–ammonification were

predominant in compost run 2, whereas, the nitrification in run 1 was found to be more intense after day 30.

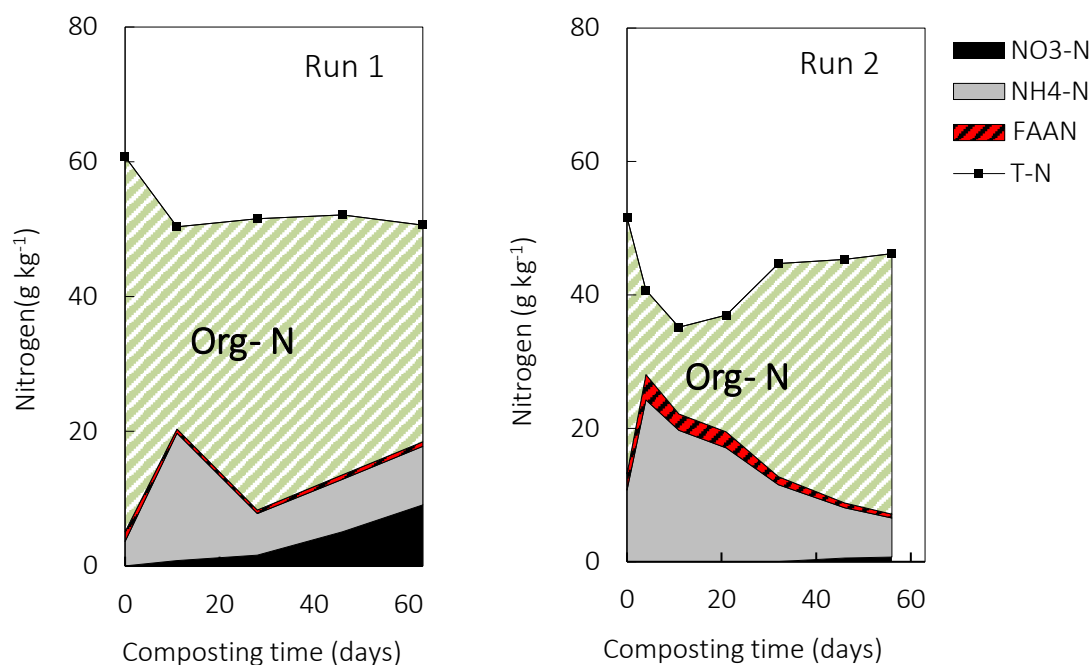


Fig. 3.4. Changes in nitrogen forms during composting process. Areas of diagonal stripes indicate values of organic nitrogen. Values are means of replicates ( $n = 6$  for run 1 and  $n = 2$  for run 2)

### 3.3.1.8. General discussion

The results of the current study demonstrated that the ambient temperatures affected the changes in physicochemical properties during the composting process. The peak temperatures achieved under low ambient temperature condition were lower than those in the high ambient temperature. The rapid increase in compost temperature in the initial phases of the composting process was associated with decomposition of the organic materials. The temperature increase in later periods could be due to the turning immature materials to desirable locations for consecutive decomposition of organic matter. The continuous introduction of warm air from the bottom was aimed to supply oxygen and prevent heating loss. This air supply directly affected convection heat currents moving upward and caused an atypical distribution of temperature in the compost in run 1. In contrast, the intermittent introduction of air in run 2 possibly produced the usual distribution of “self-heating” caused by microbial activities. The distribution of temperature in run 2 was consistent with other literature (Tiquia and Tam, 2000; Larney and Olson, 2006).

The optimum temperature range for composting SS in a forced aeration system to achieve the maximum rate of biodegradation is between 35°C and 55°C (McKinley and Vestal, 1985). However, the



compost temperature above 70°C would result in slower decomposition and nitrogen loss especially at low C/N ratios (Cofie et al., 2009). Therefore, in order to achieve an acceptable level of pathogen destruction in composting of SS, maintaining a minimum temperature of 55°C for at least three consecutive days is necessary (Burge et al. 1978). Except for a short temperature rise in the middle – bottom portions of run 2, both runs would not meet above criteria because of possible heat losses to the surroundings due to the insufficient insulation of the compost bins, especially under low air temperature condition as seen in run 1. Wang et al. (2013) used mathematical models to study the feasibility of SS composting in cold climate environments. Their results indicated that low ambient temperatures could be one of the reasons of heat losses that increased the heat transfer rate to the surroundings and it was difficult to maintain a desirable period of the thermophilic condition. The heat loss would also occur when the compost is being turned frequently preventing the compost temperatures from reaching optimum temperature (Tiquia et al., 1998).

Moisture content was documented to be more influential for microbial activity than temperature (Liang et al., 2003). A moisture content of 60–70% on wet basis (or 150–233% on dry basis) provided maximum microbial activities. Another reason for not achieving the temperature above 55°C in run 1 could be the higher initial moisture contents in its raw material, suggesting the requirement for drying of sludge to achieve to initial optimum moisture content. In aerobic conditions, the increase in pH at the beginning of the process is the result of degradation of proteins, amino acids and peptides that release ammonium or volatile ammonia (Said-Pullicino et al., 2007; Gigliotti et al., 2012). The drop in pH over time is a consequence of activity of acid-forming bacteria that break down complex compounds into organic acids in the finished product (Diaz et al., 2007).

In spite of the fact that OM loss and TN loss in run 2 were much higher than run 1, the results of TN losses measured during the composting of SS in the current study were consistent with those reported by Witter and Lopez-Real (1987). This could be explained by sludge 2 may have contained more FAAN than sludge A, resulting in this difference. Although two sludges originated from the same WWTP, they had different organic fractions depending on the season of the year. In addition, the higher compost temperature achieved in run 2 also contributed to greater reductions of volatile solids, greater OM and TN losses.

The decline in total C during aerobic composting was attributed to the mineralisation of organic matter resulting in the evolution of CO<sub>2</sub>, H<sub>2</sub>O and heat (Garcia et al. 1991; Li et al. 2013). Meanwhile, the decrease in TN was due to the loss of N as NH<sub>3</sub> as reported by Witter and Lopez-Real (1987). However, an increase in TN in from day 11 to day 32 as found in run 2 indicated a great reduction of the total weight and total volume of the compost mass.

The C/N ratio in solid phase was traditionally used to determine the degree of maturity (Iglesias Jiménez and Perez Garcia, 1989). The optimum range in C/N ratios from 25 to 35 would be

recommended for several types of commercial composts because most bacteria need approximately 30 g of C for 1 g of N uptake (Cofie et al., 2016). However, there is no general agreement regarding which value of the C/N ratio indicates maturation of SS compost due to the relative N-richness of the feedstock. Instead of C/N ratio in solid phase, some authors suggested that the C/N ratio in water extract could be a better predictor of compost stability (Chanyasak and Kubota, 1981; Hue & Liu, 1995). The results show that finished composts had smaller C/N ratios (6.5–7.2) than the optimum range, but they were not significantly different from other Japanese commercial composts based SS (data not shown). In this study, woodchips served as a bulking agent during whole composting process. The woodchips properly increased the initial C/N ratios of the feedstock to achieve better performance for composting. Malińska and Zabochnicka-Świątek (2013) indicated that mixing SS with woodchips at a ratio of 1:1 (d.b.) allows the optimal initial moisture content of 69% (w.b.), C/N ratio of 30:1 and air-filled porosity of 52% across the composting pile. Although being screened at the end of process, the woodchips could not be separated completely from the finished composts. Consequently, they might have contributed to the carbon source of the finished composts.

The increase in  $\text{NH}_4\text{-N}$  with the increase in temperature and pH during the first 4–11 days reflected the degradation of organic matter (Brewer and Sullivan, 2003). After an initial increase,  $\text{NH}_4\text{-N}$  contents decreased because of immobilisation inorganic N into organic forms as humus-like materials. However, the decline in  $\text{NH}_4\text{-N}$  during the first 30 days was not associated with an increase of  $\text{NO}_3\text{-N}$  as reported by Paré et al. (1998). The continuous introduction of inlet air at a very slow rate in run 1 might have induced a nitrification process in which  $\text{NH}_4\text{-N}$  was assimilated to produce the  $\text{NO}_3\text{-N}$  form. In run 2, the  $\text{NO}_3\text{-N}$  concentration was negligible during the first 21 days because nitrifying bacteria were likely to be inactive due to the excess amount of  $\text{NH}_4\text{-N}$  as an inhibitor (Bhoyar et al. 1979; Fang et al. 1999). Thus, the  $\text{NO}_3\text{-N}$  and  $\text{NO}_2\text{-N}$  contents were higher under better aerobic condition (Brouillette et al., 1996) and nitrification hardly occurs under thermophilic conditions (Morisaki et al. 1989). The decrease in  $\text{NH}_4\text{-N}$  combined with the increase in  $\text{NO}_3\text{-N}$ ; on the other hand,  $\text{NH}_4\text{-N}/\text{NO}_3\text{-N}$  decreased over time suggesting that compost has reached maturity (Paré et al., 1998). The optimum values of  $\text{NH}_4\text{-N}/\text{NO}_3\text{-N}$  ranged from 0.3 to 3.0 suggesting the maturity level of compost, whereas levels of above 3.0 may reveal an immature condition (Brinton, 2000, as cited in Cofie et al. 2016).

To our knowledge, no studies regarding to composting of SS indicated an optimum level of FAAN in the finished compost. This leads us to consider that measuring of this parameter might have potential to assess the compost stability and maturity, although our data presented herein are insufficient to suggest whether FAAN can be used as a reliable indicator. The overall decrease in FAAN at the end of the process, as reported above, suggested that it was gradually assimilated by microbes. Thus, the degradation of organic matter that produces amino acids was not counterbalanced by destructive degradation of amino acids with increasing composting time. Baca et al. (1994) indicated the changes in

the amino acid composition reflected the changes in the composition of the microbial population. In their study, amino acids were divided into four groups: acidic, basic, neutral and sulphur. The total acidic amino acids of the four groups decreased by about 36% of the initial value during 90 days of composting. A general decrease in the amounts of free amino acids during humification of organic matter was also reported by Lähdesmäki & Piispanen (1989).

The higher amount of FAAN in the immature sludge 2 could be one of the causes of the phytotoxicity effect. In that case, the plant could not use available nitrogen sources due to blockage of soil available nitrogen by microbial activities (see section 5.3.2.2).

#### 3.3.1.9. Variation in sludge properties and composting applicability in Vietnam

As given in Table 3.1, Vietnamese sludge (V-sludge) had a lower moisture content, volatile solids, total C, TN, TP, exchangeable cations, total Zn and Cu but higher  $\text{NO}_3\text{-N}$  and Mn than Japanese sludge, depending on the variation of wastewater sources and wastewater treatment systems. Even though originating from the same WWTP, sludge properties can also fluctuate seasonally. For example, at times of rainstorms, the flow of sewage may be too high to be accommodated by the downstream treatment stages. In that case, the sludge might contain more resistant soil particles like silica, that caused lower volatile solids and nutrient contents as seen in V-sludge properties.

However, such differences are thought not to affect the composting process, because basically, V-sludge contained similar proportions of C : N : P in comparison to Japanese sludge, which are maybe more important than the nutrient concentrations.

In Vietnam, composting is not a common practice due to a number of reasons. These include high operation and maintenance cost, inadequate management of the composting process that caused poor performance or low-quality compost. This is likely due to an insufficient control of temperature, moisture, aeration or a combination of the above factors. A wide variation in sludge constituents leads to a wide variation in compost quality, therefore, its utilization of different composts must be adjusted accordingly to ensure beneficial results.

#### 3.3.2. Effect of volumetric ratios of sludge and woodchips ratios

##### 3.3.2.1. Compost temperature, moisture content and pH

The changes in temperature over time as a function as affected by sludge: woodchip ratio are shown in Fig. 3.5A. The temperature profiles associating with vertical positions have been discussed in the previous section 3.3.1. The temperature values in this paper, however, are means of three locations in the top, middle and bottom of the compost bins. The ambient air was maintained between 25°C and 33°C. After composting the compost temperatures immediately increased to peak points of 65°C, 59°C,

51°C and 46°C during day 2–3 in run 2, run 3, run 4 and run 5, respectively. Thus, the compost temperatures were clearly affected by different sludge: woodchip ratios, in which the higher the portion of woodchips occupied, the higher temperature the compost can reach. During active phase, the compost temperatures were much higher than ambient temperatures indicating self-heating capacity in the compost piles. The rise in temperature was due to heat generated by the microbial activities and the insulation against heat loss provided by composting mass (Diaz et al., 2007). After peaking, the temperatures dropped quickly and were close to the ambient temperature on by day 11. From day 11, composts were thought to enter the curing phase. Compost temperatures slightly increased again from day 32 to day 46, and were maintained or decreased until the end of composting process. Such an increase again during later periods was maybe due to the increase of ambient or the turning effect that caused continuous decomposition of immature material (Tiquia et al., 1998). The temperature above 55°C for three consecutive days was observed in run 2 and run 3 to meet the requirements for reduction of fecal pathogens. However, Kuter et al. (1985) also emphasized that high temperature greater than 60°C may inhibit the activity of the composting microorganisms. These results, therefore, support the strategy for controlling of aeration process, in which temperature feedback control was useful to maintain the effective composting rates.

The moisture contents gradually decreased as a consequence of common evaporation of water at high temperature. At the beginning stage of composting, the lids of compost bin were closed until day 31 and the favored water vapor condensation returned to the compost piles. The bulking agent was not only to maintain the structural integrity, increase the porosity, but also to absorb some moisture. This information can explain why the drop being faster in run 2 than other runs from day 31 where the compost temperature was found to be higher and the portion of woodchip was also greater. However, the effects of sludge: woodchips as a bulking agent on moisture were not clear between run 3, run 4 and run 5. Ideally, the moisture content within the compost pile should be between 40–50% in wet basis or 67–100 % in dry basis (d.b.). A moisture content of above 100% (d.b.) is difficult to screen while that of below 43% (d.b.) creates more dust. As a result, the moisture contents in this current study were in a permissible range of 79 to 113% (Fig. 3.5B).

#### 3.3.2.2. pH, total volatile solids, organic matter loss and total nitrogen loss

The pH values of all treatments sharply increased to 9 during the first 4 days, fell gradually from day 4 to day 46 and then remained stable at around 7 until the end of composting process (Fig. 3.5C). Four compost runs had a similar trend of changes in pH values and were not affected by different sludge: woodchip ratios. As shown in Fig. 3.5D and Fig. 3.5E, the percent of volatile solid generally decreased and organic matter loss increased with composting time because of biodegradation of organic matter.

Volatile solids in run 2 at day 0 were a little bit greater than the others (84 % vs 80%) perhaps due to the contribution of more degradable carbon fractions from woodchips although woodchips were discharged during sampling.

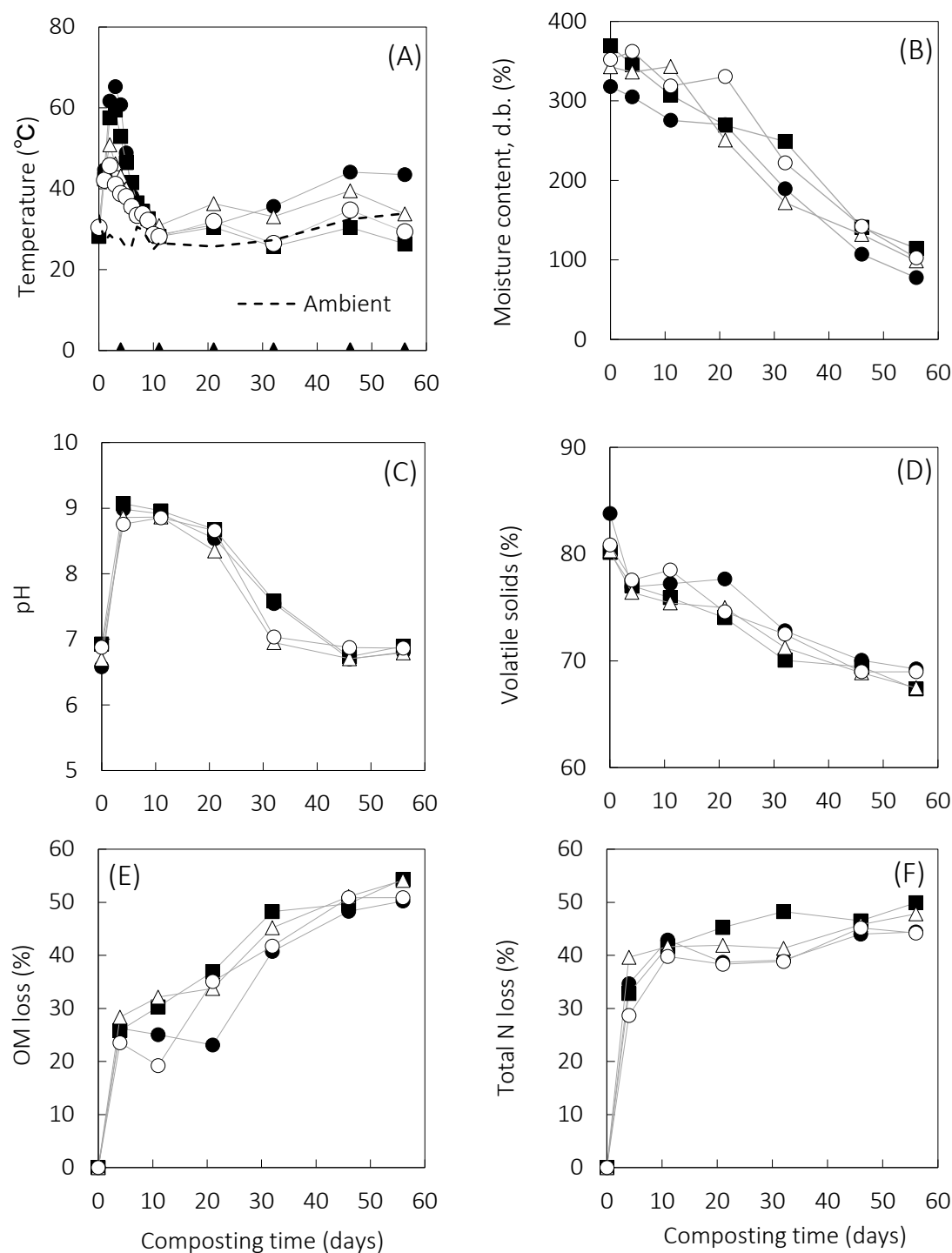


Fig. 3.5. Changes in temperature (A), moisture (B), pH (C), volatile solid (D), organic matter loss (E) and total nitrogen loss (F) as affected by sludge: woodchips recipes. Data on run 2 have been reported in section 3.3.1. Black triangles (▲) on the x-axis (A) indicate times for turning/sampling events. (● = run 2; ■ = run 3; △ = run 4, ○ = run 5)

In general, the difference in sludge: woodchips did not affect clearly on the pH, VS, OM loss and TN loss. Or on the other hand, the addition of bulking agent as woodchips to SS might not help reduce the loss of N. Concerning the nitrogen loss during composting process, Witter and Lopez-Real (1988) indicated that all nitrogen virtually all losses resulted from ammonia volatilization in the initial stages of composting. At the end of composting process, about 50-54 % total OM losses and 44–48 % of total nitrogen were lost (Fig. 3.5E and Fig.3.5F). These values are close to the nitrogen losses for the composting of SS (50%) as normally reported by other authors (Witter and Lopez-Real, 1988; Roig et al., 2001).

#### 3.3.2.3. Total carbon, total nitrogen and C/N ratios

The changes in total carbon, total nitrogen and C/N ratios are shown in Fig. 3.6A, Fig. 3.6B and Fig. 3.6C, respectively. Total carbon decreased with composting time for all treatments while total nitrogen sharply decreased during the first 11 days, increased from day 11 to day 32, and then maintained stable until the end of process. At the end of composting, the decline in carbon accounted for 20–24 % of initial total carbon, which shows the decomposition of organic matter. A part of the carbon in the decomposing materials evolved as CO<sub>2</sub> and a part was assimilated by the microbial biomass (Cabrera et al., 2005).

The decrease in TN in the first period could be due to volatilization of ammonia during composting as reported by (Morisaki et al., 1989). This result was consistent with the rise of temperature, pH and loss of total nitrogen as mentioned above. In contrast, an increase in TN from day 11 to day 32 suggested a great reduction of the total volume or dry mass, which coincided with those reported by Fang et al. (1999).

The C/N ratios increased and then decreased depending on the change in total carbon and total nitrogen values. At the end, C/N ratios of the finished compost were 7.2, 7.3, 7.1 and 7.1 in run 2, run 3, run 4 and run 5, respectively. In general, their values did not change so much compared to initial values. The microorganisms need about 25 to 40 times more carbon than nitrogen. The small decline in the C/N ratio in this study may also be attributed to SS components, such as low molecular fatty acids resulting in the inhibition of microbial metabolism (Margesin et al., 2006). However, the relatively narrow C/N ratios in finished composts may reveal a fast mineralisation after soil application.

#### 3.3.2.4. Free amino acid-N, NH<sub>4</sub>-N and NO<sub>3</sub>-N

The changes in FAAN, NH<sub>4</sub>-N and NO<sub>3</sub>-N are shown in Fig. 3.6D, Fig. 3.6E and Fig. 3.6F. The FAAN in run 2 sharply increased during first 4 days, dropped and gradually decreased till the end of composting. Free amino acids in organic N compounds were measured to assess biodegradation during the composting process. The amount of FAAN was found to increase sharply during the first 4 to 11 days, then gradually

decreased in later periods showing a similar trend to the results of  $\text{NH}_4\text{-N}$ . No significant difference was found between treatments in term of FAAN in finished composts ( $P > 0.05$ ). The overall decrease in FAAN at the end of the process has been reported in the previous chapter. The decline in FAAN was explained by the non-counterbalanced with composting time between produced and destructive amino acids from degradation of organic matter and assimilation by microorganism activities. The changes in FAAN were in agreement with Lähdesmäki & Piispanen (1989), who indicated that the general tendency for the amounts of amino acids to decrease was found with advancing humification. with the notable exceptions of proline, ornithine, arginine and  $\gamma$ -aminobutyric acid. Although the measurement of FAAN is rather preliminary, these results seem to be potential as one of the useful indices for checking composting process.

The  $\text{NH}_4\text{-N}$  contents of all treatments sharply increased during the first 4 days due to the fast ammonification, which coincided with the rise in compost temperature and pH values. After an initial increase, the  $\text{NH}_4\text{-N}$  contents gradually decreased to day 32 and maintained stable until the end of the process. Fang et al. (1999), Witter and Lopez-Real, (1988) indicated the decline in  $\text{NH}_4\text{-N}$  was due to the volatilization loss as  $\text{NH}_3$  and/or the immobilisation of  $\text{NH}_4\text{-N}$  into stable organic forms because no nitrate appeared during the active phase. The addition of  $(^{15}\text{NH}_4)_2\text{SO}_4$  in run 3 was likely to cause a slight decline in  $\text{NH}_4\text{-N}$  on day 4 through immobilisation of inorganic nitrogen to organic forms (see section 6.2.2). However, the trend of changes in  $\text{NH}_4\text{-N}$  between different treatments until the end of composting was found to be similar. The finished composts had  $\text{NH}_4\text{-N}$  in range of 5.4–7.1 g  $\text{kg}^{-1}$  and no significant differences were found between different treatments ( $P > 0.05$ ).

In contrast to the  $\text{NH}_4\text{-N}$  pattern, the  $\text{NO}_3\text{-N}$  contents were almost absent during the first 30 days maybe because of the inhibition of nitrifying bacteria caused by the excessive amount of  $\text{NH}_4\text{-N}$  at high temperature (Fang et al., 1999). The  $\text{NO}_3\text{-N}$  contents on day 46 and day 57 were significantly affected ( $P < 0.05$ ) by different sludge: woodchips ratios. For example, at the end of composting, the  $\text{NO}_3\text{-N}$  contents for run 2, run 3, run 4 and run 5 were 0.7, 0.3, 0.2 and 0.5 g  $\text{kg}^{-1}$ , respectively. In the preliminary experiment on the composting of SS using a simple aeration method in the high air temperature and low air temperature with the support of heating system. The results showed that the continuous introduction of warm air might have induced a nitrification process in which  $\text{NH}_4\text{-N}$  was assimilated to produce the  $\text{NO}_3\text{-N}$  form. Thus, the high  $\text{NO}_3\text{-N}$  contents in run 2 could be due to the higher aeration capacity caused by higher portion of woodchip, while those in run 5 (control treatment without woodchips) was perhaps associated with a lower temperature that reduced inhibition effects of nitrifying bacteria. Gómez-Brandón et al. (2008) indicated that the compost stability can be determined based on the nitrification when the temperature of compost is close to the ambient temperature. Therefore, except for temperatures in run 4 and run 5 which have not yet met the requirement for acceptable composts, the stability of run 2 and run 3 can be reached.

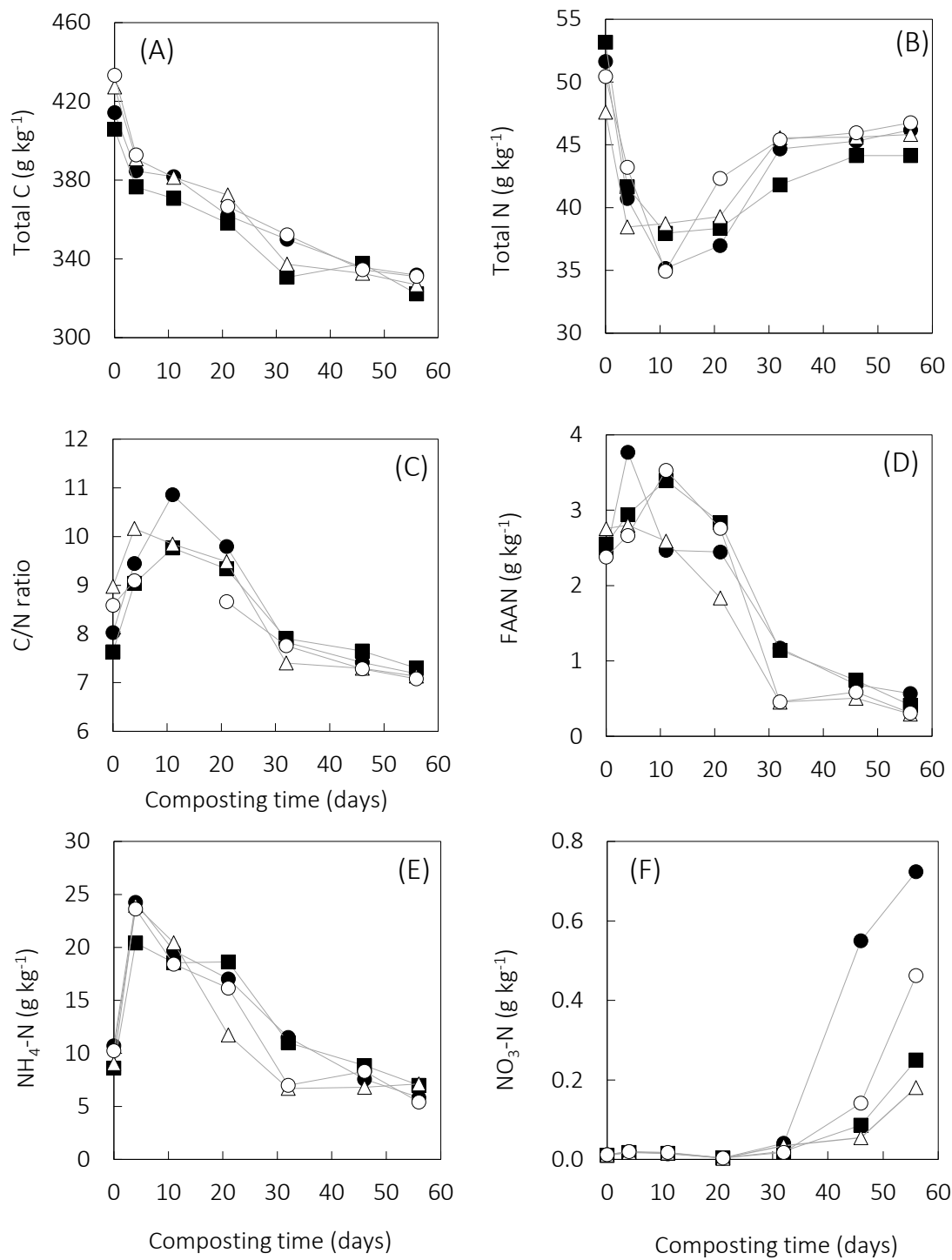


Fig. 3.6. Changes in total carbon (A), total nitrogen (B), C/N ratio (C), free amino acid-N (D),  $\text{NH}_4\text{-N}$  (E) and  $\text{NO}_3\text{-N}$  (F) as affected by sludge: woodchips recipes. Data on run 2 have been reported in section 3.3.1. (● = run 2; ■ = run 3; △ = run 4, ○ = run 5)



#### 3.3.2.5. Total phosphorus, potassium, calcium and magnesium

Some other nutrient contents of sludge during composting process are given in Table 3.3. The change of total K, total Ca and total Mg followed the similar trend to total P with a slight increase throughout the composting time due to the loss of dry mass. Available phosphorus was about 13% of total P for initial sludge and about 10% for finished composts. It is noteworthy that macronutrients (P, K, Ca and Mg) were not affected by different sludge: woodchip ratios. Total K was relatively low compared to total P and total N in both sludge and composts due to the solubility during waste water treatment process (Moretti et al., 2015). The values of total P, K, Ca and Mg in SS were in the range of those reported by (Tan, 2000).

#### 3.3.2.6. Heavy metals (Cu, Zn, Mn, Pb and Cd)

The heavy metal contents in SS varied considerably among treatments perhaps due to the non-homogeneous characteristics of the raw material (Table 3.4). At the end of composting process, total Zn, Pb and Cd in run 2 and run 3 were lower than those in run 4 and run 5, while total Cu and total Mn in run 2 and run 3 were found to be higher, respectively. In general, sludge and composts were very high in Zn and Cu, lower in Mn and relatively small in Pb and Cd. However, the results showed that composting did not remove heavy metals from raw materials. On the other hand, these heavy metals increased in concentration because of the loss in weight of the organic as noted by (Tan, 2000). In Japan, Zn content in sludge and SS compost is used as a reference to evaluate the quality standards defined by the fertiliser law. Kumazawa (1997) indicated that the upper limits for Zn, Cu and Cd in SS and composted SS were 1800, 600 and 5 mg kg<sup>-1</sup>, respectively. Therefore, the values of some heavy metals measured in this study were below the limit for agricultural use.

#### 3.3.2.7. Correlation analysis

The correlation coefficients (*r*) for the physical-chemical parameters during composting process are given in Table 3.5. Interestingly, most of observed parameters are very strongly correlated to composting time, moisture content, volatile solids, organic matter loss, FAAN, total Ca, Mg, Cu, Zn and Mn while less associated with pH, total C, total N, C/N, NH<sub>4</sub>-N, NO<sub>3</sub>-N and temperature. For example, moisture content, volatile solid, FAAN and NH<sub>4</sub>-N were significantly negatively ( $P < 0.05$ ), while organic matter loss, TN loss, NO<sub>3</sub>-N and most of the non-volatile nutrients were significantly positively ( $P < 0.05$ ), correlated with composting time.

Thus, shortening the composting time can reduce nitrogen loss but products remain high moisture content, high organic matter content and FAAN indicating immature indicators. The moisture contents of compost decreased with increasing organic matter loss, Ca, Mg and most of the heavy metals as the results evaporation and the loss in total volume of the waste, respectively. The very strong positive

correlation between pH and  $\text{NH}_4\text{-N}$  ( $r = 0.93$ ), between  $\text{NH}_4\text{-N}$  and FAAN ( $r = 0.83$ ) is the most noteworthy results indicating the biodegradation of organic matter into amino acids followed by ammonification and the increase in pH values due to  $\text{NH}_3$  volatilization. There was no apparent correlation between temperature evolution and other parameters (data not shown). This suggested that the biodegradation of SS appear to be relatively well-buffered against other measured parameters perhaps due to the heat losses to the surroundings under small-scale compost bins as reported in the previous chapter.

Table 3.3. Changes in total phosphorus, total potassium, total calcium and magnesium as affected by sludge: woodchips recipes

Composting time (days)	Total P ( $\text{g kg}^{-1}$ )				Total K ( $\text{g kg}^{-1}$ )			
	Run 2	Run 3	Run 4	Run 5	Run 2	Run 3	Run 4	Run 5
0	13.0	14.2	12.7	12.5	5.5	5.3	4.9	6.3
4	16.0	16.2	15.2	15.5	5.4	5.2	5.2	5.1
11	14.7	27.0	15.0	14.4	5.2	6.1	4.6	4.5
21	16.8	18.7	16.7	17.8	5.2	7.1	5.2	5.5
32	20.8	19.6	20.3	18.9	5.9	6.3	5.7	5.6
46	22.8	22.5	21.0	21.8	5.8	6.7	5.8	6.4
56	20.0	19.8	18.6	18.4	6.4	7.3	7.0	6.7
	Total Ca ( $\text{g kg}^{-1}$ )				Total Mg ( $\text{g kg}^{-1}$ )			
	Run 2	Run 3	Run 4	Run 5	Run 2	Run 3	Run 4	Run 5
0	8.1	8.9	10.3	10.6	1.9	2.2	1.7	1.7
4	11.2	10.5	11.1	10.8	3.0	3.2	2.5	2.5
11	10.8	10.9	11.7	12.7	3.7	3.0	2.1	1.8
21	10.5	11.7	13.0	12.3	2.9	3.4	2.7	2.6
32	13.4	12.6	12.2	12.2	4.2	4.1	3.6	3.2
46	13.7	13.5	13.7	13.1	4.8	4.7	4.1	3.8
56	13.7	13.0	14.1	13.7	4.9	4.8	3.8	3.8

Table 3.4. Changes in total heavy metals (Cu, Zn, Mn, Pb and Cd) as affected by sludge: woodchips recipes

Composting time (days)	Total Cu (mg kg <sup>-1</sup> )				Total Zn (mg kg <sup>-1</sup> )			
	Run 2	Run 3	Run 4	Run 5	Run 2	Run 3	Run 4	Run 5
0	262	258	242	305	589	818	967	937
4	369	333	299	358	931	886	982	870
11	283	316	346	310	961	950	1017	1056
21	336	357	341	350	883	1019	1109	1094
32	444	394	406	406	1083	1048	1197	1191
46	442	463	443	441	1140	1148	1239	1208
56	341	517	379	409	1219	1211	1340	1323
	Total Mn (mg kg <sup>-1</sup> )				Total Pb (mg kg <sup>-1</sup> )			
	Run 2	Run 3	Run 4	Run 5	Run 2	Run 3	Run 4	Run 5
0	129	191	169	159	13	20	22	40
4	183	198	228	172	22	16	34	18
11	182	207	182	148	30	18	29	44
21	190	232	218	190	23	27	43	38
32	227	236	230	206	23	15	41	51
46	249	267	274	221	27	30	40	46
56	272	297	269	233	52	46	70	67
	Total Cd (mg kg <sup>-1</sup> )							
	Run 2	Run 3	Run 4	Run 5	Run 2	Run 3	Run 4	Run 5
0	0.3	0.7	1.7	2.0				
4	1.2	1.0	2.2	1.3				
11	1.7	1.1	1.7	2.8				
21	0.7	2.1	3.3	2.5				
32	1.4	1.1	3.1	2.7				
46	1.2	2.2	3.2	3.1				
56	2.6	1.5	3.4	3.5				

Table 3.5. Correlation coefficients among physicochemical parameters of composts

	Time	MC	pH	VS	OM loss	T-N loss	T-C	T-N	C/N ratio	FAA-N	NH <sub>4</sub> -N	NO <sub>3</sub> -N	T-P	T-K	T-Ca	T-Mg	T-Cu	T-Zn	T-Mn	T-Pb
MC	-0.97***																			
pH	-0.53**	0.59***																		
VS	-0.94***	0.88***	0.35*																	
OM loss	0.89***	-0.83***		-0.97***																
T-N loss	0.68***	-0.61***		-0.81***	0.91***															
T-C	-0.92***	0.87***		0.95***	-0.96***	-0.85***														
T-N			-0.82***			-0.6**														
C/N ratio	-0.71***	0.71***	0.85***	0.61***	-0.47**		0.56**	-0.74***												
FAAN	-0.89***	0.89***	0.74***	0.81***	-0.73***	-0.45*	0.77***	-0.36*	0.82***											
NH <sub>4</sub> -N	-0.67***	0.69***	0.93***	0.50**			0.44*	-0.68***	0.86***	0.83***										
NO <sub>3</sub> -N	0.67***	-0.71***	-0.44*	-0.55**	0.48**		-0.51**		-0.53**	-0.55**	-0.48**									
T-P	0.65***	-0.63***		-0.71***	0.74***	0.68***	-0.76***		-0.38*	-0.47**										
T-K	0.69***	-0.66***	-0.44**	-0.64***	0.57**		-0.6***		-0.57**	-0.55***	-0.48**	0.42*	0.51**							
T-Ca	0.88***	-0.84***		-0.93***	0.91***	0.77***	-0.84***		-0.51**	-0.75***	-0.46*	0.54**	0.64***	0.54*						
T-Mg	0.85***	-0.86***		-0.86***	0.85***	0.74***	-0.89***		-0.52**	-0.71***	-0.41*	0.61***	0.72***	0.62***	0.77***					
T-Cu	0.79***	-0.78***		-0.80***	0.81***	0.65***	-0.79***		-0.59***	-0.7***	-0.41*	0.61***	0.67***	0.43*	0.83***	0.78***				
T-Zn	0.87***	-0.82***		-0.91***	0.85***	0.68***	-0.8***		-0.55**	-0.79***	-0.55**	0.51**	0.55**	0.54**	0.93***	0.66***	0.7***			
T-Mn	0.86***	-0.82***	-0.34*	-0.91***	0.85***	0.72***	-0.85***		-0.53**	-0.71***	-0.46**	0.53**	0.66***	0.66***	0.82***	0.86***	0.67***	0.80***		
T-Pb	0.69***	-0.66***		-0.63**	0.56**	0.39*	-0.51**		-0.47**	-0.67***	-0.53**	0.48*		0.5*	0.65***		0.41*	0.82***	0.52*	
T-Cd	0.57**	-0.54**		-0.60**	0.57**	0.43*	-0.46*		-0.34*	-0.57**	-0.42*				0.68***		0.45*	0.82***	0.48*	0.85***

Correlations were based on 28 average data of the 4 runs during composting; \*, \*\* and \*\*\* indicate correlation significant at  $P = 0.05$ ,  $0.01$  and  $0.001$  probability levels, respectively. Note that correlations where  $P > 0.05$  are not listed. MC = moisture content, VS = volatile solids, OM = organic matter

### 3.4. Conclusions

The composting of SS with woodchips as a bulking agent is feasible using a simple aeration method in small scale. The results show that compost temperatures were rapidly increased to the peak points of 47.4°C (run 1) and 74.8°C (run 2) within the first 2 to 3 days and varied depending on each composting run and vertical locations. The changes in physicochemical properties with particular attention to inorganic nitrogen ( $\text{NH}_4\text{-N}$ ,  $\text{NO}_3\text{-N}$ ) and FAAN indicated that the biodegradation took place by different mineralisation pathways during the composting process. The degradation of organic matter into amino acids followed by ammonification was predominant in run 2, whereas the nitrification was greater in run 1. The measurement of amino acid nitrogen may add the novelty for checking the stability of the process.

The similar trends of decrease in moisture content, volatile solid, total carbon, FAAN and  $\text{NH}_4\text{-N}$  in the four sludge: woodchips ratios (runs 2–5) suggested a possible biodegradation of organic matter. Heavy metals and “non-volatile” nutrients increased in concentration due to the loss in weight of the organic but their values were below the limit for agricultural use. Results of correlation analysis showed that most of the physicochemical parameters strongly correlated composting time and these parameters also correlated with each other. Only two of four tested volumetric ratios of sludge and woodchips (1:0.5 and 1:1) can meet the temperature requirement for acceptable composts ( $>55^\circ\text{C}$  for three consecutive days). Hence, the addition of woodchips into sludge at volumetric ratios of 1:1 or 1:0.5 was needed to promote the composting process.

The self-made composts had pH values of 5.7–6.9, C/N ratios of 6.5–7.3, TN of 44.1–50.6  $\text{g kg}^{-1}$ , FAAN of 0.3–0.6  $\text{g kg}^{-1}$  and  $\text{NH}_4\text{-N}/\text{NO}_3\text{-N}$  of 1.0–8.0, which were similar to the commercial composts in Japan. However, the finished compost had relatively low C/N ratios (6.5–7.3) predicting a fast mineralisation after soil application. This subject will be discussed in the next chapters.

## References

- Baca, M.T., I. Fernandez-Fígares, and M. De Nobili. 1994. Amino acid composition of composting cotton waste. *Sci. Total Environ.* 153(1–2): 51–56.
- Banegas, V., J.L. Moreno, J.I. Moreno, C. García, G. León, and T. Hernández. 2007. Composting anaerobic and aerobic sewage sludges using two proportions of sawdust. *Waste Manag.* 27(10): 1317–1327.
- Brewer, L.J., and D.M. Sullivan. 2003. Maturity and stability evaluation of composted yard trimmings. *Compost Sci. Util.* 11(2): 96–112.
- Brouillette, L., Trépanier, J., Gallichand, and C. Beuchamp. 1996. Composting paper mill deinking sludge with forced aeration. *Can. Agric. Eng.* 38(2): 115–122.
- Burge, W.D., W.N. Cramer, and E. Epstein. 1978. Destruction of Pathogens in Sewage Sludge by Composting. *Am. Soc. Agric. Biol. Eng.* 21(3): 0510–0514.
- Cabrera, M.L., D.E. Kissel, and M.F. Vigil. 2005. Nitrogen mineralization from organic residues: research opportunities. *J. Environ. Qual.* 34: 75–9.
- Carter, M.R., E.G. Gregorich, D. Pennock, T. Yates, and J. Braidek. 2007. *Soil Sampling and Methods of Analysis*, Second Edition. Canadian Society of Soil Science.
- Chanyasak, V., and H. Kubota. 1981. Carbon/organic nitrogen ratio in water extract as measure of composting degradation. *J. Ferment. Technol.* 59: 215–219.
- Cofie, O., D. Kone, S. Rothenberger, D. Moser, and C. Zubruegg. 2009. Co-composting of faecal sludge and organic solid waste for agriculture: Process dynamics. *Water Res.* 43(18): 4665–4675.
- Cofie, O., J. Nikiema, R. Impraim, N. Adamtey, and D. Paul, J.; Koné. 2016. Co-composting of solid waste and fecal sludge for nutrient and organic matter recovery. Colombo, Sri Lanka: International Water Management Institute (IWMI). CGIAR Research Program on Water, Land and Ecosystems (WLE). 47. (Resource Recovery and Reuse Serie.
- Diaz, L.F., M. De Bertoldi, W. Bidlinmaier, and E. Stentiford. 2007. *Compost Science and Technology*. Waste management series 8. Elsevier.
- Doane, T. a, and W.R. Horwath. 2003. Spectrophotometric determination of nitrate with a single reagent. *Anal. Lett.* 36(12): 2713–2722.
- Faithfull, N.T. 2002. *Methods in Agricultural Chemical Analysis: A Practical Handbook*. CABI publishing.
- Fang, M., J.W.C. Wong, K.K. Ma, and M.H. Wong. 1999. Co-composting of sewage sludge and coal fly ash: Nutrient transformations. *Bioresour. Technol.* 67(1): 19–24.
- El Fels, L., M. Zamama, A. El Asli, and M. Hafidi. 2014. Assessment of biotransformation of organic matter during co-composting of sewage sludge-lignocelulosic waste by chemical, FTIR analyses, and phytotoxicity tests. *Int. Biodeterior. Biodegrad.* 87: 128–137.
- Garcia, C., T. Hernandez, and F. Costa. 1991. Changes in carbon fractions during composting and

- maturation of organic wastes. *Environ. Manage.* 15(3): 433–439.
- Gigliotti, G., P. Proietti, D. Said-Pullicino, L. Nasini, D. Pezzolla, L. Rosati, and P.R. Porceddu. 2012. Co-composting of olive husks with high moisture contents: Organic matter dynamics and compost quality. *Int. Biodeterior. Biodegrad.* 67: 8–14.
- Gómez-Brandón, M., C. Lazcano, and J. Domínguez. 2008. The evaluation of stability and maturity during the composting of cattle manure. *Chemosphere* 70(3): 436–444.
- Haug, R.T. 1993. *The Practical Handbook of Compost Engineering*. Lewis Publishers, Boca Raton, FL.
- Hue, N.V., and J. Liu. 1995. Predicting Compost Stability. *Compost Sci. Util.* 3(2): 8–15.
- Iglesias Jiménez, E., and V. Perez Garcia. 1989. Evaluation of city refuse compost maturity: a review. *Biol. Wastes* 27(2): 115–142.
- Jones, D.L., and K. Kielland. 2002. Soil amino acid turnover dominates the nitrogen flux in permafrost-dominated taiga forest soils. *Soil Biol. Biochem.* 34(2): 209–219.
- Kumazawa, K. 1997. Use of sewage sludge for agriculture in Japan. *Proceedings of consultants meetings on sewage sludge and wastewater for use in agriculture*. Oct. 1997. 199 pp. 111-127. Joint FAO/IAEA Div. of Nuclear Techniques in Food and Agriculture, ViennaAustria, Intern.
- Kuter, G.A., H.A.J. Hoitink, and L.A. Rossman. 1985. Effects of aeration and temperature on composting of municipal sludge in a full-scale vessel system. *J. (Water Pollut. Control Fed.* 57(4): 309–315.
- Lähdesmäki, P., and R. Piispanen. 1989. Changes in concentrations of free amino acids during humification of spruce and aspen leaf litter. *Soil Biol. Biochem.* 21(7): 975–978.
- Larney, F.J., and A.F. Olson. 2006. Windrow temperatures and chemical properties during active and passive aeration composting of beef cattle feedlot manure. *Can. J. Soil Sci.*: 783–797.
- Li, Y., W. Li, B. Liu, K. Wang, C. Su, and C. Wu. 2013. Ammonia emissions and biodegradation of organic carbon during sewage sludge composting with different extra carbon sources. *Int. Biodeterior. Biodegrad.* 85: 624–630.
- Liang, C., K.C. Das, and R.W. McClendon. 2003. The influence of temperature and moisture contents regimes on the aerobic microbial activity of a biosolids composting blend. *Bioresour. Technol.* 86(2): 131–137.
- Lu, L.A., M. Kumar, J.C. Tsai, and J.G. Lin. 2008. High-rate composting of barley dregs with sewage sludge in a pilot scale bioreactor. *Bioresour. Technol.* 99(7): 2210–2217.
- Malińska, K., and M. Zabochnicka-Świątek. 2013. Selection of bulking agents for composting of sewage sludge. *Environ. Prot. Eng.* 39(2): 91–103.
- Malińska, K., M. Zabochnicka-Świątek, and J. Dach. 2014. Effects of biochar amendment on ammonia emission during composting of sewage sludge. *Ecol. Eng.* 71: 474–478.
- Margesin, R., J. Cimadom, and F. Schinner. 2006. Biological activity during composting of sewage sludge at low temperatures. *Int. Biodeterior. Biodegrad.* 57(2): 88–92.

- McGuckin, R.L., M.A. Eiteman, and K. Das. 1999. Pressure Drop through Raw Food Waste Compost containing Synthetic Bulking Agents. *J. Agric. Engng Res.*: 375–384.
- McKinley, V.L., and J.R. Vestal. 1985. Physical and chemical correlates of microbial activity and biomass in composting municipal sewage sludge. *Appl. Environ. Microbiol.* 50(6): 1395–1403.
- Moretti, S.M.L., E.I. Bertoncini, and C.H. Abreu-Junior. 2015. Composting sewage sludge with green waste from tree pruning. *Sci. Agric.* 72(5): 432–439
- Morisaki, N., Chae Gun Phae, K. Nakasaki, M. Shoda, and H. Kubota. 1989. Nitrogen transformation during thermophilic composting. *J. Ferment. Bioeng.* 67(1): 57–61.
- Onofri, A., and E. Pannacci. 2014. Spreadsheet tools for biometry classes in crop science programmes. *Commun. Biometry Crop Sci.* 9(2): 3–13.
- Paré, T., H. Dinel, M. Schnitzer, and S. Dumontet. 1998. Transformations of carbon and nitrogen during composting of animal manure and shredded paper. *Biol. Fertil. Soils* 26(3): 173–178.
- Paredes, C., A. Roig, M.P. Bernal, M.A. Sanchez-Monedero, and J. Cagarra. 2000. Evolution of organic matter and nitrogen during co-composting of olive mill wastewater with solid organic wastes. *Biol. Fertil. soils* 32: 222–227.
- Roig, A., A. García-Gómez, M.P. Bernal, and J. Cagarra. 2001. Composting of the solid fraction of olive mill wastewater. *Acta Hort.* 549: 19–27.
- Sah, R.N., and P.H. Brown. 1997. Boron Determination — A Review of Analytical Methods. *Microchem. J.* 304(56): 285–304.
- Said-Pullicino, D., F.G. Erriquens, and G. Gigliotti. 2007. Changes in the chemical characteristics of water-extractable organic matter during composting and their influence on compost stability and maturity. *Bioresour. Technol.* 98(9): 1822–1831.
- Tan, K.H. 1996. *Soil Sampling, Preparation, and Analysis*. Marcel Dekker, New York.
- Tan, K.H. 2000. *Environmental Soil Science*, Third Edition. CRC Press, UK.
- Tiquia, S.M., N.F.Y. Tam, and I.J. Hodgkiss. 1998. Composting of spent pig litter at different seasonal temperatures in subtropical climate. *Environ. Pollut.* 98(1): 97–104.
- Tiquia, S.M., and N.F.Y. Tam. 2000. Co-composting of spent pig litter and sludge with forced-aeration. *Bioresour. Technol.* 72(1): 1–7.
- Wang, K., W. Li, Y. Li, X. Gong, C. Wu, and N. Ren. 2013. The modelling of combined strategies to achieve thermophilic composting of sludge in cold region. *Int. Biodeterior. Biodegrad.* 85: 608–616.
- Witter, E., and J.M. Lopez-Real. 1987. The potential of sewage sludge and composting in a nitrogen recycling strategy for agriculture. *Biol. Agric. Hort.* 5(1): 1–23.
- Witter, E., and J. Lopez-Real. 1988. Nitrogen losses during the composting of sewage sludge, and the effectiveness of clay soil, zeolite, and compost in adsorbing the volatilized ammonia. *Biol. Wastes* 23(4): 279–294.



## Chapter 4: Soil nitrogen mineralisation–immobilisation

### 4.1. Introduction

N mineralisation from organic materials is one of the most important processes in the soil N dynamics by which organic N is mineralized to plant-available inorganic form. Compost can serve as a soil organic fertiliser to supply a source of nutrients for plant uptake. The management of compost-N plays an important role from both agricultural and environmental point of view. The available N derived from SS compost has been found to depend on gross N mineralisation–immobilisation (Binh et al., 2015). One of the most common problems associated with utilization of composted SS in advanced agricultural technologies is the risk of nitrate leaching into the groundwater through the application of fast-release N-fertilisers (Insam and Merschak, 1997; Łuczkiewicz and Quant, 2007).

Incorporating plant residues into the soil not only improves carbon sequestration but also prevents N loss by immobilizing inorganic form into organic one (Buford et al., 1998; Radersma and Smit, 2011). Materials with narrow C/N ratios can result in releasing of inorganic N via mineralisation, whereas materials with wide C/N ratios lead to temporary N immobilisation (Winsor and Pollard, 1956; Van Kessel et al., 2000). Several researchers have tested the immobilisation of soil N in the presence of added carbon and nitrogen sources (Kai et al., 1973; Paustian et al., 1974; Sakala et al., 2000). In general, to reach the highest N immobilisation, the added materials need to have a low level of lignin and low N content, but high C/N ratio with easily decomposable organic carbon.

However, the diversity in quality of carbon sources together with the large range in C/N ratios of amendments made it more difficult in predicting the N mineralisation. From this point of view, this study was carried out to provide more information concerning the MIT in the fertilized soil using the carbon sources from common plant residues.

The hypotheses are: 1) The incorporation of different carbon sources, i.e., powder of bamboo stem (BS), wood for building (WB) and rice straw (RS), into soil would change the MIT in a short-term period; 2) The same result would occur if BS was added to compost and/or urea with controlled C/N ratios.

### 4.2. Materials and methods

#### 4.2.1. Preparation for incubation 1, 2 and 3

The soil used for incubation 1 was a sandy soil, with a very low content of organic matter and a pH of 7.0 (data not shown). The soil used in both incubations 2 and 3 was collected from paddy fields at a depth of 0-30 cm in Minami district, Okayama Province, Japan (34°32'N, 133°53'E). The soil (237 g, d.b.) was air dried, sieved to pass through a 2-mm sieve. Soil (237 g, d.b.), and pre-incubated in Neubauer' pots (surface area: 100 cm<sup>2</sup>, height: 6.6 cm) for two days before starting incubations.

Commercial SS compost was obtained from Green Yuki composting plant in Okayama, Japan. Three plant residues containing carbon sources, i.e., bamboo stem (*Phyllostachys bambusoides*), residual wood for building (a mixture of Japanese cedar, *Cryptomeria japonica* and Red pine, *Pinus densiflora*, and *Abies firma*) and rice straw (*Oryza sativa* L.), were chopped, oven-dried at 105°C and ground to make fine powder for controlling the amendments' C/N ratios. The properties of the soil, compost and materials containing carbon are given in Table 4.1.

For incubation 1, one of the three different carbon sources, i.e. BS, WB and RS, was separately added to compost to increase the C/N ratio of compost up to 25. Modified compost was then applied to sandy soil at the application rate of 100 mg total N kg<sup>-1</sup> soil. For incubation 2, only BS was added to either compost or urea at different levels to increase the C/N ratios of amendments up to 10 and 25. For incubation 3, four combined treatments between compost and urea (100% compost, 90% compost + 10% urea, 70% compost + 30% urea, and 100% urea) at two application rates of 50 and 150 mg N kg<sup>-1</sup> soil were compared in the absence of BS in the same manner.

The amounts of additional carbon were exactly calculated by multiplying total nitrogen in compost by the target C/N ratios (10 or 25) and then subtracting those values from ready carbon contents in compost or urea. The nitrogen contents of plant residues were not accounted for this calculation. Consequently, the C/N ratios of amendments were adjusted up to 10 or 25. The treatment without any amendments (soil only) served as a control.

After mixing all materials and adjusting to 50% water holding capacity, the pots were covered with aluminum foil perforated with five small holes to ensure gas exchange and to reduce moisture losses. All the treatments were incubated at 25°C for twenty days. The soil samples in triplicates were destructively taken on days 10 and 20 for laboratory analysis. Therefore, each treatment consists of in six replications. There was a total of 3, 11 and 21 treatments for incubation 1, 2 and 3, respectively. The detail of treatment combinations and weight of ingredients in incubations 1-2 and 3 is given in Table 4.2 and Table 4.3, respectively.

#### 4.2.2. Soil analyses

Inorganic N was extracted from the fresh soil samples in 2.0 M KCl (w: v = 1:5). The filtrate was analyzed for NH<sub>4</sub>-N and NO<sub>3</sub>-N according to the phenol-hypochlorite method (Tan, 1996) and the Vanadium (III) chloride reduction method (Doane and Horwath, 2003), respectively, using the spectrophotometer (UV-1200, Shimadzu, Japan). All values are expressed on dry weight basis.

#### 4.2.3. Calculation and statistical analysis

Net N mineralisation, net ammonification and net nitrification rates were calculated for the incubation

period by subtracting the initial total inorganic nitrogen,  $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$ , respectively, from final respective N concentrations in the soil.

One-way, two-way and three-way ANOVA were used to determine statistically significant differences between treatments. The mean values of treatments were compared according to Fisher's protected least significant difference (LSD) test at  $P < 0.05$  using EXCEL<sup>®</sup> macro add-ins DSAASTAT version 1.512 (Onofri and Pannacci, 2014).

Table 4.1. Chemical properties of materials used for incubations 1, 2 and 3

Experimental materials	Total C (g kg <sup>-1</sup> )	Total N (g kg <sup>-1</sup> )	C/N	Inorganic N	
				NH <sub>4</sub> -N	NO <sub>3</sub> -N
				(g kg <sup>-1</sup> )	(g kg <sup>-1</sup> )
Soil					
Sandy	1.0	0.2	5.0	nd	nd
Paddy	23.9	2.1	11.7	7.3×10 <sup>-3</sup>	9.1×10 <sup>-3</sup>
Nitrogen sources					
Compost	293.0	42.3	6.4	1.6	8.1×10 <sup>-3</sup>
Urea	200.0	467.0	0.4	—	—
Added carbon sources					
BS	290.0	—	—	—	—
WB	330.0	—	—	—	—
RS	310.0	—	—	—	—

nd, not detected; BS = bamboo stem; WB = wood for building; RS = rice straw

Table 4.2. Treatment combinations and weight of ingredients in incubations 1 and 2

Treatments	Added C sources (mg C kg <sup>-1</sup> soil)			N sources (mg N kg <sup>-1</sup> soil)		C/N
	RS	WB	BS	Compost	Urea	
Incubation 1						
① (BS)	—	—	1944.5	100	0	25.0
② (WB)	—	1944.5	—	100	0	25.0
③ (RS)	1944.5	—	—	100	0	25.0
Incubation 2—set 1						
① (P-BS)	—	—	0.0	0	0	—
② (P+BS)	—	—	444.5	0	0	—
③ (P++BS)	—	—	957.2	0	0	—
④ (P+++BS)	—	—	1944.5	0	0	—
⑤ (P++++BS)	—	—	2457.2	0	0	—
Incubation 2—set 2						
⑥ (PC-BS)	—	—	0.0	100	0	6.4
⑦ (PC+BS)	—	—	444.5	100	0	10.0
⑧ (PC+++BS)	—	—	1944.5	100	0	25.0
⑨ (PU-BS)	—	—	0.0	0	100	0.4
⑩ (PU++BS)	—	—	957.2	0	100	10.0
⑪ (PU++++BS)	—	—	2457.2	0	100	25.0

BS = bamboo stem; WB = wood for building; RS = rice straw; P = paddy soil only; PC = paddy soil and compost; PU = paddy soil and urea

Table 4.3. Treatment combinations and weight of ingredients in incubation 3

Treatments	Added BS (mg C kg <sup>-1</sup> soil)	N sources (mg N kg <sup>-1</sup> soil)			C/N <sup>a</sup>	C/N <sup>b</sup>
		Compost <sup>b</sup>	Urea	Total <sup>a</sup>		
① S	0	0	0	0	-	-
② SC	0	50	0	50	5.6	5.6
③ SCU10	0	45	5	50	5.0	5.6
④ SCU30	0	35	15	50	4.0	5.7
⑤ SU	0	0	50	50	0.4	-
⑥ SCB	972	50	0	50	25	25
⑦ SCU10B	998	45	5	50	25	28
⑧ SCU30B	1049	35	15	50	25	36
⑨ SUB	1229	0	50	50	25	-
⑩ SCU10b	873	45	5	50	22.5	25
⑪ SCU30b	674	35	15	50	22.5	25
⑫ SC	0	150	0	150	5.6	5.6
⑬ SCU10	0	135	15	150	5.0	5.6
⑭ SCU30	0	105	45	150	4.0	5.7
⑮ SU	0	0	150	150	0.4	-
⑯ SCB	2917	150	0	150	25	25
⑰ SCU10B	2994	135	15	150	25	28
⑱ SCU30B	3147	105	45	150	25	36
⑲ SUB	3686	0	150	150	25	-
⑳ SCU10b	2619	135	15	150	22.5	25
㉑ SCU30b	2022	105	45	150	22.5	25

C/N<sup>a</sup> total carbon to total nitrogen; C/N<sup>b</sup> total carbon to total compost-N only; BS = bamboo stem; S, C, U and B (or b) represent factors of soil, compost, urea and bamboo stem addition, respectively; suffix numbers, i.e. 10, 30, indicate Urea-N percentage of total applied N

### 4.3. Results and discussion

#### 4.3.1. Effects of different carbon sources addition on soil N mineralisation–immobilisation

The sandy soil amended with compost had initial  $\text{NH}_4\text{-N}$  of  $3.81 \text{ mg N kg}^{-1}$  soil and  $\text{NO}_3\text{-N}$  of  $0 \text{ mg N kg}^{-1}$  (data not shown). The  $\text{NH}_4\text{-N}$  slightly decreased to 2.00, 3.27 and  $2.66 \text{ mg N kg}^{-1}$  soil after 20 days of incubation in the absence of BS, WB and RS, respectively. However,  $\text{NO}_3\text{-N}$  in treatment WB increased from 0 to  $6.66 \text{ mg N kg}^{-1}$  soil on day 10 and  $10.62 \text{ mg N kg}^{-1}$  soil on day 20. Consequently, the total inorganic N in the treatment WB was significantly higher than those in the treatments of BS and RS (Table 4.4). Similarly, compared to BS and RS, the treatment WB had significantly greater net nitrification and net N mineralisation rates. The results also show that the rates of net ammonification, net nitrification and net N mineralisation did not differ significantly between two treatments BS and RS (Table 4.5).

At a wide C/N of 25, the potential N immobilisation was greater in soil incorporated with BS or RS rather than that with WB, suggesting the difference in immediate bio-availability of added carbon sources. These results implied that either BS, RS may contain more labile carbon sources or WB may contain carbon compounds that are recalcitrant to decomposition, like lignin content (Rasse et al., 2006). Upadhyaya et al. (2004) conducted a 90-day laboratory incubation on soil amended with freshly fallen bamboo foliage from two different species in India. They found that amendments of foliage bamboo did not contribute to the increasing N mineralisation in the soil due to a readily available supply of C to soil microbes resulting in heterotrophic immobilisation.

#### 4.3.2. Effects of BS with controlled C/N ratios on soil N mineralisation- immobilisation

##### 4.3.2.1. Effect of BS levels on blank soil N mineralisation

The incubation 2–set 1

The  $\text{NO}_3\text{-N}$  in the paddy soil (P-B) increased rapidly during the first half and then maintained stability during the second half of the incubation time. Meanwhile, the change in  $\text{NH}_4\text{-N}$  was relatively small throughout the entire incubation period (Fig. 4.1). The results show that  $\text{NO}_3\text{-N}$  was more strongly affected by BS addition than  $\text{NH}_4\text{-N}$ . The  $\text{NO}_3\text{-N}$  concentration significantly decreased with increasing application rates of BS, suggesting a preferential immobilisation of nitrate rather than ammonium N.

Further evidence of immobilisation during the first 10 days of incubation in the presence of BS with application rates over  $957.2 \text{ mg C kg}^{-1}$  soil is shown in Fig. 4.2. The net N mineralisation rates and net nitrification rates during 20 days of incubation were positively correlated with the amount of BS ( $r^2 = 0.98$  and  $r^2 = 0.95$ , respectively). However, the net ammonification rate was not clearly affected by the amount of added BS.

Table 4.4. Soil inorganic N ( $\text{mg N kg}^{-1}$  soil) as affected by compost and carbon sources addition at controlled C/N = 25

Added C sources	10 days of incubation			20 days of incubation		
	$\text{NH}_4\text{-N}$	$\text{NO}_3\text{-N}$	Total	$\text{NH}_4\text{-N}$	$\text{NO}_3\text{-N}$	Total
BS	1.84	0.99	2.83 b	2.00	1.24 b	3.24 b
WB	3.29	6.66	9.95 a	3.27	10.62 a	13.89 a
RS	2.13	1.02	3.15 b	2.66	1.40 b	4.06 b
$P > F$	ns	ns	*	ns	**	**
LSD	1.75	5.93	5.11	1.27	4.68	4.55

Values within the same column followed by the same letter are not significantly different according to

LSD test at  $P < 0.05$

Table 4.5. Soil net Ammonification rate, net nitrification rate and net N mineralization rate as affected by compost and carbon sources addition at controlled C/N = 25

Added C sources	Net ammonification rate	Net nitrification rate	Net N mineralisation rate
	( $\text{mg N kg}^{-1}$ soil day $^{-1}$ )	( $\text{mg N kg}^{-1}$ soil day $^{-1}$ )	( $\text{mg N kg}^{-1}$ soil day $^{-1}$ )
BS	-0.09	0.06 b	-0.03 b
WB	-0.03	0.53 a	0.50 a
RS	-0.06	0.07 b	0.01 b
$P > F$	ns	**	**
LSD	6.32	0.23	0.23

Values within the same column followed by the same letter are not significantly different according to

LSD test at  $P < 0.05$

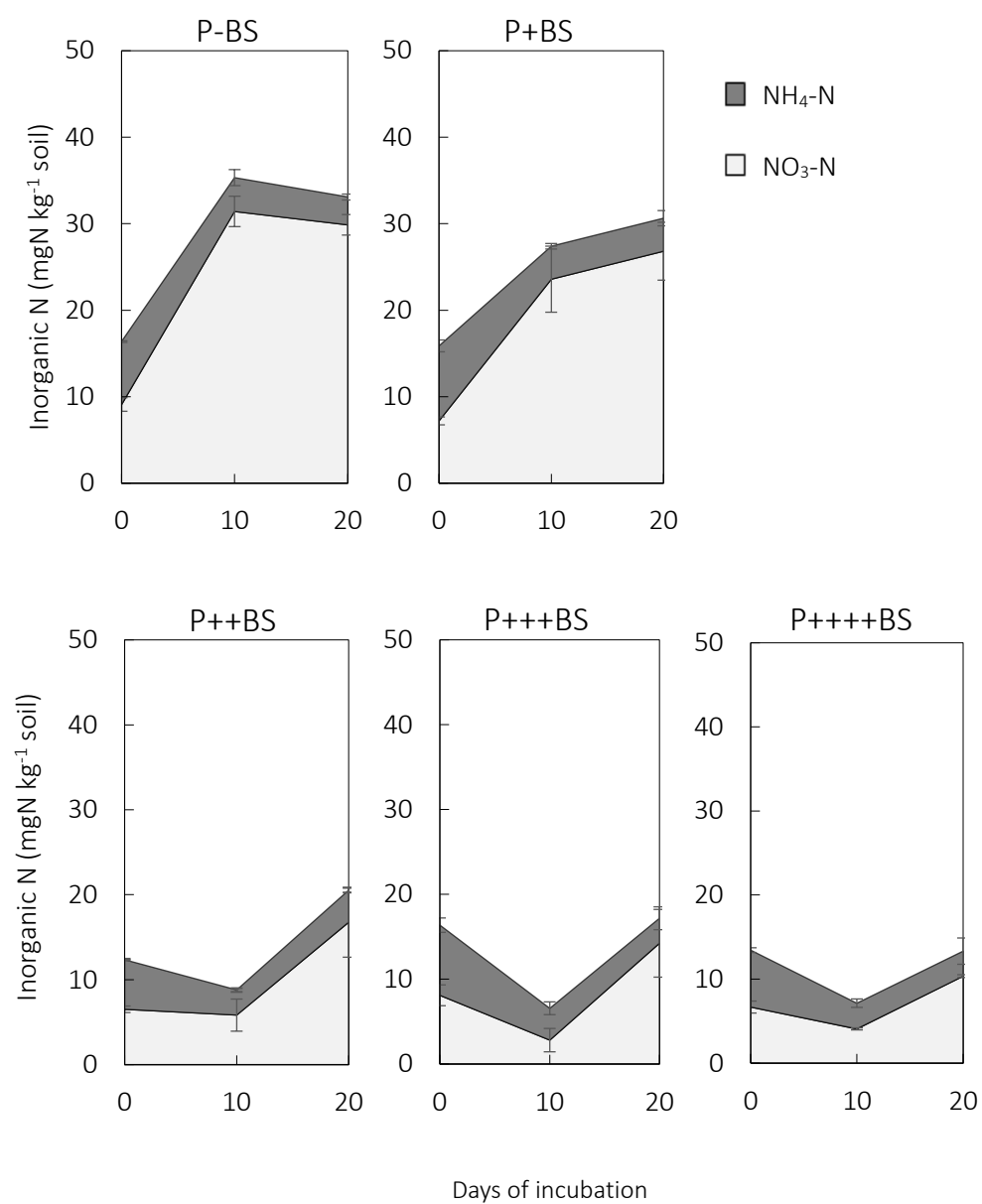


Fig. 4.1. Changes in inorganic nitrogen in paddy soil as affected by BS addition. Error bars represent standard deviation of the mean (n = 3)



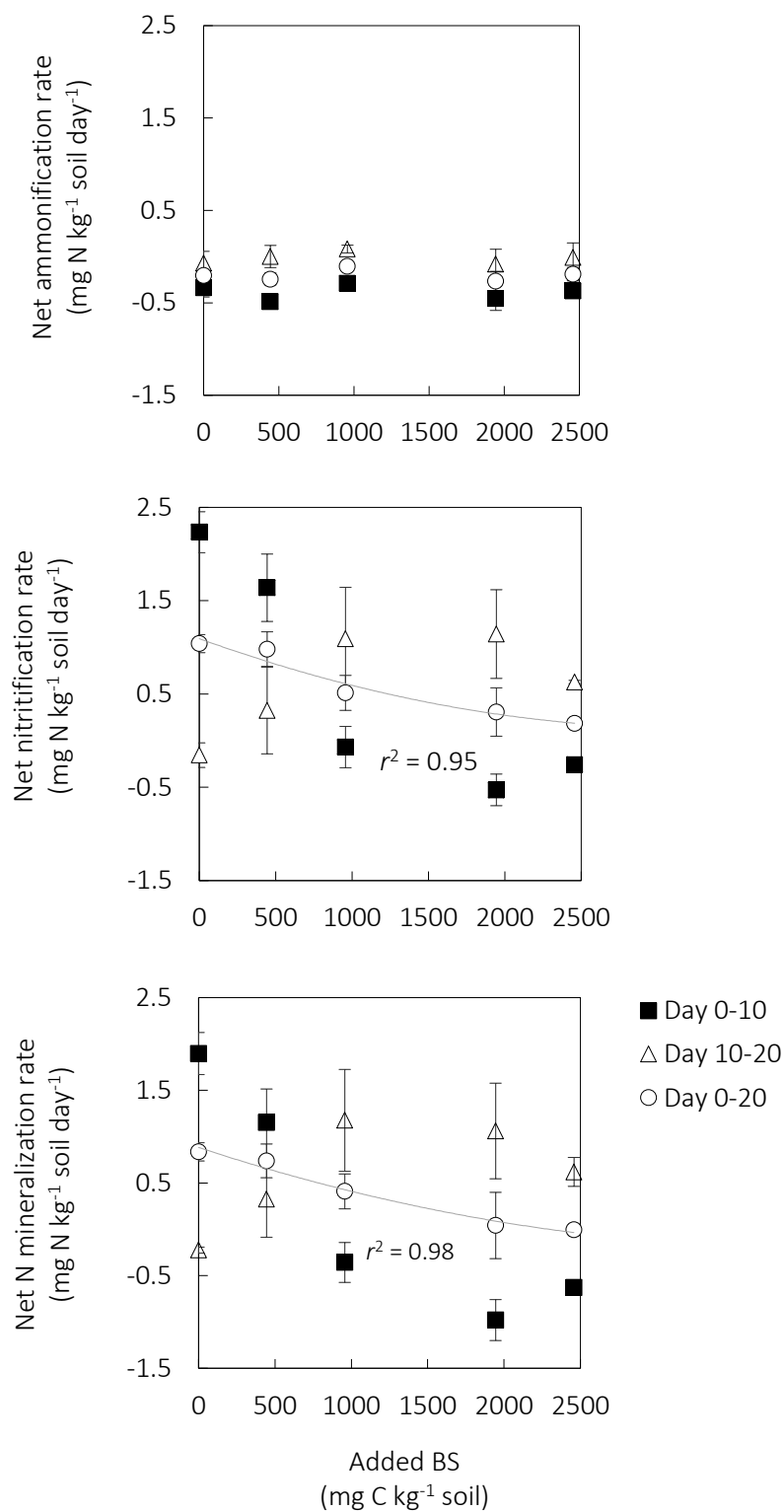


Fig. 4.2. The relationship between net ammonification, net nitrification, net N mineralisation rates and amount of BS addition. Error bars represent the standard deviation of the mean ( $n = 3$ ). R-squared values indicate the correlation during the whole incubation period of 0–20 day

#### 4.3.2.2. Effect of BS addition when either compost or urea was applied to soil

##### The Incubation 2–set 2

The changes in  $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$  in soil amended with N fertilisers and BS are shown in Fig. 4.3. In treatments amended with urea (PU), the  $\text{NO}_3\text{-N}$  increased continuously during the 20 days of incubation. However, in treatments amended with compost (PC), the  $\text{NO}_3\text{-N}$  rapidly increased during the first half and decreased during the second half of incubation. The  $\text{NH}_4\text{-N}$  gradually decreased with incubation time in PC treatments, but significantly increased during the first half and then decreased during the second half time of incubation in PU treatments. Thus, the changes in  $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$  in compost amended soil were similar to those observed in aforementioned paddy soil. The increase in  $\text{NO}_3\text{-N}$  was remarkably inhibited in the treatment PC+++BS (C/N=25) only. Meanwhile,  $\text{NH}_4\text{-N}$  was affected by BS additions in both treatments PU++BS (C/N=10) and PU++++BS (C/N=25).

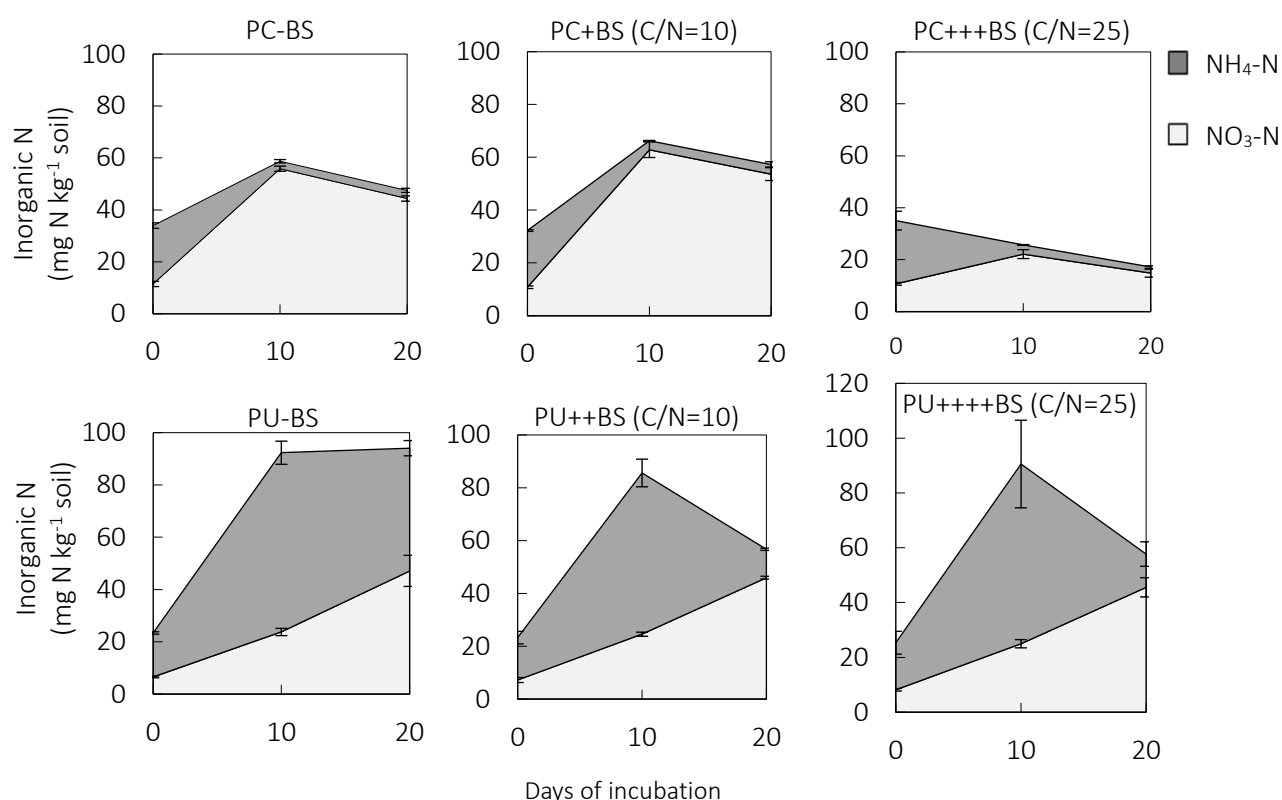


Fig. 4.3. Changes in inorganic nitrogen as affected by BS addition with controlled C/N= 10 and C/N=25 when either compost or urea was applied to soil at a rate of 100 mg kg<sup>-1</sup> soil. Bars represent standard deviation of the mean (n = 3); PC = paddy soil and compost; PU = paddy soil and urea; BS = bamboo stem

In regards to mineralisation rates, the net N mineralisation rates were significantly higher in urea than compost-amended treatments. The N immobilisation was found in the treatment PC+++BS (C/N=25) due to lowering net nitrification rate (0.20 mg N kg<sup>-1</sup> soil day<sup>-1</sup>) as compared to the control treatment PC-BS (1.64 mg N kg<sup>-1</sup> soil day<sup>-1</sup>). Meanwhile, net N mineralisation rates significantly decreased from 3.53 to

1.67 mg N kg<sup>-1</sup> soil day<sup>-1</sup> in the treatment PU++BS (C/N=10) and 1.62 mg N kg<sup>-1</sup> soil day<sup>-1</sup> in the treatment PU++++BS (C/N=25) (Table 4.6).

Table 4.6. Net ammonification, net nitrification and net N mineralisation rates of N-fertilized soil as affected by BS addition with controlled C/N=10 and C/N=25

Treatments	Net ammonification rate		Net nitrification rate		Net N mineralisation rate	
	(mg N kg <sup>-1</sup> soil day <sup>-1</sup> )		(mg N kg <sup>-1</sup> soil day <sup>-1</sup> )		(mg N kg <sup>-1</sup> soil day <sup>-1</sup> )	
	Compost	Urea	Compost	Urea	Compost	Urea
Control (no BS)	-0.97 c	1.51 a	1.64 b	2.03 a	0.68 d	3.53 a
C/N=10 (less BS)	-0.89 c	-0.27 b	2.14 a	1.94 ab	1.25 c	1.67 b
C/N=25 (more BS)	-1.10 c	-0.25 b	0.20 c	1.87 ab	-0.89 e	1.62 bc
Probability for:						
N sources	<0.001**		<0.001**		<0.001**	
BS levels	<0.001**		<0.001**		<0.001**	
N sources x BS levels	<0.001**		<0.001**		<0.001**	

Values within two columns for each parameter followed by the same letter are not significantly different according to LSD test at  $P < 0.05$

In the incubation 2–set 1, the nitrate nitrogen was rapidly immobilized in the presence of a BS. Thus, when the bio-available plant residues were supplied together with the available nitrogen source, the heterotrophic organisms in native soil could use carbon as a source of energy and nitrogen for increasing their population. These microbial assimilations induced a temporary reduction of inorganic nitrogen in the soil. Winsor and Pollard (1956) found a similar pattern of rapid N immobilisation within two days in soils treated with starch and sucrose. Their results indicated that the N immobilisation was greater in soil treated with higher levels of added carbon compounds.

The increase in immobilisation with increasing C/N ratios as observed in the incubation 2–set 2 was in agreement with other literature (Winsor and Pollard, 1956; Ahmad et al., 1972; Kai et al., 1973). At a given C/N ratio, the preferable assimilation for either ammonia or nitrate nitrogen might depend upon the nitrogen source and the quantity of added carbon. For example, the preference for nitrate was virtually complete at wider value of C/N ratio of 25 (more carbon added) in the case of soil amended with compost, whereas ammonia was easily assimilated at narrower value of C/N ratio of 10 (less carbon added) in the case of soil amended with urea. It should be noted that NO<sub>3</sub>-N content in current paddy soil was much higher than to NH<sub>4</sub>-N content after 10 and 20 days of incubation. However, when both inorganic nitrogen forms of NH<sub>4</sub>-N and NO<sub>3</sub>-N were present in the soil, such as treatments with urea, ammonia appeared to be more suitable for microbial immobilisation than nitrate nitrogen. A preference

for ammonia over nitrate was also reported by Recous et al. (1990), who indicated that in soil added with glucose at a rate of 500 mg C kg<sup>-1</sup> soil, the amount of <sup>15</sup>N immobilized was much higher in <sup>15</sup>N-Urea, (<sup>15</sup>NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and <sup>15</sup>NH<sub>4</sub>NO<sub>3</sub> than in NH<sub>4</sub><sup>15</sup>NO<sub>3</sub> treatments. One possible reason was that the amount of carbon needed to immobilize one unit of nitrogen was larger for nitrate than for ammonia as suggested by Ahmad et al. (1972).

#### 4.3.3. Effect of BS addition on soil N mineralisation when compost was combined with urea

##### The incubation 3

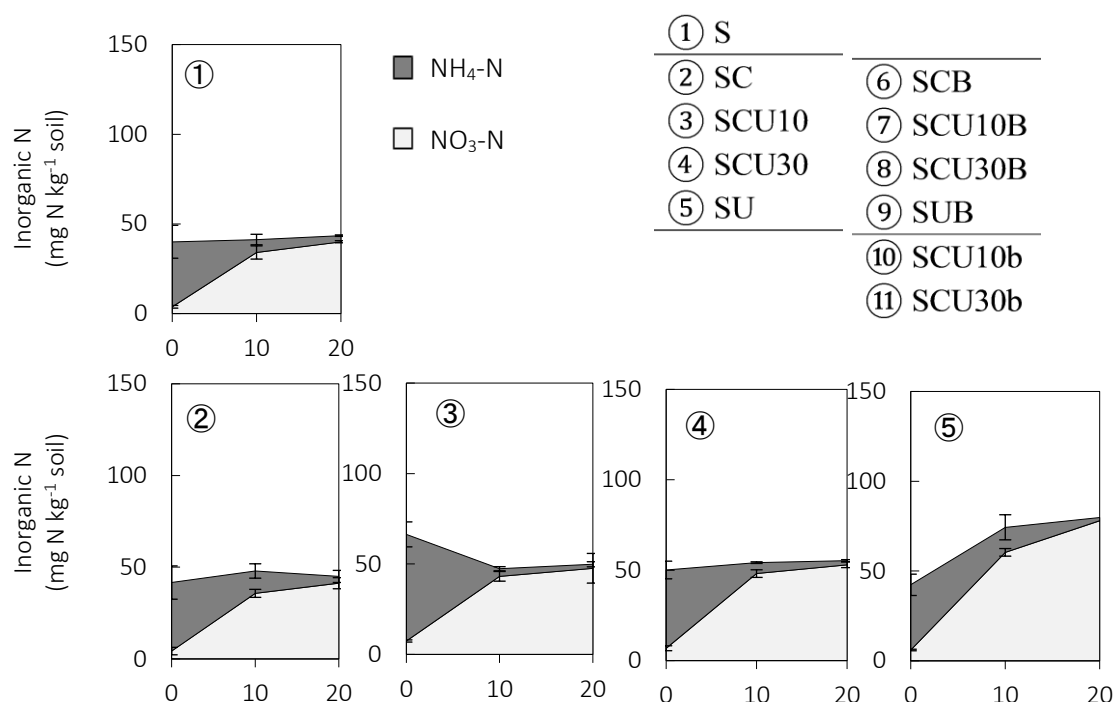
The changes in inorganic N (NH<sub>4</sub>-N and NO<sub>3</sub>-N) as affected by BS addition with controlled C/N=25 when compost was combined with urea and applied to soil at two rates are shown in Fig. 4.4 and Fig. 4.5. The results showed that NO<sub>3</sub>-N concentrations rapidly increased during the first 10 days and fluctuate depending on the presence or absence of BS addition. Meanwhile, NH<sub>4</sub>-N concentration of almost treatments significantly decreased with incubation time.

At low N application rate of 50 mg kg<sup>-1</sup> soil, the concentrations and changes in NH<sub>4</sub>-N and NO<sub>3</sub>-N in the treatment (2) and (4) were similar to those in the soil-only (Fig. 4.4). The inorganic N concentrations were improved at the higher application rate of 150 mg N kg<sup>-1</sup> soil and these values increased with increasing proportion of CF (Fig. 4.5).

However, the combination of compost and CF at two recipes have not shown the improvement compared to compost alone. In the same N application rate, the total inorganic N was highest in the treatments using 100% CF (treatments (5) and (15)).

The incorporation of BS into compost and/or CF strongly decreased the inorganic N concentrations (almost in NO<sub>3</sub>-N form) at all treatments. The difference between two types of controlled C/N ratios (first, total carbon to total nitrogen and second, total carbon to total nitrogen from compost) was not clear. It was likely that NH<sub>4</sub>-N was consumed greater at the beginning of incubation time and immobilisation of NO<sub>3</sub>-N was reduced under the second-type C/N ratio (treatments (20) and (21)) where BS was added with a little bit lower application rates (Table 4.3). However, this rule seems to be not right in case of lower N application rate of 50 mg N kg<sup>-1</sup> soil.

Without BS addition



With BS addition

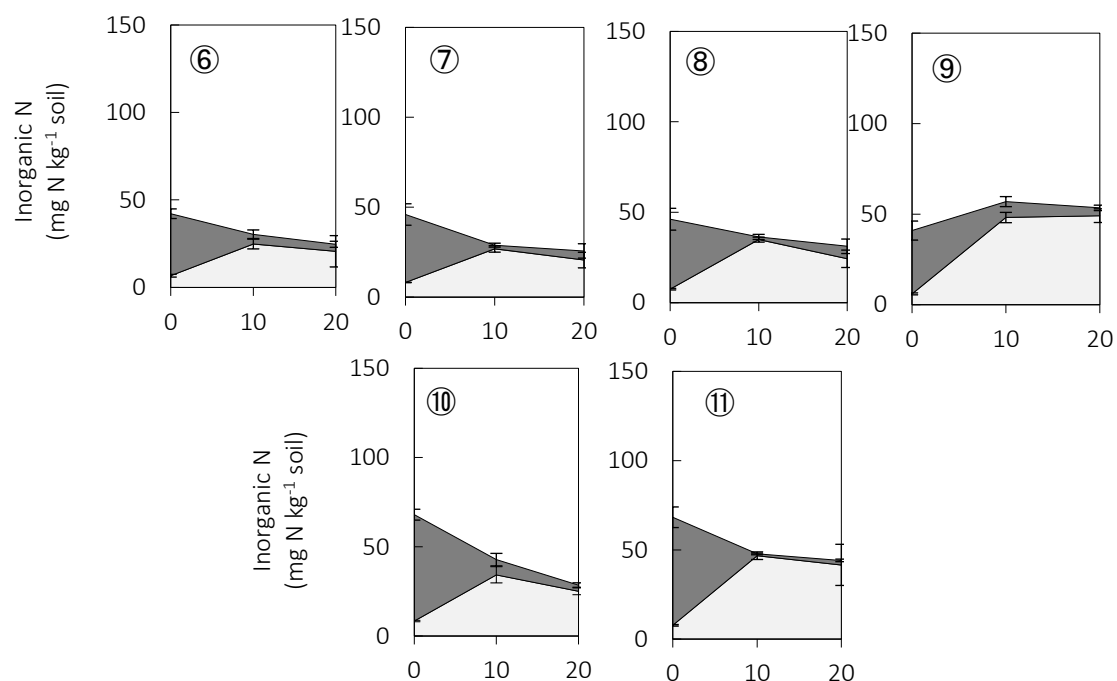


Fig. 4.4. Changes in inorganic N as affected by BS addition with controlled C/N=25 when compost was combined with urea and applied to soil at rate of 50 mg kg<sup>-1</sup> soil. Bars represent standard deviation of the mean (n = 3); S, C, U and B (or b) represent factors of soil, compost, urea and BS addition, respectively; suffix numbers, i.e. 10, 30, indicate Urea-N percentage of total applied N

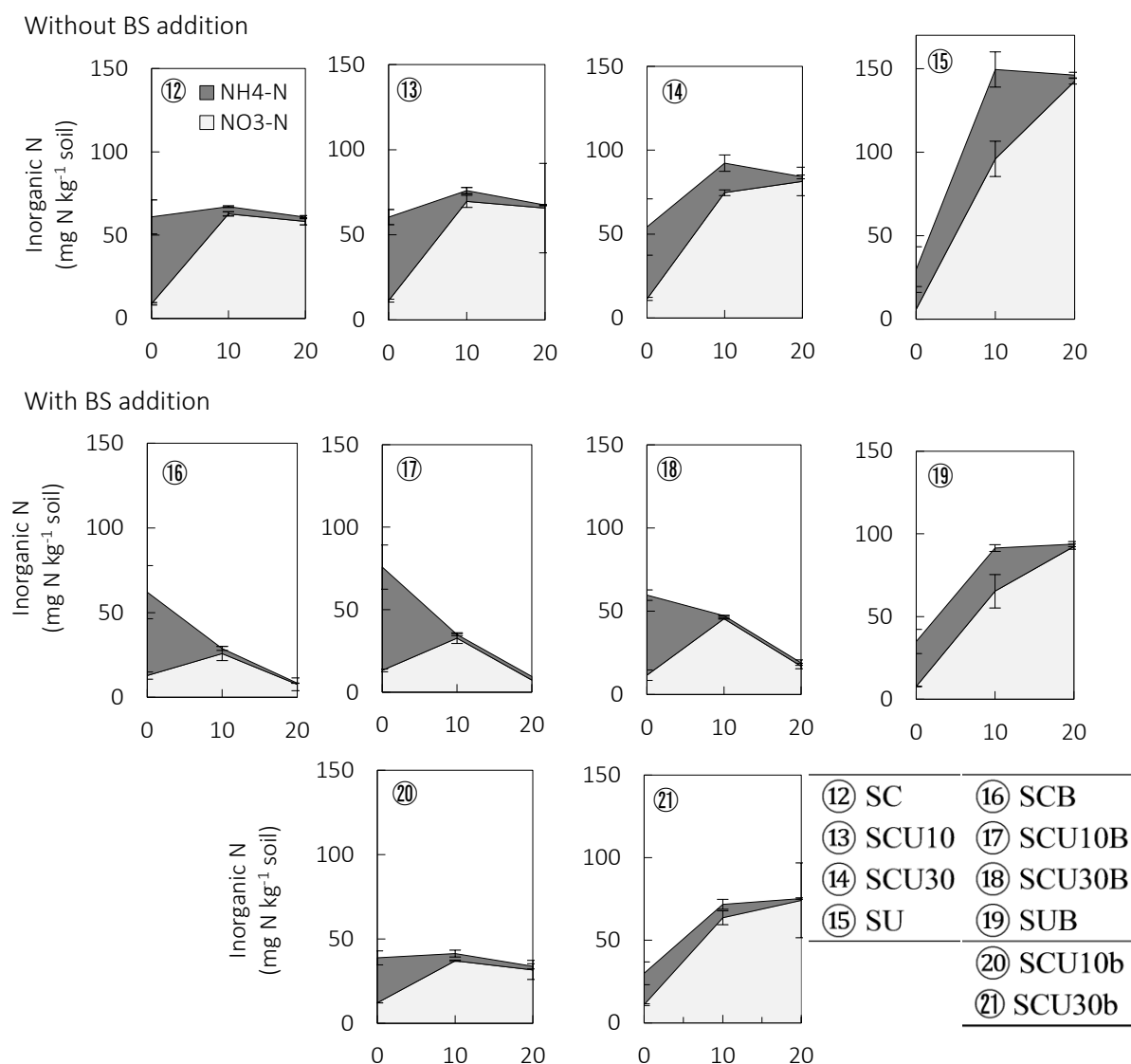


Fig. 4.5. Changes in inorganic N as affected by BS addition with controlled C/N=25 when compost was combined with urea and applied to soil at rate of  $150 \text{ mg kg}^{-1} \text{ soil}$ . Bars represent standard deviation of the mean ( $n = 3$ ); S, C, U and B (or b) represent factors of soil, compost, urea and BS addition, respectively; suffix numbers, i.e. 10, 30, indicate Urea-N percentage of total applied N

Net ammonification, net nitrification and net N mineralisation rates of N-fertilized soil as affected by BS addition with controlled C/N=25 are given in Table 4.7. The results showed that the net N ammonification rates were affected by nitrogen application rates and combination recipes rather than BS addition. Meanwhile, net nitrification rates were affected by all factors. As a result, net N mineralisation rates were affected by BS addition and combinations. The higher N application rate at  $150 \text{ mg kg}^{-1} \text{ soil}$  caused a higher net N mineralisation but it was not significantly different from that of the lower N application rate at  $50 \text{ mg kg}^{-1} \text{ soil}$ . The net nitrification rates and net N mineralisation rates gradually increased with increasing proportion of urea from 10 to 100%, while net ammonification rates were highest when compost was applied singly (Table 4.7).

Table 4.7. Net ammonification, net nitrification and net N mineralisation rates of N-fertilized soil as affected by BS addition with controlled C/N=25

Treatments	Net ammonification rate	Net nitrification rate	Net N mineralisation rate
Nitrogen rates (N)			
50 mg kg <sup>-1</sup> soil	-0.18 a	-0.05 b	-0.24
150 mg kg <sup>-1</sup> soil	-0.47 b	0.62 a	0.15
BS addition (B)			
No	-0.38	1.35 a	0.97 a
Yes	-0.28	-0.78 b	-1.06 b
Combinations (C)			
100% compost	-0.40 a	-0.62 c	-1.02 bc
90% compost + 10% urea	-0.82 b	-0.55 bc	-1.37 c
70% compost + 30% urea	-0.34 b	-0.08 b	-0.43 b
100% urea	-0.26 b	2.39 a	2.65 a
Probability for:			
N	0.044*	<0.001***	0.057 <sup>ns</sup>
B	0.479 <sup>ns</sup>	<0.001***	<0.001***
C	<0.001***	<0.001**	<0.001***
N x B	<0.007**	<0.001***	<0.001***
N x C	0.014*	<0.001***	<0.001***
B x C	0.872 <sup>ns</sup>	0.673 <sup>ns</sup>	0.892 <sup>ns</sup>
N x B x C	0.133 <sup>ns</sup>	0.859 <sup>ns</sup>	0.312 <sup>ns</sup>

Values within the same column followed by the same letter are not significantly different according to LSD test at  $P < 0.05$ . All rate values are expressed in the unit of mg N kg<sup>-1</sup> soil day<sup>-1</sup>. \*, \*\* and \*\*\* indicate significance at  $P = 0.05$ , 0.01 and 0.001, respectively

The incorporation of BS into all treatments resulted in a significant decrease in net N mineralisation rate as a consequence of the immobilisation of nitrate N. The nitrogen application rate was found to interact with the BS addition and combinations but no interaction was found between two factors of BS addition and combination recipes. In addition, there was no interaction between three experimental factors. The effect of two types of controlled C/N ratios on net N mineralisation rates was not shown in Table 4.7 due to the unbalanced design. However, their values were used for correlation analysis which will be mentioned in the section 5.3.1.2 of the next chapter.

#### 4.4. Conclusions

The incubation studies showed the following:

For incubation 1: The quality of added carbon is likely the more important factor to control the soil N mineralisation–immobilisation turnover in soil. The incorporation of bio-available carbon sources like bamboo stem powder and rice straw powder into N-fertilized soil caused a temporary decrease in net N mineralisation or reveal a microbial immobilisation.

For incubation 2: When compost or urea was applied to soil at a rate of 100 mg N kg soil<sup>-1</sup> and bamboo stem powder was added, the immobilisation of either nitrate or ammonium occurred depending on the supplemented nitrogen source. The immobilisation of nitrate was found at all treatments receiving BS, meanwhile, the immobilisation of ammonium was found in treatments receiving urea only.

For incubation 3: More urea additions to compost resulted in an increase in N mineralisation rate, while the addition of BS in N-fertilized soil caused a decrease in N mineralisation rate due to immobilisation of nitrate.

Clearly, the availability of carbon sources will control the mineralisation or immobilisation of N. Total C/N ratio can give a reliable indicator of N availability from compost, but further study is needed to clarify the effects of carbon and nitrogen sources on plant–soil system after their application. This subject will be discussed in the next chapters.



## References

- Ahmad, Z., H. Kai, and T. Harada. 1972. Effect of Nitrogenous Forms on Immobilization and Release of Nitrogen in Soil. 65: 49–65.
- Binh, N.T., H.T. Quynh, and K. Shima. 2015. Effect of Composts Combined with Chemical N Fertilizer on Nitrogen Uptake by Italian Ryegrass and N Transformation in Soil. *J. Agric. Chem. Environ.* 4(2): 37–47.
- Buford, M.A., B.J. Stokes, F.G. Sanchez, and E.A. Carter. 1998. Using biomass to improve site quality and carbon sequestration. *Proceedings of IEA Bioenergy Task 18 workshop “Developing systems for integrating bioenergy into environmentally sustainable”*. : 97–103.
- Doane, T. a, and W.R. Horwath. 2003. Spectrophotometric determination of nitrate with a single reagent. *Anal. Lett.* 36(12): 2713–2722.
- Insam, H., and P. Merschak. 1997. Nitrogen leaching from forest soil cores after amending organic recycling products and fertilizers. *Waste Manag. Res.* 15(3): 277–291.
- Kai, H., Z. Ahmad, and T. Harada. 1973. Factors affecting immobilization and release of nitrogen in soil and chemical characteristics of the nitrogen newly immobilized III. Transformation of the nitrogen immobilized in soil and its chemical characteristics. *Soil Sci. Plant Nutr.* 19(4): 275–286.
- Van Kessel, J.S., J.B. Reeves, and J.J. Meisinger. 2000. Nitrogen and carbon mineralization of potential manure components. *J. Environ. Qual.* 29(5): 1669–1677.
- Łuczkiwicz, a, and B. Quant. 2007. Soil and Groundwater Fecal Contamination as a Result of Sewage Sludge Land Application. *Polish J. Environ. Stud.* 16(4): 587–593.
- Onofri, A., and E. Pannacci. 2014. Spreadsheet tools for biometry classes in crop science programmes. *Commun. Biometry Crop Sci.* 9(2): 3–13.
- Paustian, K., W.J. Parton, and J. Persson. 1974. (1992) Modeling Soil Organic Matter in Organic-Amended and Nitrogen-Fertilized Long-Term Plots. *Structure (1950)*: 476–488.
- Radersma, S., and A.L. Smit. 2011. Assessing denitrification and N leaching in a field with organic amendments. *NJAS- Wageningen J. Life Sci.* 58(1–2): 21–29.
- Rasse, D.P., M.F. Dignac, H. Bahri, C. Rumpel, A. Mariotti, and C. Chenu. 2006. Lignin turnover in an agricultural field: From plant residues to soil-protected fractions. *Eur. J. Soil Sci.* 57(4): 530–538.
- Recous, S., B. Mary, and G. Faurie. 1990. Microbial immobilization of ammonium and nitrate in cultivated soils. *Soil Biol. Biochem.* 22(7): 913–922.
- Sakala, W.D., G. Cadisch, and K.E. Giller. 2000. Interactions between residues of maize and pigeonpea and mineral N fertilizers during decomposition and N mineralization. *Soil Biol. Biochem.* 32(5): 679–688.
- Tan, K.H. 1996. *Soil Sampling, Preparation, and Analysis*. Marcel Dekker, New York.

- Upadhyaya, K., A. Arunachalam, and K. Arunachalam. 2004. Effect of bamboo foliage on soil respiration, microbial biomass and N mineralization. *J. Bamboo Ratt.* 3(2): 169–183.
- Winsor, G.W., and A.G. Pollard. 1956. Carbon-nitrogen relationships in soil. I.-The immobilization of nitrogen in the presence of carbon compounds. *J. Sci. Food Agric.* 7(2): 134–141.

## Chapter 5: Effectiveness of compost as soil fertilizer

### 5.1. Introduction

Composted sludge can be used as a soil amendment to supply nutrients to plants, improve soil physical properties, and increase the percentage of organic matter (Warman and Termeer 2005; Pedra et al. 2007), although negative effects associated with heavy metal accumulation and phytotoxicity phenomena in plant-soil systems should be taken into consideration (Tan 2000; Alvarenga et al. 2015).

Several studies on compost quality have focused on the changes in compost physicochemical properties (Brewer & Sullivan 2003; El Fels et al., 2014) followed by a seed germination test or plant growth bioassays (Said-Pullicino et al., 2007; Eklind et al., 2016) as indicators of compost stability and maturity, but few have attempted to relate compost properties to plant growth and N uptake (Cooperband et al., 2003). Thus, the information on the effects of active fractions of organic nitrogen like amino acids on plant uptake is very scarce. Free amino acids are thought to be an easily decomposable organic N and become an important N source for plants (Jones and Kielland, 2002), but also the factor limiting N availability for plant uptake due to N source for heterotrophic microorganisms in the soil system.

The preliminary incubation studies, as discussed chapter 4, provided information on N mineralisation from compost and chemical N fertiliser. The results indicated that the incorporation of BS or RS caused a temporary immobilisation of nitrate nitrogen. However, it is not clear that whether the nitrogen was ready for nitrogen uptake. Therefore, the planting experiments are necessary to correlate the results from incubation studies with plant response.

In this chapter, two pot-culture experiments were performed under greenhouse condition. In the first one, the same combined treatments of compost and urea, of which C/N ratios were adjusted by BS addition as described in the incubation 3, were applied to Komatsuna to test the results obtained from incubation studies. In the second one, the effectiveness of the two self-made composts from runs 1 and 2 to Komatsuna were compared. The results were compared to their raw materials and chemical N fertiliser as control treatments. Plant growth, dry matter production, nutrient uptake and heavy metals accumulation were measured and discussed. In this chapter, some properties of composts were related to plant yield and nitrogen uptake as a means to evaluate the compost quality.

### 5.2. Materials and methods

#### 5.2.1. Preparation for pot-culture experiment 1 and 2

##### 5.2.1.1. Pot-culture experiment 1

In Oct 2015, a preliminary pot-culture experiment was carried out using twenty-one treatments as

described in the incubation 3 (Table 4.3). In this experiment, the effects of combined application of compost and urea (SC, 100% compost; SCU10, 90% compost + 10% urea; SCU30, 70% compost + 30% urea; and SU, 100% urea) with and without BS addition were studied. A mixture of 474 g soil (d.b.) of paddy soil and sandy soil (1:1, w/w) was prepared in Neubauer' pots (surface area: 100 cm<sup>2</sup>, height: 6.6 cm). Soil was thoroughly mixed with amendments at two application rates of 50 and 150 mg N kg<sup>-1</sup> soil. Komatsuna was sown at a density of 25 seeds pot<sup>-1</sup> and then thinned to 20 plants pot<sup>-1</sup>. The pots were watered daily and the plants were harvested after 30 days of cultivation.

#### 5.2.1.2. Pot-culture experiment 2

Two finished composts (called compost 1 and 2 which were obtained from run 1 and run 2, respectively) and their raw materials (sludge 1 and sludge 2) were employed in the greenhouse experiment at Okayama University. Properties materials used in this experiment are given in Table 3.1 and Table 5.1. Sandy soil (3.02 kg, d.b.) was passed through a 2-mm sieve and put into a Wagner pot (surface area: 200 cm<sup>2</sup>, height: 20 cm) for all treatments. There were total nine treatments in this experiment. The detail of treatments and weight of ingredients is given in Table 5.2. Basically, either sludges or composts was applied at a rate of 600 mg N pot<sup>-1</sup> at the top 0–5 cm for each pot, which is equivalent to 300 kg N ha<sup>-1</sup>. After calculating the total P and K contents contributed by sludge or compost, all treatments except the control (soil only) were supplemented with P as super phosphate and K as potassium chloride to get the same rates of 450 mg P pot<sup>-1</sup> and 300 mg K pot<sup>-1</sup> at the top 0–5 cm, respectively.

Komatsuna (*Brassica rapa* var. *perviridis*) was sown at a density of 15 seeds pot<sup>-1</sup>. After emergence, seedlings were thinned to a density of 12 plants pot<sup>-1</sup>. The growing season lasted for 41 days of cultivation. All plant parts (leaf and root) were separately harvested and dried at 105°C for 26 hours. Plant dry matter production was expressed per unit of the dry weight (g pot<sup>-1</sup>). All treatments were arranged in a randomized complete block design with three replications.

#### 5.2.2. Soil–plant analysis and calculation

The P concentration in plants were determined using molybdenum blue method; and heavy metals (Zn, Cu, Mn, Pd, Cd) accumulation in plants were measured by atomic absorption spectrometry (AA-6800, Shimadzu, Japan) after dry ashing samples and dissolving in 10 ml 50% v/v HCl (Faithfull, 2002).

The N concentration in plants (leaf and root) was measured by C-N analyzer (CORDER MT-700; Yanaco, Japan). Plant N uptake was calculated by multiplying the dry matter with respective N concentration of each plant part (leaf and root). Total plant N uptake in this pot-culture experiment is given in unit of mg N plant<sup>-1</sup>.

### 5.2.3. Statistical tests

The data were analyzed according to Fisher's protected least significant difference (LSD) test at  $P < 0.05$  after a one-way ANOVA using EXCEL<sup>®</sup> macro add-ins DAAASTAT version 1.512 (Onofri and Pannacci, 2014).

Table 5.1. Physicochemical properties of composts and soil in the pot-culture experiment 2

Parameters	Compost 1	Compost 2	Sandy soil <sup>†</sup>
Moisture content (% d.b.)	118.8	74.0	8.8
pH (1:5, w/v)	5.7	6.8	7.3
Volatile solids (%)	69.2	69.2	–
Total C (g kg <sup>-1</sup> )	329.3	331.7	nd
TN (g kg <sup>-1</sup> )	50.5	46.2	0.2
C/N ratio	6.5	7.2	–
Amino acid-N (g kg <sup>-1</sup> )	0.7	0.6	–
NH <sub>4</sub> -N (g kg <sup>-1</sup> )	8.7	5.8	< 0.01
NO <sub>3</sub> -N (g kg <sup>-1</sup> )	9.1	0.7	< 0.01
Total P (g kg <sup>-1</sup> )	18.6	20.0	0.1
Troug-P (g kg <sup>-1</sup> )	1.0	1.8	–
Total cations (g kg <sup>-1</sup> )			
K	7.9	6.4	2.3
Ca	1.9	13.7	1.3
Mg	11.8	4.9	1.1
Total heavy metal (mg kg <sup>-1</sup> )			
Zn	819	1219	–
Cu	256	341	–
Mn	200	272	–
Pb	7.5	52	–
Cd	nd	2.6	–

Values in parentheses are standard deviation (n = 3); nd, not detected.

<sup>†</sup>Sandy soil (sand 86.0%; silt 4.7%; clay 9.3%)

Table 5.2. Treatment combinations and weight of ingredients in the pot-culture experiment 2

Treatments	N sources (mg N pot <sup>-1</sup> )			
	Sludge	Compost	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	Total
① Soil-only (SO)	–	–	0	0
② N0 <sup>†</sup>	–	–	0	0
③ N150	–	–	150	150
④ N300	–	–	300	300
⑤ N600	–	–	600	600
⑥ Sludge 1 (S1)	600	–	–	600
⑦ Compost 1 (C1)	–	600	–	600
⑧ Sludge 2 (S2)	600	–	–	600
⑨ Compost 2 (C2)	–	600	–	600

### 5.3. Results and discussion

#### 5.3.1. Pot-culture experiment 1

##### 5.3.1.1. Effects of combined compost and urea with BS addition on total N uptake

Total N uptake at two N application rates was strongly affected by nitrogen sources, the combination recipes and BS addition (Fig. 5.1). Total N uptake by plant in the treatments ② and ④ did not significantly ( $P > 0.05$ ) differ from that of the treatment 1, indicating the lower N supplying potential of compost than chemical N fertiliser (treatment 5) at low application rate. The N uptake in presence of compost was improved at higher application rate of 150 mg N kg<sup>-1</sup> soil (treatments 12, 13 and 14) compared to the lower rate and control treatment. However, the combinations between compost and CF at two recipes have not shown the improvement compared to compost alone. In the same N application rate, the total N uptake was highest in the treatments using 100% CF (treatments 5 and 15).

The incorporation of BS into compost and/or CF significantly decreased total N uptake at all treatments, indicating the microbial immobilisation as discussed in section 4.3.3. Noticeably, the N uptake reduction was likely not different between two types of controlled C/N ratios (total carbon to total nitrogen vs total carbon to total nitrogen from compost). This implies that the nitrogen source from compost may more effective than that from CF for N immobilisation by microbes in the absence of bioavailable carbon source. Or on the other hand, the N mineralisation can be controlled by adjusting the C/N ratio of compost rather than C/N ratio of the total amendments (compost and chemical N fertiliser).

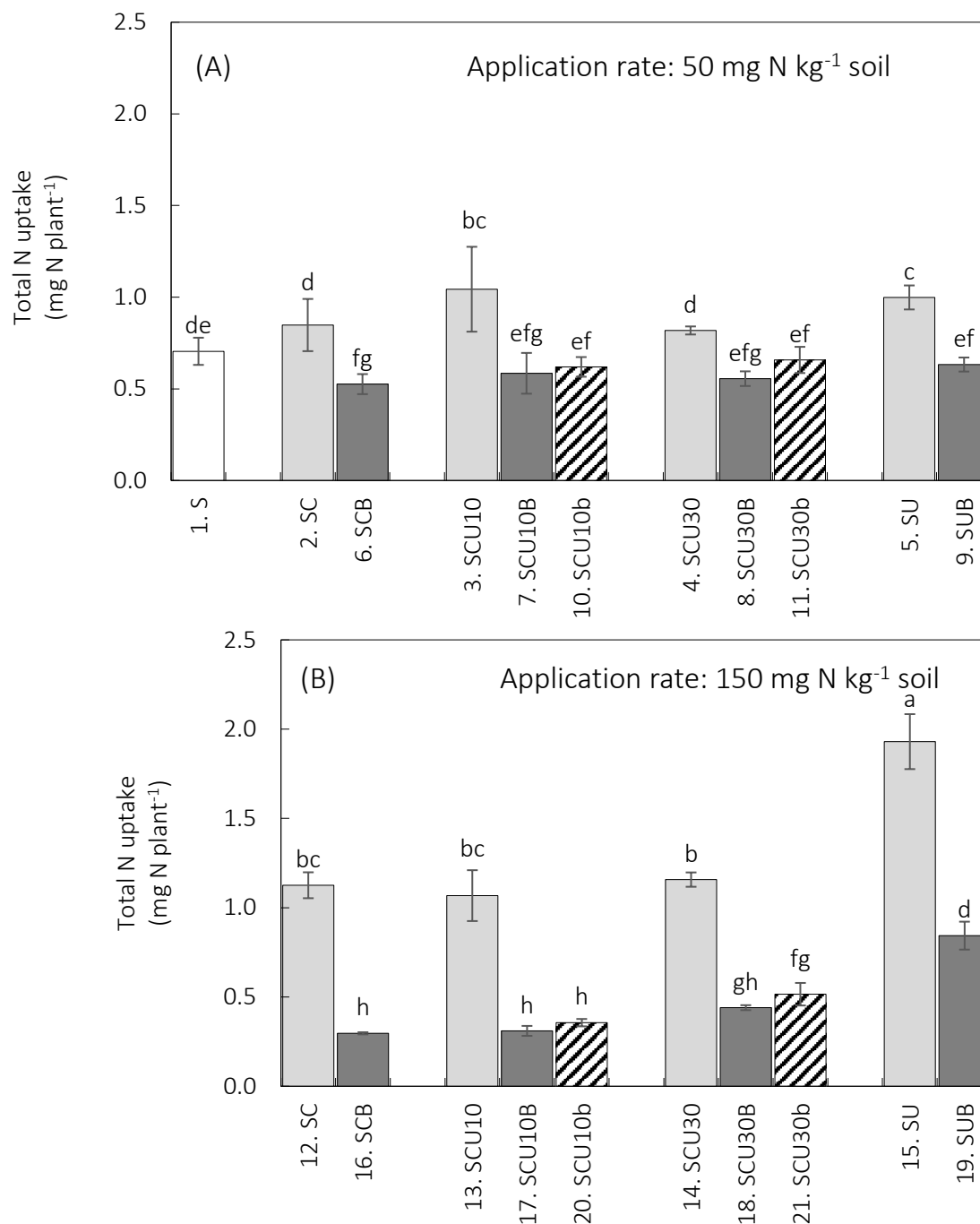


Fig. 5.1. Total plant N uptake when compost and urea were applied to soil as affected by BS addition with controlled C/N=25. Different letters indicate significant differences among treatments ( $P < 0.05$ );

S, C, U and B (or b) represent factors of soil, compost, urea and BS addition, respectively; suffix numbers, i.e. 10, 30, indicate Urea-N percentage of total applied N

#### 5.3.1.2. Relationship between soil net N mineralisation and total plant N uptake

Fig. 5.2 shows the relationship between soil net ammonification rate, net nitrification rate, net N mineralisation rate in the incubation study 3 and total plant N uptake in the pot-culture experiment 1. The results showed that total N uptake was highly related with the net nitrification rates ( $r^2 = 0.58\text{--}0.87$ ), but not related to the ammonification rates. As a result, total plant N uptake was significantly correlated with N mineralisation ( $r^2 = 0.42$ ,  $P = 0.023$  and  $r^2 = 0.74$ ,  $P = 0.003$ ) with presence and absence of BS addition, respectively.

These results are in agreement with other authors in previous published studies who have reported that there were moderate correlations between N mineralisation in the soil incubations and the plant N uptake in greenhouse experiments (Keeney and Bremner, 1966; Haney et al., 2001). Although the models of N mineralisation from aerobic incubation resulted in promising relationships, these authors found that such models applied for field experiments were less reliable probably due to the variation of environmental factors.

#### 5.3.2. Pot-culture experiment 2

##### 5.3.2.1. Effects of control treatments on plant growth and dry matter

The treatments from ① to ⑤ were set up to determine the nitrogen requirement for this case study. The leaf length in the control treatments throughout the growing periods is shown in Fig. 5.3. The soil-only and N0 treatments showed the lowest growth in leaf due to nitrogen deficiency. For fertilized treatments, the leaf length rapidly increased from 15 days after sowing (DAS) and there were significant differences ( $P < 0.05$ ) between application rates of nitrogen during 15–41 DAS. The order of leaf length was  $N600 > N300 > N150 > N0 = \text{soil-only}$ , indicating a clear response of plant to increasing amount of applied nitrogen.

Similarly, plant dry matter showed a pronounced increase in leaf and root production between fertilized and unfertilized treatments. Plant dry matter as affected by chemical N application rates was shown in Fig. 5.4. Total plant dry matter was highest at treatment of N600, significantly different from the other treatments. At harvest, the order of total plant dry matter was  $N600 > N300 > N150 > N0$ . The application rate of nitrogen at  $600 \text{ mg N pot}^{-1}$ , therefore, was used as reference for later sets of experiment.



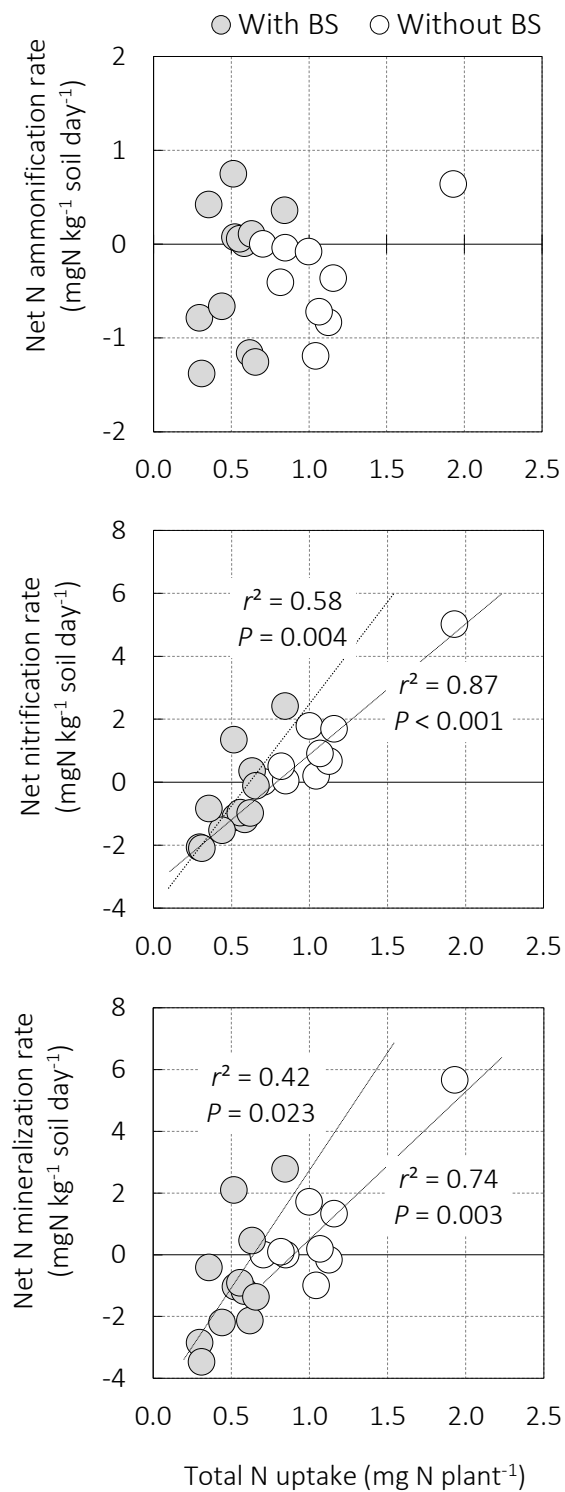


Fig. 5.2. Relationship between soil net ammonification rate, net nitrification rate, net N mineralisation rate and total plant N uptake as affected by BS addition with controlled C/N=25

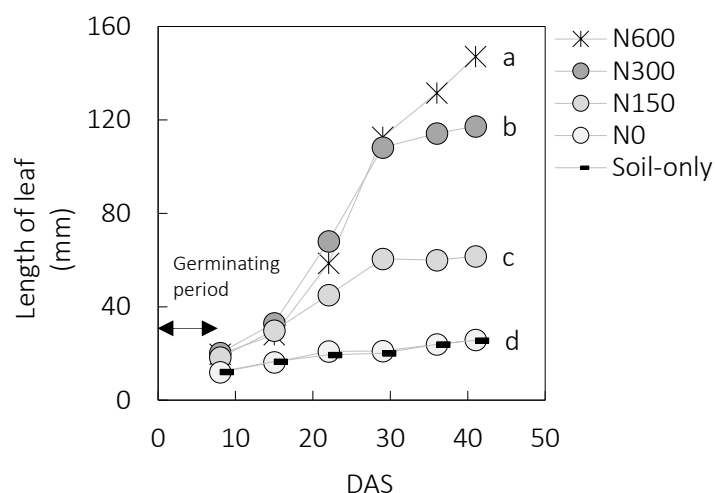


Fig. 5.3. Leaf length as affected by CF application rate.

Different letters indicate significant differences among treatments ( $P < 0.05$ )

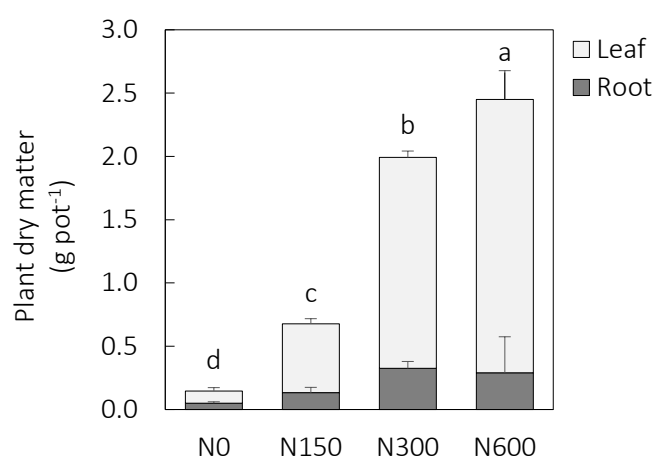


Fig. 5.4. Plant dry matter as affected by CF application rate. Bars represent the standard deviation of the mean ( $n = 3$ ). Different letters indicate significant differences among treatments ( $P < 0.05$ )

#### 5.3.2.2. Effects of sludge/compost treatments on plant growth and dry matter

The treatments from (6), (7), (8) and (9) were employed to assess the overall quality of finished composts and identify if there were potential phytotoxic substances caused by immature sludge. The growth of Komatsuna from emergency to harvest was greatly affected by treatments (Fig. 5.5). On 41 DAS, the length of leaf did not differ between group treatments of S1, C1, C2 vs N600, but they were significantly ( $P < 0.05$ ) higher than those of S2 and control treatment (soil-only).

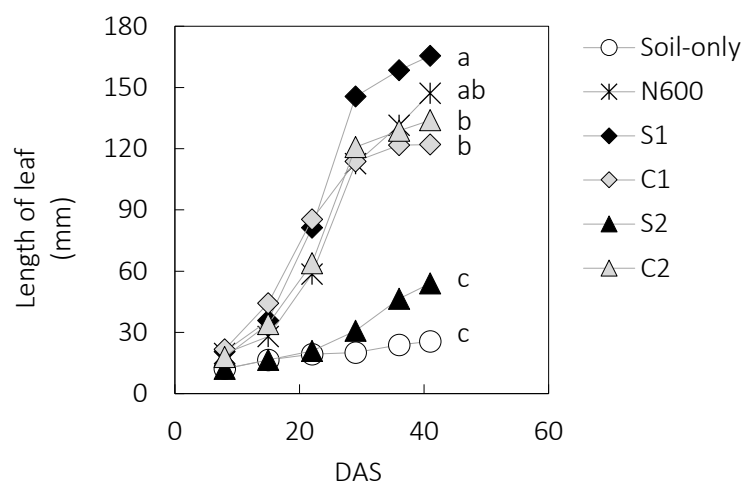


Fig. 5.5. Leaf length as affected by sludges and composts application at rate of 600 mg N pot<sup>-1</sup>

Total dry matter as the sum of leaf and root dry weight was found to be significantly affected by sludges and finished composts (Fig. 5.6). Treatments S1, C1 and C2 produced the same total dry matter (mostly as leaf dry matter) as that of N600. In contrast, total dry matter produced by S2 was very low, which was not significantly higher ( $P > 0.05$ ) than that obtained by the control treatment (soil only).

The results from the pot experiments indicated the ability of sludge and compost to supply available nitrogen for plant growth. However, the use of sludge as a soil fertiliser is not recommended due to high moisture content and high risk of phytotoxic effects as seen in sludge 2. Thus, total plant dry matter produced by sludge 2 was very low, suggesting an inhibition effect of this immature material. It should be noted that all treatments were carried out on sandy soil with a low background in buffer capacity and nutrient contents (Table 5.1); hence, the amended materials could directly affect the plant growth. The transformation of protein to amino acids is considered as the major factor limiting N availability in soil (Jones and Kielland, 2002). In another study conducted by Hara et al. (1999), free amino acids were measured using paper chromatography for evaluating the degree of swine compost maturity. Their results indicated that the growth inhibition disappeared when the free amino acids decreased and were in equilibria.

The higher amount of FAAN in the immature sludge 2 could be one of the causes of the phytotoxicity effect. In that case, the plant could not use available nitrogen sources due to blockage of soil available nitrogen by microbial activities. Padgett and Leonard (1996) indicated that the addition of an individual amino acid (2 mM glutamine, glycine, aspartic acid, or arginine) to the culture medium with 1 mM nitrate completely inhibited NO<sub>3</sub>-N uptake. The inhibition effects on growth of Komatsuna were also investigated by Kubota & Hirai |Satoshi (1983), who demonstrated that the presence of low fatty acids and relatively high values of org-C/org-N ratios in the immature composts resulted in a lower plant dry matter. In addition, the emission of ammonia from the raw materials having a high concentration of NH<sub>4</sub>-N may also contribute to the phytotoxic effect on plant growth as reported by Wong & Chu (1985).

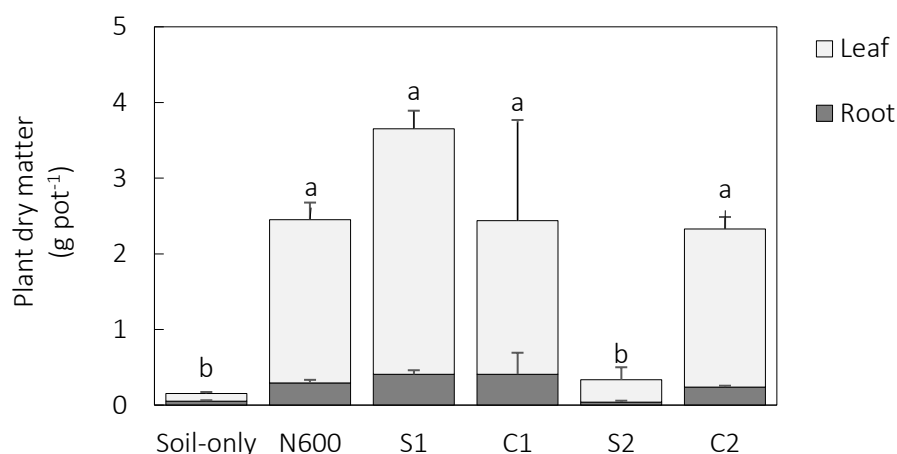


Fig. 5.6. Plant dry matter as affected by sludges and composts application at rate of 600 mg N pot<sup>-1</sup>. Bars represent standard deviation of the mean (n = 3). Different letters indicate significant differences among treatments ( $P < 0.05$ )

#### 5.3.2.3. Relationship between FAAN/TN, NH<sub>4</sub>-N/NO<sub>3</sub>-N, C/N ratios and plant dry matter

The inhibition effects were more clearly when the relationship between total plant dry matter and several important variables of sludges and composts were analyzed. The results show that total dry matter was negatively correlated with the compost FAAN/TN, NH<sub>4</sub>-N/NO<sub>3</sub>-N and C/N ratios ( $r^2 = 0.52$ ,  $r^2 = 0.53$  and  $r^2 = 0.89$ , respectively) (Fig. 5.7 A–C).

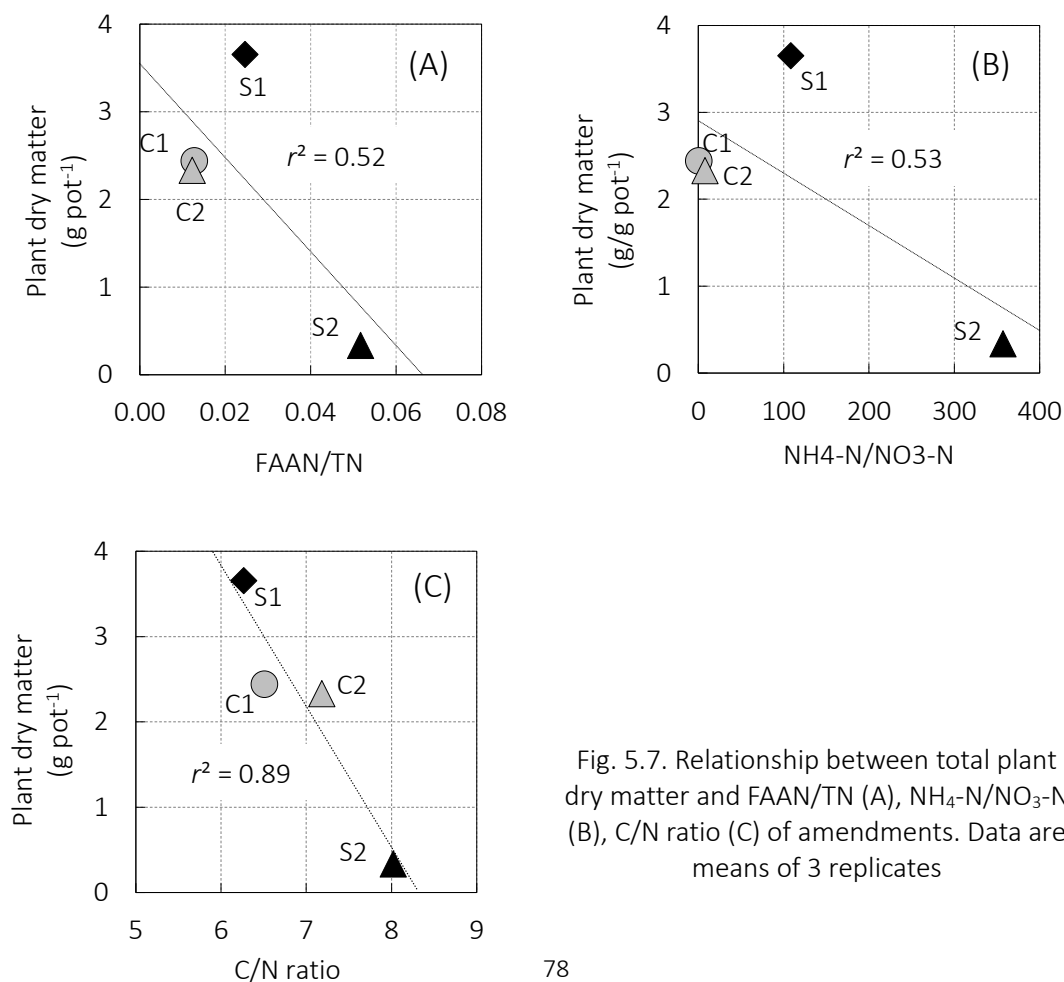


Fig. 5.7. Relationship between total plant dry matter and FAAN/TN (A), NH<sub>4</sub>-N/NO<sub>3</sub>-N (B), C/N ratio (C) of amendments. Data are means of 3 replicates

#### 5.3.2.4. Effect of sludge/compost on concentration and total uptake of P, K in plant

The concentrations of P and K in leaf as affected by sludge and compost treatments were shown in Fig. 5.8 A. Leaf P concentrations of the treatments of S1, C2 were similar to that of N600 and 3-folds higher than control treatment (soil only), while those of S2, C1 were lower and 2-folds higher than the control. Leaf K concentrations at N amended treatments were not significantly different but higher than the control treatment.

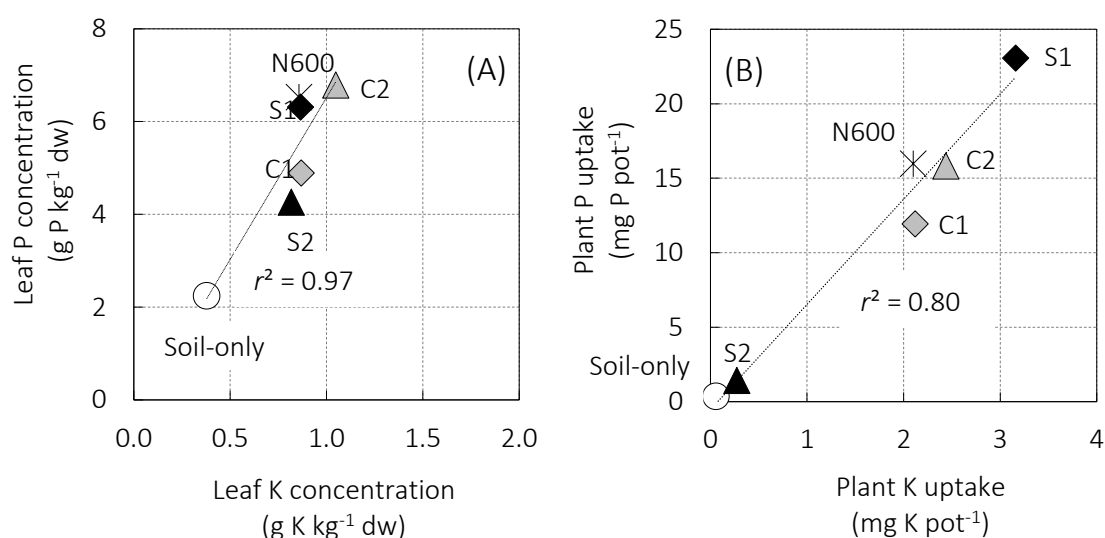


Fig. 5.8. Concentrations of P, K in leaf (A) and plant P, K uptake (B) as affected by sludges and composts application at rate of 600 mg N pot<sup>-1</sup>

The results showed that total P and K uptake in plant of the treatments were in the order of S1 > C2 = C1 = N600 > S2 = soil only (Fig. 5.8 B). The P and K uptake in plant was maybe affected by plant N uptake that caused the difference in the total dry matter as discussed in section 5.3.2.2. Significant positive relationships were found between P and K concentrations in leaf ( $r^2 = 0.80$ ) as well as total P and K uptake in plant ( $r^2 = 0.97$ ).

#### 5.3.2.5. Effect of sludge/compost on concentration of Cu and Zn in leaf

Fig. 5.9 show the accumulation of Cu and Zn in the leaf of Komatsuna after application of sludge and compost. The Zn concentrations in leaf ranged from 38 to 80 mg kg<sup>-1</sup> dw while those of Cu were less than 5 mg kg<sup>-1</sup> dw. The Zn concentrations in leaf were not clearly different between treatments but they were higher than standard limits of zinc (60 mg kg<sup>-1</sup>) for the vegetable as regulated by WHO in 1991. The accumulation of Cu in leaf was relatively low (<5 mg kg<sup>-1</sup>) compared to that of Zn. The Cu concentrations in leaf were in the order of S2 = C2 > N600 = S1 > C1 = soil only, but all values were much lower than standard limits of copper (40 mg kg<sup>-1</sup>). These results were also consistent with the heavy metal contents in sludge and compost at the beginning of the experiment (see Table 3.1, Table 3.4 and Table 5.1). However, effects of the long-term application of SS compost on heavy metal accumulation in plant-soil

system are required.

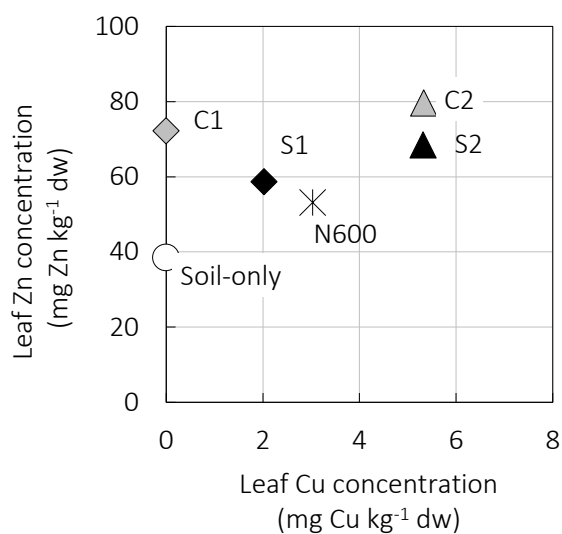


Fig. 5.9. Concentrations of Cu and Zn in leaf as affected by sludges and composts application at rate of 600 mg N pot<sup>-1</sup>

#### 5.4. Conclusions

The first pot-culture experiment indicated that the total N uptake was in the order of 100% urea > 70% compost + 30% urea = 90% compost + 10% urea = 100% compost > control treatment. When BS was added, total N uptake was significantly decreased at all treatments.

The second pot-culture experiment showed that total plant yield produced by the self-made composts was similar to chemical N fertiliser. The lowering percent of FAAN to total nitrogen, C/N ratio and NH<sub>4</sub>-N/NO<sub>3</sub>-N ratios in the composts compared to those in raw materials was found to correlate with the increase in plant yield that may reduce the phytotoxicity in the raw material.

Total N uptake from compost and chemical N fertiliser was directly correlated to net N mineralisation rates, therefore, can predict plant available N. However, the MIT processes can occur simultaneously, making it more difficult to estimate the contribution of N from each source. This subject will be discussed more in chapter 6.

## References

- Alvarenga, P., C. Mourinha, M. Farto, T. Santos, P. Palma, J. Sengo, M.C. Morais, and C. Cunha-Queda. 2015. Sewage sludge, compost and other representative organic wastes as agricultural soil amendments: Benefits versus limiting factors. *Waste Manag.* 40: 44–52.
- Brewer, L.J., and D.M. Sullivan. 2003. Maturity and stability evaluation of composted yard trimmings. *Compost Sci. Util.* 11(2): 96–112.
- Cooperband, L.R., A.G. Stone, M.R. Fryda, and J.L. Ravet. 2003. Relating compost measures of stability and maturity to plant growth. *Compost Sci. Util.* 11(2): 113–124.
- Eklind, Y., L. Salomonsson, M. Wivstad, and B. Rämert. 2016. An International Journal for Sustainable Production Systems Use of Herbage Compost as Horticultural Substrate and Source of Plant Nutrients Use of Herbage Compost as Horticultural Substrate and Source of Plant Nutrients. 8765(May).
- Faithfull, N.T. 2002. *Methods in Agricultural Chemical Analysis: A Practical Handbook*. CABI publishing.
- El Fels, L., M. Zamama, A. El Asli, and M. Hafidi. 2014. Assessment of biotransformation of organic matter during co-composting of sewage sludge-lignocellulosic waste by chemical, FTIR analyses, and phytotoxicity tests. *Int. Biodeterior. Biodegrad.* 87: 128–137.
- Haney, R.L., F.M. Hons, and M.A. Sanderson. 2001. A rapid procedure for estimating nitrogen mineralization in manured soil. *Biol. Fertil. Soils* 33: 100–104.
- Hara, M., K. Hirose, and H. Ishikawa. 1999. Detection of free amino acids using paper chromatography for evaluating degree of compost maturity. *Japanese J. Soil Sci. Plant Nutr.* 70: 306–314.
- Jones, D.L., and K. Kielland. 2002. Soil amino acid turnover dominates the nitrogen flux in permafrost-dominated taiga forest soils. *Soil Biol. Biochem.* 34(2): 209–219.
- Keeney, D.R., and J.M. Bremner. 1966. Comparison and Evaluation of Laboratory Methods of Obtaining an Index of Soil Nitrogen Availability<sup>1</sup>. *Agron. J.* 58: 498–503
- Kubota, H., and M.F. Hirai | Satoshi. 1983. Effects of compost maturity on growth of komatsuna (*Brassica Rapa* var. *pervidis*) in neubauer's pot. *Soil Sci. Plant Nutr.* 29(3): 251–259.
- Onofri, A., and E. Pannacci. 2014. Spreadsheet tools for biometry classes in crop science programmes. *Commun. Biometry Crop Sci.* 9(2): 3–13.
- Padgett, P.E., and R.T. Leonard. 1996. Free amino acid levels and the regulation of nitrate uptake in maize cell suspension cultures. *J. Exp. Bot.* 47(300): 871–883.
- Pedra, F., A. Polo, A. Ribeiro, and H. Domingues. 2007. Effects of municipal solid waste compost and sewage sludge on mineralization of soil organic matter. *Soil Biol. Biochem.* 39(6): 1375–1382.
- Said-Pullicino, D., F.G. Erriquens, and G. Gigliotti. 2007. Changes in the chemical characteristics of water-extractable organic matter during composting and their influence on compost stability and

- maturity. *Bioresour. Technol.* 98(9): 1822–1831.
- Tan, K.H. 2000. *Environmental Soil Science*, Third Edition. CRC Press, UK.
- Walter, I., F. Martí'nez, and V. Cala. 2006. Heavy metal speciation and phytotoxic effects of three representative sewage sludges for agricultural uses. *Environ. Pollut.* 139(3): 507–514.
- Warman, P.R., and W.C. Termeer. 2005. Evaluation of sewage sludge, septic waste and sludge compost applications to corn and forage: Yields and N, P and K content of crops and soils. *Bioresour. Technol.* 96(8): 955–961.
- Wong, M.H., and L.M. Chu. 1985. The responses of edible crops treated with extracts of refuse compost of different ages. *Agric. Wastes* 14(1): 63–74.



## Chapter 6: Nitrogen dynamics in plant-soil system

### 6.1. Introduction

Understanding of nitrogen dynamics in plant–soil system plays an important role in sustainable development of agricultural and environmental systems. In the preceding chapter, total nitrogen uptake from compost and chemical N fertiliser was found to positively correlate with the net N mineralisation rates. As discussed earlier, however, the MIT processes can occur simultaneously, making it more difficult to estimate the contribution of N from each source.

Combining compost and inorganic fertiliser has generally been more effective than a separate application of either material alone. Adding nitrogen also aims to ensure the available N for plant uptake because most of the compost-N is in organic forms and thus they are not immediately available for plant uptake. Asghar et al. (2006) indicated that compost combined with 50% recommended inorganic N fertiliser gave almost same results as the 100% N fertiliser alone. However, such above report provides very little information on the partitioning of applied N as well as the availability of nitrogen from compost.

The introduction of isotopic pool dilution and tracer technique has proven very advantageous in determining the transformation and fate of fertiliser N in the ecosystem (Wolf et al., 1994; Hood et al., 1999; Barbanti et al., 2011). Basically, it is assumed that the  $^{15}\text{N}$  is assimilated and transformed identically to  $^{14}\text{N}$  in plant–soil system. The nitrogen isotope ratios can, therefore, be used as a tool to differentiate the origin of nitrogen supply.

The availability of N from organic sources has been extensively investigated under laboratory, greenhouse and field conditions. Currently, Chalk et al. (2013) reviewed publications on dynamics of compost-N in plant–soil system using  $^{15}\text{N}$  tracer. A comparison between of direct and indirect  $^{15}\text{N}$  isotope techniques for estimating crop N uptake from organic residues were also investigated by Hood et al. (1999). The indirect technique is whereby  $^{15}\text{N}$  labelled fertiliser is added to soil amended with unlabelled compost (Nishio and Oka, 2003). On the contrary, the direct technique is whereby unlabelled fertiliser is added to soil which has received  $^{15}\text{N}$  labelled compost (Hood et al., 1999).

Several studies have been published where  $^{15}\text{N}$  labelled composts were produced by using  $^{15}\text{N}$ -labelled plant feedstock after providing  $^{15}\text{N}$  enriched fertiliser (plant origin). Another alternative method is to feed the animal with  $^{15}\text{N}$ -labelled roughage or grain to obtain their faeces for composting purpose (animal origin). However, these methods usually consume a lot of time for labeling and the efficiency of incorporation of fed  $^{15}\text{N}$ -labelled into excreta is low (Chalk et al., 2013). In addition, waste based composts make it impossible to produce  $^{15}\text{N}$  enriched compost following above procedures (Asagi and Ueno, 2008). Alternately, the labelling of compost can be carried out simply by adding the manure with  $^{15}\text{N}$ -labelled fertiliser at the beginning of composting as introduced by Cheneby et al. (1994). However, only very few published studies have applied this methodology, particularly for SS composting (Oba and

Nguyen, 1982; Kchaou et al., 2010).

Therefore, the primary objective of this chapter is to study two isotopic techniques including the indirect and direct method for (1) measuring the nitrogen transformations in soil amended with compost and (2) distinguishing between sources of the mineralized N which derived from compost and from chemical N fertiliser.

## 6.2. Materials and methods

### 6.2.1. Initial experiments using $^{15}\text{N}$ labelled $(\text{NH}_4)_2\text{SO}_4$

#### 6.2.1.1. Incubation 4

A soil incubation study was conducted to determine gross total N mineralisation and gross total N immobilisation rates after combining a commercial compost with  $^{15}\text{N}$  labelled  $(\text{NH}_4)_2\text{SO}_4$ . The sandy soil was air-dried and passed through a 2-mm sieve. The soil had pH 7.1 (in 1:2.5 soil: water suspension), total C of  $0.2 \text{ g kg}^{-1}$ , total N of  $0.051 \text{ g kg}^{-1}$  and a C/N ratio of 3.9. A sample of commercial Vietnamese compost (V-comp), which was derived from municipal solid waste, was obtained from Thuy Phuong composting plant and then carried to Japan for investigation.

Soil sample (650 g, d.b.) was hand mixed well with V-comp at application rate of  $130 \text{ mg N pot}^{-1}$ , and transferred into Neubauer' pots (surface area:  $100 \text{ cm}^2$ , height: 6.6 cm).  $^{15}\text{N}$ -labelled ammonium sulfate (13.172 atom%) in solution was then added at two levels of  $130 \text{ mg N pot}^{-1}$ , 1N and  $260 \text{ mg N pot}^{-1}$ , 2N. The pots were additionally covered with aluminum foil perforated five small needle holes to ensure gas exchange and minimize water loss. Then the amended soil samples were incubated in dark at  $25^\circ\text{C}$  and  $35^\circ\text{C}$  for 10 days. During the incubation time, soil was weighed daily and adjusted with distilled water to 60% holding capacity. The sampling was destructed at 2, 5, and 10 days by removing pots in triplicate for each treatment. To ensure the homogeneity without changing inorganic nitrogen contents, soil was dried at low temperature in vacuum chambers before sampling.

Inorganic N was completely extracted from the soil samples by 2.0 M KCl (w: v = 1:5) after shaking for 1 hour. The filtered solution was immediately analyzed for  $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$  using phenol-hypochlorite method (Tan, 1996) and Vanadium (III) chloride reduction method (Doane and Horwath, 2003), respectively. After extraction, the residual soil was washed in alcohol 60%, dried and ground to a very fine powder before analyzing  $^{15}\text{N}$  immobilized in organic soil by the C-N analyzer (CORDER MT-700; Yanaco, Japan) coupled with isotope Quadrupole mass spectrometry (ULVAC).

#### 6.2.1.2. Pot-culture experiment 3

To determine if the combination of compost and chemical N fertiliser could change the soil N mineralisation and distinguish the fertiliser  $^{15}\text{N}$  uptake by ryegrass, a pot-culture experiment with Italian

ryegrass (*Lolium multiflorum* L.) was conducted from September to October 2011 in Okayama, Japan under room condition where air temperature was maintained at 35°C/25°C (day/night). Soil, two composts (V-comp, a commercial Vietnamese municipal solid waste compost; and J-comp, a commercial Japanese SS compost), and <sup>15</sup>N-labelled (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> (13.172 atom%) were prepared as the same method with above incubation study. The chemical properties of two composts are summarized in Table 6.1. All treatments except control received 100 mg P pot<sup>-1</sup> as Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>.

Ryegrass was sown at a density of 20 seeds pot<sup>-1</sup>, which was equivalent to 2000 seeds m<sup>-2</sup>, over a 0.5 cm soil surface layer to facilitate seed germination. After sowing 45 days, all plant parts (including shoot and root) were harvested separately, dried at 100°C for 26 hours. Plant parts N contents were determined using the C-N analyzer (CORDER MT-700; Yanaco, Japan). Shoot and root dry weight were multiplied by the respective N content, and then summarized in total plant N uptake. All treatments were arranged in a randomized complete block design with three replications. The details of different treatments are given in Table 6.2.

#### 6.2.1.3. Calculations and statistical analysis

Total plant N uptake derived from chemical N fertiliser (N<sub>dfCF</sub>) was calculated directly using the formula of Hauck and Bremner (1976):

$$N_{dfCF} = TNU (c - b) / (a - b) \quad (1)$$

where, TNU is total N uptake in plant part; *a*, *b*, and *c* are the atom% <sup>15</sup>N-labelled (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> (13.172%), plant part or soil receiving no <sup>15</sup>N, and plant part receiving <sup>15</sup>N, respectively. The denominator *b* value in the formula (1) was assumed to be equal to natural abundance (0.366%). Total plant N uptake derived from compost (D<sub>dfComp</sub>), from soil (N<sub>dfs</sub>) and nitrogen uptake efficiency (NUE) were calculated using difference method of Westerman and Kurtz (1971). Total plant N uptake in this pot-culture experiment is given in unit of mg N pot<sup>-1</sup>.

The gross N mineralisation rate was simply calculated using the following derivation of isotope dilution:

$$G_{mi} \left( \text{mg N kg}^{-1} \text{soil} \right) = \frac{\Delta^{15}\text{N}_{\text{Org}} + \left( \Delta \text{N}_{\text{Inorg}} \times {}^{15}\text{N}_{\text{Inorg}(0)} \text{atom \%} \right)}{{}^{15}\text{N}_{\text{Inorg}(0)} \text{atom \%} - {}^{15}\text{N}_{\text{Org}(0)} \text{atom \%}} \quad (2)$$

where, <sup>15</sup>N<sub>Org(0)</sub> and <sup>15</sup>N<sub>Inorg(0)</sub> are <sup>15</sup>N atom% in organic and inorganic pools, respectively, on day 0. Δ<sup>15</sup>N<sub>Org</sub> is difference of <sup>15</sup>N content (mg N kg<sup>-1</sup>) in organic pools between day *t* and day 0. ΔN<sub>Inorg</sub> is the difference (mg N kg<sup>-1</sup>) of total inorganic nitrogen (NH<sub>4</sub> + NO<sub>3</sub>) between day *t* and day 0. For isotopic signal data from mass spectrometry, <sup>15</sup>N atom% in organic soil was calculated from following formula:

$${}^{15}\text{N}_{\text{Org}} \text{atom \%} = 100 / \left[ 1 + 2 \times \left( {}^{28}\text{N}_2 / {}^{29}\text{N}_2 \right) \right] \quad (3)$$

To obtain  $G_{mi}$  of each interval,  $^{15}\text{N}_{\text{Inorg}(t)}$  was calculated indirectly by subtracting  $^{15}\text{N}_{\text{Org}(t)}$  on day  $t$  from total initial known  $^{15}\text{N}_{[\text{Org}+\text{Inorg}](0)}$  on day 0. Therefore, in this model, the loss of N as unaccounted proportions was assumed to be negligible.

Gross N immobilisation rate ( $G_{im}$ ) was calculated using the equations of Murphy et al. (2003):

$$G_{im} \left( \text{mg} \cdot \text{kg}^{-1} \text{soil} \right) = G_{mi} - N_{mi}$$

where,  $N_{mi}$  is net mineralisation rate calculated by the difference of total inorganic nitrogen between day  $t$  and 0.

Means of values were compared with those of others by Fisher's protected least significant difference (LSD) at  $P < 0.05$  and  $P < 0.01$  after ANOVA test, using the EXCEL<sup>®</sup> macro add-ins DSAASTAT Version 1.512 (Onofri and Pannacci, 2014).

Table 6.1. Chemical properties of composts in incubation 4 and pot-culture experiment 3

Constituent	V-comp	J-comp
pH	8.7	7.2
Total carbon ( $\text{g kg}^{-1}$ )	123.0	248.1
Total nitrogen ( $\text{g kg}^{-1}$ )	11.4	37.7
C/N ratio	10.8	6.6
Inorganic N		
NH <sub>4</sub> -N ( $\text{g kg}^{-1}$ )	0.97	11.65
NO <sub>3</sub> -N ( $\text{g kg}^{-1}$ )	0.03	0.79
Total phosphorus ( $\text{g kg}^{-1}$ )	-	53.2
Exchangeable cations ( $\text{cmol kg}^{-1}$ )		
K <sup>+</sup>	15.6	12.6
Na <sup>+</sup>	10.0	6.3
Ca <sup>2+</sup>	34.0	16.6
Mg <sup>2+</sup>	5.6	33.0
Total heavy metal ( $\text{mg kg}^{-1}$ )		
Zn	202.1 (5.3) <sup>†</sup>	205.4 (0.3)
Cu	318.6 (3.5)	137.7 (0.5)
Cd	8.2	8.0
Ni	99.3	18.3

<sup>†</sup>Numbers in parentheses are available heavy metals

Table 6.2. Treatment combinations and weight of ingredients in the incubation 4 and the pot-culture experiment 3

Treatment	Soil (mg N pot <sup>-1</sup> )	V-comp (mg N pot <sup>-1</sup> )	J-comp (mg N pot <sup>-1</sup> )	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> (mg N pot <sup>-1</sup> )	Total N (mg N pot <sup>-1</sup> )
① Control (0N)	33.15	–	–	–	0
② CF (1N)	33.15	–	–	130	130
③ CF (2N)	33.15	–	–	260	260
④ V-comp	33.15	130	–	–	130
⑤ V-comp + 1N	33.15	130	–	130	260
⑥ V-comp + 2N	33.15	130	–	260	390
⑦ J-comp	33.15	–	130	–	130
⑧ J-comp + 1N	33.15	–	130	130	260
⑨ J-comp + 2N	33.15	–	130	260	390

#### 6.2.2. Later experiments on <sup>15</sup>N labelled compost

##### 6.2.2.1. Production of <sup>15</sup>N-labelled compost

As described in chapter 3, one of the four composting runs was labelled with <sup>15</sup>N artificially-enriched compost. The amount of added (<sup>15</sup>NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> was calculated based on the amount of native NH<sub>4</sub>-N in sludge sample (see Table 3.1). For this purpose, fresh sludge (5.09 kg d.b.) was spread over a blue sheet. A <sup>15</sup>N labelled ammonium solution (6.34 g (<sup>15</sup>NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> in 600 ml H<sub>2</sub>O, 99.74 atom%) was uniformly sprayed onto the surface of the sludge at the beginning of composting. After labelling <sup>15</sup>N, sludge was mixed with woodchips and loaded into compost bin at the same time with other runs 2, 4 and 5. The finished composts were homogenized and then ground (< 2 mm) for the later pot-culture experiment.

##### 6.2.2.2. Pot-culture experiment 4

The procedures of this experiment were the same with those described in the pot-culture experiment 1 (section 5.2.1.1). The sandy soil (86% sand, 5% silt and 9% clay) had pH<sub>H2O</sub> value of 7.3, total carbon of 1.0 g kg<sup>-1</sup>, total nitrogen of 0.2 g kg<sup>-1</sup> and C/N ratio of 5.0. The fresh soil (3.02 kg d.b.) passing through a 2-mm sieve was put in Wagner pots (200 cm<sup>2</sup> surface area and 20 cm height). In the compost treatment, <sup>15</sup>N labelled compost (0.639 atom%), unlabelled chemical N fertiliser as (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and RS were thoroughly mixed with 1.0 kg of the top soil at 0-5 cm depth at a rate of 600 mg N pot<sup>-1</sup> (Table 2). All treatments except the control (soil only) were supplemented with chemical fertilisers to give a total of 450 mg P pot<sup>-1</sup> and 300 mg K pot<sup>-1</sup>. After mixing soil with fertilisers, Komatsuna (*Brassica rapa* var. *perviridis*) was sown at a density of 15 seeds pot<sup>-1</sup> and then thinned to 12 plants pot<sup>-1</sup>. For

irrigation, water was introduced into pipelines and automatically sprinkled on the plant-soil system using a time delay relay to ensure the normal growth of Komatsuna. After 41 days of cultivation, all plant parts (leaf and root) were separately harvested and dried at 105°C for 26 hours. Soil was collected at two depths (0-5 cm and 5-20 cm) for further laboratory analysis. All treatments were arranged in a randomized complete block design with three replications. The details of different treatments are given in Table 6.3.

For analysis of  $^{15}\text{N}$  enrichment in compost, soil and tissue, dry samples were ground to a very fine powder before analyzing  $^{15}\text{N}$  isotope analyse using a PDZ Europa ANCA-GSL elemental analyser interfaced to a PDZ Europa 20-20 isotope ratio mass spectrometer (Sercon Ltd., Cheshire, UK).

### 6.2.2.3. Calculations

$$N_{\text{dfComp}} = \text{TN} \times \% N_{\text{dfComp}} = \text{TN} \times ({}^{15}\text{N enrichment}_{\text{plant or soil}}) / ({}^{15}\text{N enrichment}_{\text{compost}})$$

where:  $N_{\text{dfComp}}$ , total N uptake in plant parts or remaining in soil after cropping derived from compost; TN, total N uptake in plant parts or remaining in soil in soil;  ${}^{15}\text{N enrichment}_{\text{plant or soil}}$  was calculated by subtracting  ${}^{15}\text{N}$  atom% of plant parts or soil receiving unlabelled compost from those of labelled compost;  ${}^{15}\text{N enrichment}_{\text{compost}}$  was calculated by subtracting  ${}^{15}\text{N}$  atom% of unlabelled compost from that of labelled compost. Total plant N uptake derived from chemical N fertiliser ( $N_{\text{dfCF}}$ ) and from soil ( $N_{\text{dfs}}$ ) were calculated using difference methods (Powlson and Barraclough, 1992). Total plant N uptake in this pot-culture experiment is given in the unit of mg N pot<sup>-1</sup>.

Table 6.3. Treatment combinations and weight of ingredients in pot-culture experiment 4

Treatments	Added RS (mg C kg <sup>-1</sup> soil)	N sources (mg N pot <sup>-1</sup> )			C/N <sup>a</sup>	C/N <sup>b</sup>
		<sup>15</sup> N-labeled compost	( <sup>14</sup> NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	Total		
① Soil-only	0	0	0	0	-	-
② 100% CF	0	600	0	600	7.3	7.3
③ 100% CP	0	600	0	600	-	-
④ 75% CP 25%CF	0	450	150	600	5.5	7.3
⑤ 50% CP 50% CF	0	300	300	600	3.6	7.3
⑥ 75% CP 25%CF/RS	3465	450	150	600	11.3	15
⑦ 50% CP 50% CF/RS	2310	300	300	600	7.5	15

C/N<sup>a</sup> total carbon to total nitrogen; C/N<sup>b</sup> total carbon to total N of compost only. RS = rice straw powder

The % recovery of  $^{15}\text{N}$  in plant or in soil derived from compost was calculated using the formula of Hauck and Bremmer (1976):  ${}^{15}\text{NR}_{\text{Comp}} = 100 \times N_{\text{dfComp}}/R$ ; where: R, amount of total applied N from

compost. The possible N leaching/loss from the surface soil (0–5 cm) was estimated by subtracting N uptake and remaining in soil from initial applied N values as described by Ueno and Yamamuro (2001).

### 6.3. Results and discussion

#### 6.3.1. The experiments using $^{15}\text{N}$ labelled $(\text{NH}_4)_2\text{SO}_4$

##### 6.3.1.1. Gross total N transformation and net N mineralisation rates

The incubation 4

Table 6.4 summarizes data of gross total N transformation and net N mineralisation rates in soil. Some  $^{15}\text{N}$  data of V-comp + 2N treatment on day 5 were not available due to the failure of the isotopic analysis system. Net mineralisation rates ( $N_{mi}$ ) were calculated by the difference of total inorganic nitrogen (data not shown). The results show that temperature and added N rates had significant effects ( $P < 0.05$  and  $P < 0.01$ , respectively) on net mineralisation rates during the incubation. The results show that  $N_{mi}$  rates in all treatments had large negative values, suggesting a net immobilisation in soil (since immobilisation > mineralisation). Except for V-comp + 1N at 35°C during 5–10 days of incubation,  $N_{mi}$  shows a small positive value (2.68 mg kg<sup>-1</sup> soil day<sup>-1</sup>).

Although  $N_{mi}$  rates are useful to evaluate the size of inorganic nitrogen in soil, they do not provide further information regarding flow of nitrogen into or out of each N pool. Therefore, the gross total N mineralisation ( $G_{mi}$ ) and gross total N immobilisation ( $G_{im}$ ) are more useful to understand the interaction between organic and inorganic sources. The results show that  $G_{im}$  rates were much higher than  $G_{mi}$  at almost intervals of incubation.  $G_{im}$  was also affected by the change of temperature, which was significantly higher ( $P < 0.05$ ) at 25°C than 35°C. At the same temperature, V-comp + 2N gave larger  $G_{im}$  than did V-comp + 1N. In contrast,  $G_{mi}$  rates were not affected by temperature, while those of V-comp + 2N were significantly higher ( $P < 0.01$ ) than V-comp + 1N.

After 10 days of incubation, the results show that 16.08–29.62 mg N kg<sup>-1</sup> soil day<sup>-1</sup> from inorganic-N were immobilised into organic pools, while only 0.40–0.66 mg N kg<sup>-1</sup> soil day<sup>-1</sup> from organic-N were mineralised to inorganic pools.

The net mineralisation occurs when compost had C/N ratios lower than 20:1 and N concentration is greater than 1.6% (Ozores-Hampton and Obreza, 1999). However, Hartz et al. (1996) noticed that compost nitrogen could also be immobilized for a short time despite a low C/N ratio (<12) due to N concentration lower than 1.6%. In this study, such high values of gross immobilisation rates and low values gross mineralisation rates measured at the initial stage may be related to term “lag period”, in which the amount of microbial biomass N increased remarkably, as reported by Aoyama and Nozawa, (1993). The pattern of net immobilisation herein for 10 days incubation study was consistent with other literature (Hadas and Portnoy, 1994; Yevdokimov et al., 2012). They indicated that the portion of added

inorganic nitrogen was immobilized by soil microbial community. Despite a temporary reduction of mineral N, retention of nitrogen in the compost amended soil could reduce environmental risk of N losses from agriculture land.

Table 6.4. Gross total N transformation and net N mineralisation rates as affected by temperature and inorganic-N addition to compost

Time	Temp.	Treatments	Gross mineralisation (mg N kg <sup>-1</sup> soil day <sup>-1</sup> )	Gross immobilisation (mg N kg <sup>-1</sup> soil day <sup>-1</sup> )	Net (mg N kg <sup>-1</sup> soil)
t = 0- 2	25°C	V-comp + 1N	1.90	83.70	-81.78
		V-comp + 2N	2.66	118.59	-115.93
	35°C	V-comp + 1N	1.65	68.90	-67.20
		V-comp + 2N	1.06	56.89	-55.83
t = 2- 5	25°C	V-comp + 1N	0.23	1.50	-1.27
	35°C	V-comp + 1N	1.97	13.90	-11.93
t = 5- 10	25°C	V-comp + 1N	0.52	1.07	-0.55
	35°C	V-comp + 1N	2.95	0.24	2.68
t = 0- 10	25°C	V-comp + 1N	0.42	17.43	-17.01
		V-comp + 2N	0.66	29.62	-28.96
	Mean		0.54	23.50	-22.99
	35°C	V-comp + 1N	0.40	16.08	-15.68
		V-comp + 2N	0.55	26.50	-25.94
	Mean		0.48	21.30	-20.81
P > F	Temp		ns	*	*
	N rate		**	**	**
	Temp × N rate		ns	ns	ns

\* and \*\* indicate significant differences at  $P = 0.05$  and  $0.01$ , respectively

A sharp increase of organic <sup>15</sup>N atom% after combining compost with <sup>15</sup>N labelled ammonium sulfate (data not shown) indicated that there was a considerable input of <sup>15</sup>N from inorganic N pools. These were properly corresponding to the significant decrease of NH<sub>4</sub>-N content of the inorganic N pools, as observed by Compton and Boone (2002). The addition net cumulative nitrate NO<sub>3</sub>-N released after 10 days of incubation, suggesting that microorganisms (nitrifiers) could be activated by consumption of NH<sub>4</sub>-N and producing NO<sub>3</sub>-N. The effects of temperature and added N rates from chemical fertiliser on gross total N transformation were also observed. These data could provide initial information about nitrogen cycling between organic and inorganic N sources in soil. The slight decrease of immobilisation rates as affected by higher temperature in this study could attribute to a possible immobilisation by microbial biomass rather than abiotic physical fixation. Joergensen et al. (1990) indicated that increasing



incubation temperature from 25°C to 35°C resulted in enhanced substrate decomposition due to increased specific death rates of microorganisms.

#### 6.3.1.2. Total plant N uptake and partitioning of total plant N uptake derived from compost

Table 6.5 shows the partitioning of nitrogen derived from compost ( $N_{dfComp}$ ), chemical N fertilizer ( $N_{dfCF}$ ), and from soil ( $N_{dfs}$ ) as influenced by different fertiliser treatments in the pot-culture experiment 3. Thus,  $N_{dfComp}$  indicates more clearly the difference between compost alone and blend treatments.  $N_{dfComp}$  was significantly decreased ( $P < 0.01$ ) with two additional N rates from CF compared to compost alone, except for V-comp + 1N treatment. When combining J-comp with CF at rates of 1N and 2N, the  $P_{Ndfcomp}$  decreased from 82% to 27% and 23%, respectively. This means that approximately one-third of the total plant N uptake was derived from compost when treated with compost and CF at a ratio of 1:1. The  $N_{dfCF}$  values were useful to compare difference between CF alone and blend treatments. Similarly, combining CF with either V-comp or J-comp caused a significant decrease ( $P < 0.01$ ) in  $N_{dfCF}$  compared to CF alone, except for V-comp + 1N. Consequently, the  $P_{NdfCF}$  were also lowered from 73–81% to 52–66% and to 43–53% for V-comp and J-comp blended treatments, respectively (Table 6.5).

$N_{dfs}$  of CF fertilized treatments were observed to be higher than CF unfertilized treatments. These values were not used for statistical analysis within because data were simulated from control and CF alone treatments.

Total nitrogen uptake (TNU) was generally similar to total dry weight data (*data not shown*). The only differences were that N uptake from V-comp + 1N was significantly higher ( $P < 0.01$ ) than V-comp + 2N, V-comp + 2N was higher than V-comp, and V-comp was higher than control treatment. The results also show that combining composts with CF did not increase the TNU and NUE compared to CF alone. Except for V-comp + 1N treatment, TNU was remarkably enhanced by 137.69 mg N pot<sup>-1</sup>. Remarkably, TNU and NUE of J-comp (98.83 mg N pot<sup>-1</sup> and 62.0%) were similar to CF (1N) (103.31 mg N pot<sup>-1</sup> and 65.4%), but significantly higher ( $P < 0.01$ ) than V-comp alone (55.24 mg N pot<sup>-1</sup> and 28.2%, respectively).

Although some initial results were obtained from the experiments using <sup>15</sup>N labelled (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, these data may be not satisfactory. However, it was suspected that some problems could occur using <sup>15</sup>N isotope dilution technique to measure gross total nitrogen transformation and N uptake from compost inputs. If <sup>15</sup>N labelled (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and compost were added simultaneously, there may be a rapid reduction of <sup>15</sup>N of the N pool and the amount of immobilized nitrogen could be subsequently re-mineralized that may cause the pool substitution and underestimation of the calculation. Or in the other hand, the re-mineralisation of immobilized <sup>15</sup>N can lead to errors associated with the isotope dilution method.

Therefore, the use of <sup>15</sup>N labelled composts allows a different approach to distinguish better the nitrogen derived from compost. This subject will be discussed in the next section.

Table 6.5. Total plant nitrogen uptake and partitioning of total plant N derived from compost, CF, and soil as affected by different treatments

Treatments	Compost source		CF source		Soil	Total	N uptake
	N <sub>dfComp</sub>	P <sub>Ndfcomp</sub>	N <sub>dfCF</sub>	P <sub>NdfCF</sub>	N <sub>dfs</sub>	N uptake	efficiency
	(mg N pot <sup>-1</sup> )	(%)	(mg N pot <sup>-1</sup> )	(%)	(mg N pot <sup>-1</sup> )	(mg N pot <sup>-1</sup> )	(%)
Control (0N)	-	-	-	-	18.27	18.27 d	-
CF (1N)	-	-	75.48 b	73	27.83	103.31 b	65.4 a
CF (2N)	-	-	111.09 a	81	26.67	137.76 a	46.0 b
V-comp	36.97 b	67	-	-	18.27	55.24 c	28.2 c
V-comp + 1N	37.66 b	27	72.20 bc	52	27.83	137.69 a	45.9 b
V-comp + 2N	4.61 c	5	59.91 bc	66	26.67	91.19 b	18.7 c
J-comp	80.56 a	82	-	-	18.27	98.83 b	62.0 a
J-comp + 1N	24.60 b	27	39.03 d	43	27.83	91.46 b	28.4 c
J-comp + 2N	24.46 b	23	57.39 c	53	26.67	108.52 b	23.2 c
<i>P</i> > <i>F</i>	**		**			**	**
LSD	18.89		17.60			24.96	13.28

Values within the same column followed by the same letter are not significantly different according to LSD test at  $P < 0.05$

### 6.3.2. The experiments using <sup>15</sup>N-labelled compost

#### 6.3.2.1. Immobilisation of <sup>15</sup>N into organic N pool during composting process

##### Composting run 3 (cont.)

The immobilisation process of <sup>15</sup>N into organic N pool after labelling started from the beginning of composting process. Before labelling with <sup>15</sup>N, sludge had the natural <sup>15</sup>N abundance of 0.371 atom% that was a little bit higher than that in the atmosphere (0.366 atom%). After labelling, the <sup>15</sup>N atom% in total N pool immediately increased to 0.551% and fluctuated between 0.575% and 0.617% between day 4 and day 46. Meanwhile, the values of <sup>15</sup>N atom% in organic nitrogen pool were gradually increased with composting indicating that the successful immobilisation of (<sup>15</sup>NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> into the organic form. The analysis of three different samples (at top – middle – bottom locations) gave similar results (percent difference of less than  $\pm 5\%$ ), indicating that labelling was homogenous. As a result, the enrichments of <sup>15</sup>N in total N pool and organic N pool at the end of composing were 0.639% and 0.609%, respectively. The majority of <sup>15</sup>NH<sub>4</sub>-N was immobilized during the first 21 days when the difference between <sup>15</sup>N atom percentage of two pools was gradually reduced with composting time (Fig. 6.1).

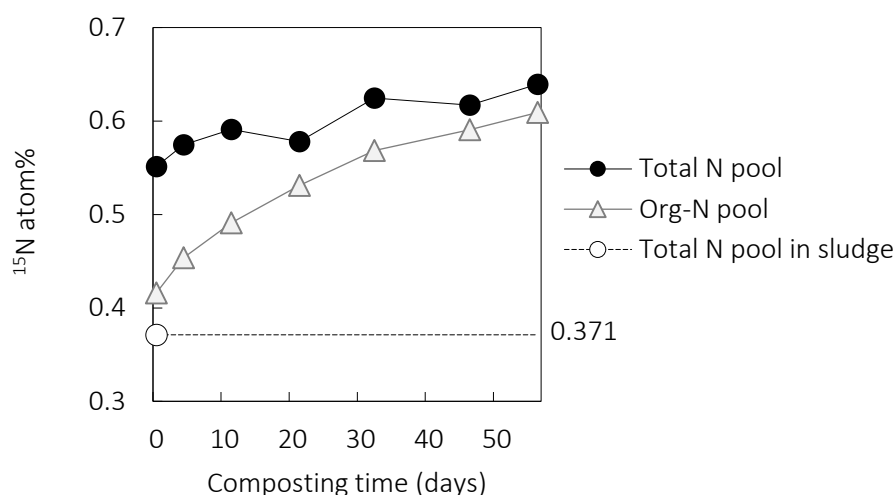


Fig. 6.1.  $^{15}\text{N}$  atom percent in pools of total N and organic during composting process after labelling with  $(^{15}\text{NH}_4)_2\text{SO}_4$  at the beginning of the experiment. Values are means of 2 or 3 replicates

These results were also consistent with the changes in  $\text{NH}_4\text{-N}$  during composting process as reported in section 3.3.2.4, providing a useful information about the appropriate time for adding with  $^{15}\text{N}$ -labelled fertiliser. Therefore, labelling should be done as soon enough before composting process entered the curing phase to ensure the significant immobilisation of  $^{15}\text{N}$  into organic forms. However, careful control of the composting process is required to minimize N losses via  $\text{NH}_3$  volatilization in the initial stages of composting as reported by (Witter and Lopez-Real, 1988). The immobilisation of  $^{15}\text{N}$  into the organic pool was similar to the results of Tran et al. (2012), who studied turnover of manure  $^{15}\text{N}$ -labelled ammonium during composting as affected by lime and superphosphate addition. They found that 16-62% of the initial  $^{15}\text{NH}_4\text{-N}$  was immobilized into the compost from day 1 to day 19 depending on additional materials.

#### 6.3.2.2. Leaf, root and total plant dry matter

Table 6.6 shows leaf, root and total plant dry matter of Komatsuna as affected by N sources and combinations with RS. The total plant dry matter was contributed mostly by leaf dry matter rather than root dry matter. A single application of SS compost (CP) or chemical N fertiliser (CF) led to a significant increase in leaf, root and total plant dry matter compared to the control treatment (soil-only). Among the combination treatments between CP and CF, leaf dry matter and total plant dry matter were higher with higher proportion of CF but there was no significant difference in leaf and total plant dry matter between two combinations in the absence of RS. As a result, the treatment of 50% CP 50% CF resulted in the highest production. The addition of RS into compost significantly decreased the leaf, root and total plant matter, and total plant dry matter of treatment 75% CP 25% CF/RS compared to no addition of RS

but they were not different from the control treatment.

It should be noted that all treatments, except for control (soil-only), received the same amounts of phosphorus and potassium. Therefore, the differences between treatments were governed by the contribution of carbon and nitrogen sources.

Table 6.6. Leaf, root and total plant dry matter as affected by N sources and rice straw addition

Treatments	Leaf dry matter (g pot <sup>-1</sup> )	Root dry matter (g pot <sup>-1</sup> )	Total plant dry matter (g pot <sup>-1</sup> )
① Soil-only	0.10 d	0.05 c	0.16 d
② 100% CF	2.16 bc	0.29 ab	2.45 bc
③ 100% CP	2.46 b	0.26 b	2.72 b
④ 75% CP 25%CF	2.84 ab	0.24 b	3.08 ab
⑤ 50% CP 50% CF	3.26 a	0.38 a	3.64 a
⑥ 75% CP 25% CF/RS	0.60 d	0.09 c	0.69 d
⑦ 50% CP 50% CF/RS	1.72 c	0.26 b	1.99 c
<i>P</i> > <i>F</i>	**	**	**
LSD	0.69	0.10	0.73

Values within the same column followed by the same letter are not significantly different according to LSD test at  $P < 0.05$

### 6.3.2.3. Nitrogen uptake by plant and N derived from compost and chemical N fertiliser

The supply of available N reflected in cumulative plant N uptake. The results of nitrogen uptake by leaf, root and total plant were given in Table 6.7. Not surprisingly, most of plant N uptake was accumulated in leaves. The effects of N sources and RS addition was similar to those of dry matter. However, leaf and total plant N uptake at treatment of 100% CF were 1.6-fold higher than that of 100% CP although having the similar dry matter production. Thus, the N concentrations in leaf of 100% CF treatment were 2 times higher than that of 100% CP (data not shown), indicating the more ready availability of nitrogen from CF than CP. The maximum total plant N uptake was attained when N was applied with a half of CP and a half of CF, which was comparable to 100% CF and significantly different from other recipes. The incorporation of RS remarkably reduced the N uptake in both leaf, root and total plant which were consistent with the decrease in total plant dry matter.

These results reflected that N uptake depends very much on the N mineralisation from CF and compost as discussed in the Section 5.3.1.2 of Chapter 5. The incorporation of RS into compost caused

the soil N immobilisation when its C/N ratio was adjusted up to 15. The nitrogen uptake from soil amended with compost greatly decreased with the rice straw treatments was also observed by Luo et al. (2004), who studied the fate of nitrogen derived from composts and urea in soil under long-term N management in pot experiments. Their results indicated that N uptake by rice was as high as 36–45% of total applied N under chemical N application, but as low as 5–6% when rice straw-compost was applied to soils.

Table 6.7. Leaf, root and total plant N uptake as affected by N sources and rice straw addition

Treatments	Leaf N uptake (mg N pot <sup>-1</sup> )	Root N uptake (mg N pot <sup>-1</sup> )	Total plant N uptake (mg N pot <sup>-1</sup> )
① Soil-only	0.93 e	0.44 e	1.37 e
② 100% CF	86.69 ab	7.04 ab	93.73 ab
③ 100% CP	53.12 c	4.65 bc	57.77 c
④ 75% CP 25%CF	74.67 b	4.27 bc	78.94 b
⑤ 50% CP 50% CF	98.04 a	9.20 a	107.23 a
⑥ 75% CP 25%CF/RS	8.05 e	1.39 de	9.44 e
⑦ 50% CP 50% CF/RS	28.59 d	3.73 cd	32.32 d
<i>P</i> > <i>F</i>	**	**	**
LSD	17.16	2.07	18.05

Values within the same column followed by the same letter are not significantly different according to LSD test at *P* < 0.05. CF = chemical N fertilizer; CP = compost; RS = rice straw

Table 6.8 shows the nitrogen uptake derived from CP and/or from CF. It should be noted that the amount of N uptake derived from blank soil is not accounted for N uptake in this calculation because it has been subtracted from the total values as shown in Table 6.6. The results show that total N uptake and leaf N uptake from compost was significantly improved in the treatment of 75% CP 25%CF compared to the treatment of 100% CP. Whereas, total plant N uptake, leaf N uptake and root N uptake from CF significantly decreased in the presence of compost compared to the treatment of 100% CF. The co-application of high rate of compost and low rate of CF perhaps stimulated the immobilisation of inorganic N into organic forms and then re-mineralisation of immobilized N to provide available N for plant uptake. The plant N uptake from CF source was less in the presence of compost at each rate of application that was emphasized in the review of N dynamics of compost N in the plant–soil system using <sup>15</sup>N tracer (Chalk et al., 2013). The same author indicated that compost promoted the retention of fertiliser N in soil through biological immobilisation, and therefore, decreased overall N losses to the environment.

Thus, the nitrogen derived from compost showed a majority contribution to total N uptake, indicating a very available N source from compost. The incorporation of RS into compost caused an apparent decrease in total N uptake from both compost and CF sources in all plant parts.

Table 6.8. Leaf, root and total plant N uptake derived from compost and CF

Treatments	Leaf N uptake (mg N pot <sup>-1</sup> )		Root N uptake (mg N pot <sup>-1</sup> )		Total plant N uptake (mg N pot <sup>-1</sup> )	
	N <sub>dfComp</sub>	N <sub>dfCF</sub>	N <sub>dfComp</sub>	N <sub>dfCF</sub>	N <sub>dfComp</sub>	N <sub>dfCF</sub>
② 100% CF	–	85.49 a	–	6.60 a	–	92.36 a
③ 100% CP	52.19 b	–	4.21 a	–	56.39 b	–
④ 75% CP 25%CF	73.75 a	0.00 c	3.13 a	0.70 b	76.87 a	0.70 cd
⑤ 50% CP 50% CF	56.11 b	37.54 b	3.82 a	4.93 a	59.93 b	45.93 b
⑥ 75% CP 25%CF/RS	7.12 c	0.00 c	0.66 b	0.29 b	7.78 c	0.29 d
⑦ 50% CP 50% CF/RS	16.91 c	5.09 c	1.50 b	1.77 b	18.41 c	12.52 c
<i>P</i> > <i>F</i>	**	**	**	**	**	**
LSD	11.83	10.84	1.19	1.68	11.52	12.08

Values within the same column followed by the same letter are not significantly different according to LSD test at *P* < 0.05. CF = chemical N fertilizer; CP = compost; RS = rice straw

#### 6.3.2.4 Total Nitrogen retention in the surface soil

The soil nitrogen retention in the surface soil (0–5 cm) derived from compost in the treatment of 100% compost was 2.5-fold higher than that from chemical N fertilizer in the treatment of 100% CF (Table 6.9). When compost was combined with CF, soil N<sub>dfComp</sub> and total N retention in soil decreased with increasing percent of CF. Or on the other hand, compost contributed very significantly to the N remaining in the soil. When a half of nitrogen was supplied from each source as treatment ⑤, N<sub>dfcomp</sub> and soil N<sub>dfCF</sub> were 202.84 mg N pot<sup>-1</sup> and 171.77 mg N pot<sup>-1</sup>, respectively. The incorporation of RS into compost and CF significantly increased the soil N<sub>dfCF</sub> rather than soil N<sub>dfcomp</sub>. As a result, total N remaining in the surface soil at the end of pot-culture experiment strongly differed between treatments, highest in the treatments of ⑥, ③ and ⑦ where compost was applied singly or co-applied with CF in the presence of RS, and lowest in the treatment of ① (soil-only). These results confirm that organic N inputs from compost may indeed be of the key role for N retention in the soil system. Additional RS would reduce N

losses from chemical N fertiliser by promoting microbial N immobilisation.

The application of compost has reported previously to increase N and C inputs in the surface soil in short-term and long-term experiments. For example, it was found that compost-N remaining in the top soil (0–30 cm) accounted for 34.2% and 42.5% of total application rate of 148 and 442 kg N ha<sup>-1</sup>. In a long-term study (30 years) on paddy soil, total N concentrations in 0–15 cm soil layer were found to increase about 5.6% per year with the annual application of 36 tons ha<sup>-1</sup> year<sup>-1</sup> on wet basis (Nishida et al., 2007). The use of <sup>15</sup>N-labelled compost proved a very useful method to estimate the N retention in soil derived from different N sources. More than 91% of applied N was recovered in soil when rice straw compost or cattle compost was applied to two soils of long-term continuous application of organic N sources (straw compost + soybean cake) and chemical N fertiliser (Luo et al., 2004).

#### 6.3.2.5. Total nitrogen leaching/loss from the surface soil

Nitrogen denitrification, volatilization and leaching or movement of N downward to the lower soil layer can result in N losses from the surface soil that is irrelevant from the agronomic and environmental point of view. In this experiment, the losses of applied N were estimated by the difference between the amount of <sup>15</sup>N recovered from soil + plant at harvest and the known amount of <sup>15</sup>N applied at the beginning of the experiment. The highest loss of N derived from chemical N application and total N loss were observed in the treatment of 100% CF (Table 6.10). The N loss from CF significantly increased from 0 to 297.49 mg N pot<sup>-1</sup> with the increase of the proportion of CF from 25% to 100%. Meanwhile, the N losses from compost and total N loss were not significantly different ( $P > 0.05$ ) between treatments in the presence of compost. The N loss from CF significantly decreased when RS was incorporated to soil. Thus, the higher RS was added, the higher N loss was reduced from CF. These results indicated that compost not only improve the N uptake but also prevent N loss by leaching from the surface soil. The combined incorporation of compost and RS to soil increased N retention in soil, therefore minimize the N losses due to microbial nitrogen immobilisation.

Table 6.9. Total soil N retention in the surface soil derived from compost and CF

Treatments	Soil N <sub>dfcomp</sub> (mg N pot <sup>-1</sup> )	Soil N <sub>dfCF</sub> (mg N pot <sup>-1</sup> )	Soil total N retention (mg N pot <sup>-1</sup> )
① Soil-only	—	—	28.54 e
② 100% CF	—	210.16 b	238.70 d
③ 100% CP	530.71 a	—	559.25 a
④ 75% CP 25%CF	292.32 bc	119.32 d	440.17 bc
⑤ 50% CP 50% CF	202.84 c	171.77 c	403.15 c
⑥ 75% CP 25%CF/RS	391.26 b	177.72 bc	597.51 a
⑦ 50% CP 50% CF/RS	254.00 c	250.05 a	532.60 ab
<i>P</i> > <i>F</i>	**	**	**
LSD	105.59	35.50	103.20

Values within the same column followed by the same letter are not significantly different according to LSD test at *P* < 0.05. CF = chemical N fertilizer; CP = compost; RS = rice straw

Table 6.10. Leaching/loss of nitrogen from the surface soil derived from compost and CF

Treatments	Leaching N <sub>dfcomp</sub> (mg N pot <sup>-1</sup> )	Leaching N <sub>dfCF</sub> (mg N pot <sup>-1</sup> )	Total N Leaching (mg N pot <sup>-1</sup> )
② 100% CF	—	297.49 a	297.49 a
③ 100% CP	12.89	—	12.89 b
④ 75% CP 25% CF	80.81	29.99 cd	110.80 b
⑤ 50% CP 50% CF	37.23	82.29 b	119.53 b
⑥ 75% CP 25% CF/RS	22.96	0.00 d	22.96 b
⑦ 50% CP 50% CF/RS	27.59	37.43 c	65.01 b
<i>P</i> > <i>F</i>	ns	**	**
LSD	104.25	36.45	115.13

Values within the same column followed by the same letter are not significantly different according to LSD test at *P* < 0.05. CF = chemical N fertilizer; CP = compost; RS = rice straw

#### 6.3.2.6. Models of N dynamics in plant–soil system

The distribution of applied N in plant–soil system and percent of N loss at the end of experiment was significantly affected by N sources and RS addition (Fig. 6.2). The recovery of applied N in plant ranged



from a low of 0.05% in treatment (6) to a high of 17.6% in the treatment (5). The percent N remaining in the surface soil ranged from 35.0% to 94.8%, lowest in the treatment (2) (35.0%) and highest in the treatment (6) (94.8%). The percent of N loss was also highest in the treatment (2) (49.6%) but lowest in the treatment (3) (2.1%) when soil was applied with 100% compost.

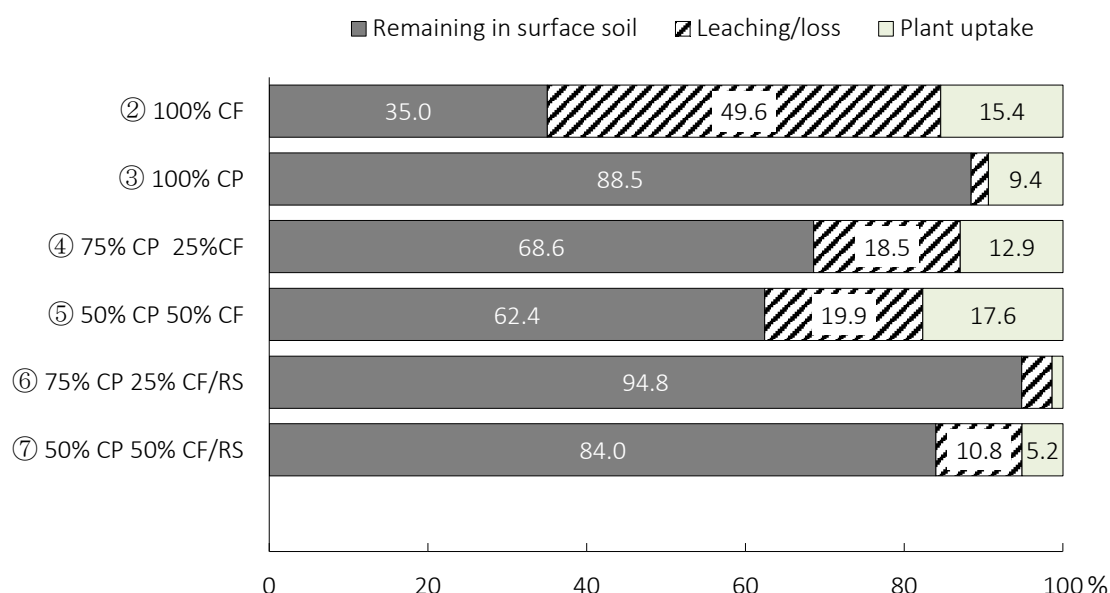


Fig. 6.2. The distribution of applied N in plant–soil system as affected by N sources and rice straw addition. Numbers indicate percent of total applied N at rate of 600 mg N pot<sup>-1</sup>. CF = chemical N fertilizer; CP = compost; RS = rice straw

The use of <sup>15</sup>N tracer allows quantifying the recovery of applied N in various supply sources. Three N dynamic models, called control (CTRL), non-rice straw addition (NRSA) and rice straw addition (RSA), were developed to estimate the plant N uptake, N retention in soil and N loss from compost and chemical N fertiliser. For CTRL, where compost or CF was applied singly to the soil, N uptake from CF was 1.6 times higher than from compost, but half of total applied N was lost by N leaching or moving to lower soil layer (Fig. 6.3). For NRSA models where compost was combined with CF, for example in the treatment (4), about 12.8% and 0.1% of total applied N (600 mg N pot<sup>-1</sup>) derived from compost and CF were taken up by plant, respectively. Meanwhile, about 48.7% and 19.9% of applied N derived from compost and chemical N fertiliser were remained in the soil, respectively. Consequently, N loss accounted for 13.5% and 5.0% which derived from compost and CF, respectively. In another recipe, the combination ratio of 1:1 (as seen in treatment (5)) can reduce the N leaching by 2.5 times while maintaining a comparable N uptake to conventional application of 100% CF (treatment (2)), in which 56% of the total N uptake derived from compost source (Table 6.8 and Fig. 6.4). For RSA models where compost was combined

with CF in the presence of RS. For example, in the treatment ⑥, about 1.3% and 0.05% of total applied N were taken up by plant which derived from compost and CF, respectively. The nitrogen retention in soil derived from compost and chemical N fertiliser accounted for about 65.2% and 29.6% of total applied N. Likewise, N losses accounted for 13.5% and 5.0% of applied N which derived from compost and chemical N fertiliser sources, respectively (Fig. 6.5). Thus, the incorporation of RS into compost and CF as shown in the RSA resulted in the highest retention of N in the surface soil.

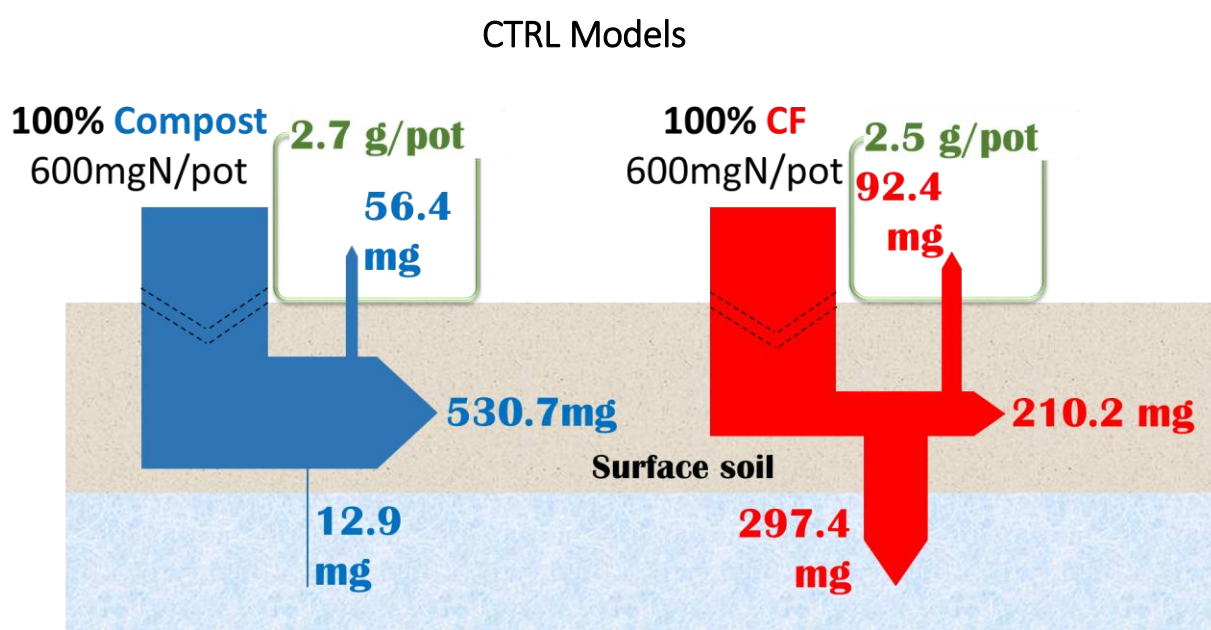


Fig. 6.3. N dynamics in plant–soil system as affected by the single application of CF and compost. The numbers in green, red and blue color indicate total plant dry weight (g pot<sup>-1</sup>), the amount of N derived from ammonium sulfate and compost (mg N pot<sup>-1</sup>), respectively

## NRSA Models

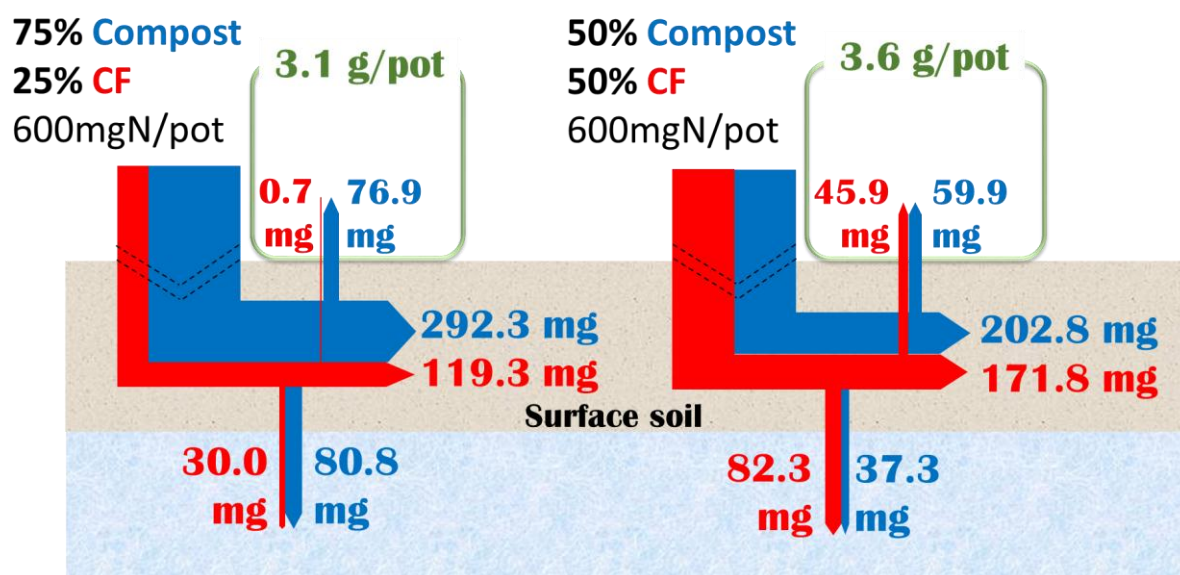


Fig. 6.4. N dynamics in plant–soil system as affected by the combined application of CF and compost in the absence of RS. The numbers in green, red and blue color indicate total plant dry weight (g pot<sup>-1</sup>), the amount of N derived from ammonium sulfate and compost (mg N pot<sup>-1</sup>), respectively

## RSA Models

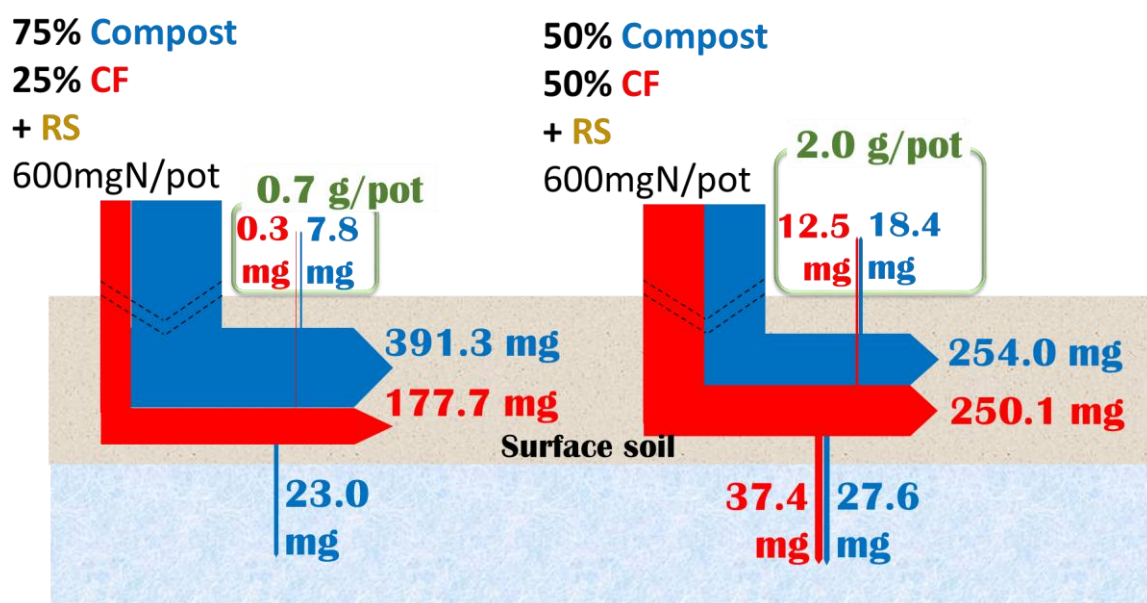


Fig. 6.5. N dynamics in plant–soil system as affected by the combined application of CF and compost in the presence of RS. The numbers in green, red and blue color indicate total plant dry weight (g pot<sup>-1</sup>), the amount of N derived from ammonium sulfate and compost (mg N pot<sup>-1</sup>), respectively

#### 6.4. Conclusions

The soil N transformation was changed when compost was supplemented with inorganic N fertiliser. The incubation study indicated that 16.08–29.62 mg N kg<sup>-1</sup> soil day<sup>-1</sup> from inorganic-N were immobilized into organic pools, while only 0.40–0.66 mg N kg<sup>-1</sup> soil day<sup>-1</sup> from organic-N were mineralized to inorganic pools.

The <sup>15</sup>N data in the pot-culture experiment using isotope dilution approach showed that 29% of added N derived from solids compost, while 56% of added N derived from chemical N fertiliser. In this study, the N recovery by plant was likely to be lower in the presence of compost, indicating that compost promoted the retention of fertiliser N in soil through microbial immobilisation.

Labeling the compost with <sup>15</sup>N allows more accurate estimation of plant N uptake, N retention in soil and N leaching/loss in plant–soil system in comparison to isotope dilution technique. From the above-mentioned results, the following conclusion regarding the use of <sup>15</sup>N labelled compost can be drawn. For making <sup>15</sup>N-labelled compost, the majority of added <sup>15</sup>NH<sub>4</sub>-N was immobilized into organic forms of compost during the first 21 days of composting. Therefore, labelling can be done as soon as possible before composting process entered the curing phase to ensure the significant immobilisation of <sup>15</sup>N into organic forms. However, the loss of N during this period should be also taken into consideration.

The application of sewage sludge compost not only maintain the dry matter production but also increase N uptake under combined application with chemical N fertiliser at appropriate rates. Interestingly, the results suggested that the compost-N contributed more than a half of total nitrogen uptake and nitrogen retention in soil at two combination ratios of 2:1 and 1:1 based on levels of N from compost and chemical N fertiliser. Three models of N dynamics can be understood. For CTRL model, where compost or chemical N fertiliser was applied singly to the soil, N uptake from CF was 1.6 times higher than from compost, but half of total applied N was lost by N leaching or moving to lower soil layer. For NRSA model, the combination of 1:1 ratio can reduce the N leaching by 2.5 times while maintaining similar N uptake to CF alone, in which 56% total N uptake derived from compost. The incorporation of rice straw into compost and CF as shown in the RSA model resulted in the highest retention of N in the surface soil.

## References

- Aoyama, M., and T. Nozawa. 1993. Microbial biomass nitrogen and mineralization-immobilization processes of nitrogen in soils incubated with various organic materials. *Soil Sci. Plant Nutr.* 39(1): 23–32.
- Asagi, N., and H. Ueno. 2008. Determination of application effects of Sewage sludge on growth , soil properties , and N uptake in Komatsuna by using the indirect  $^{15}\text{N}$  isotope method. *Commun. Soil Sci. Plant Anal.* 39: 1928–1942.
- Asghar, H.N., M. Ishaq, Z.A. Zahir, M. Khalid, and M. Arshad. 2006. Response of radish to integrated use of Nitrogen fertilizer and recycled organic waste. *Pakistan J. Bot.* 38(3): 691–700.
- Barbanti, L., M. Grigatti, and C. Ciavatta. 2011. Nitrogen release from a  $^{15}\text{N}$ -labeled compost in a sorghum growth experiment. *J. Plant Nutr. Soil Sci.* 174(2): 240–248.
- Chalk, P.M., A.M.T. Magalhães, and C.T. Inácio. 2013. Towards an understanding of the dynamics of compost N in the soil-plant-atmosphere system using  $^{15}\text{N}$  tracer. *Plant Soil* 362(1–2): 373–388.
- Cheneby, D., B. Nicolardot, B. Godden, and M. Penninckx. 1994. Mineralization of composted  $^{15}\text{N}$ -labelled farmyard manure during soil incubations. *Biol. Agric. Hortic.* 10(May 2015): 255–264.
- Compton, J.E., and R.D. Boone. 2002. Soil nitrogen transformations and the role of light fraction organic matter in forest soils. *Soil Biol. Biochem.* 34(7): 933–943.
- Doane, T. a, and W.R. Horwath. 2003. Spectrophotometric determination of nitrate with a single reagent. *Anal. Lett.* 36(12): 2713–2722.
- Hadas, A., and R. Portnoy. 1994. Nitrogen and Carbon Mineralization Rates of Composted Manures Incubated in Soil. *J. Environ. Qual.* 23: 1184–1189.
- Hartz, T.K., F.J. Costa, and W.L. Schrader. 1996. Suitability of Composted Green Waste for Horticultural Uses. 31(6): 961–964.
- Hauck, R.D., and J.M. Bremner. 1976. Use of Tracers For Soil And Fertilizer Nitrogen Research. *Adv. Agron.* 28(C): 219–266.
- Hood, R.C., K.N. Goran, M. Aigner, and G. Hardarson. 1999. A comparison of direct and indirect  $^{15}\text{N}$  isotope techniques for estimating crop N uptake from organic residues. *Plant Soil* 208: 259–270.
- Joergensen, R.G., P.C. Brookes, and D.S. Jenkinson. 1990. Survival of the soil microbial biomass at elevated temperatures. *Soil Biol. Biochem.* 22(8): 1129–1136.
- Kchaou, R., M.N. Khelil, F. Gharbi, S. Rejeb, B. Henchi, T. Hernandez, J.P. Destain, I. National, D. Recherche, G. Rural, and F. Bp. 2010. Isotopic Evaluations of Dynamic and Plant Uptake of N in Soil Amended with  $^{15}\text{N}$ -Labelled Sewage Sludge. 19(2): 363–370.
- Luo, L., M. Kondo, and S. Itoh. 2004. Fate of  $^{15}\text{N}$  derived from composts and urea in soils under different long-term N management in pot experiments. *Compost Sci. Util.* 12(October 2014): 18–24.
- Murphy, D. V, S. Recous, E.A. Stockdale, I.R.P. Fillery, L.S. Jensen, D.J. Hatch, and K.W.T. Goulding. 2003.

- Gross Nitrogen Fluxes in Soil: Theory, Measurement and Application of  $^{15}\text{N}$  Pool Dilution Techniques. *Adv. Agron.* 79: 69–118.
- Nishida, M., K. Iwaya, H. Sumida, and N. Kato. 2007. Changes in natural  $^{15}\text{N}$  abundance in paddy soils under different , long-term soil management regimes in the Tohoku region of Japan. : 310–317.
- Nishio, T., and N. Oka. 2003. Soil Science and Plant Nutrition Effect of Organic matter application on the fate of N-labeled ammonium fertilizer in an upland soil Effect of Organic Matter Application on the Fate of  $^{15}\text{N}$ -Labeled Ammonium Fertilizer in an Upland Soil. *Soil Sci. Plant Nutr.* 49(3): 397–403.
- Oba, Y., and Q.-L. Nguyen. 1982. Decomposition of sewage sludge in soil. *Soil Sci. Plant Nutr.* 28(1): 119–130.
- Onofri, A., and E. Pannacci. 2014. Spreadsheet tools for biometry classes in crop science programmes. *Commun. Biometry Crop Sci.* 9(2): 3–13.
- Ozores-Hampton, M., and T.A. Obreza. 1999. Evaluation of in situ N Mineralization of Composted Organic Wastes Applied to Sandy Soil. *HortScience* 34(3): 486.
- Powlson, D.S., and D. Barraclough. 1992. Mineralization and assimilation in soil-plant systems. In *Nitrogen Isotope Techniques*. Eds. T H Blackburn and R Knowles. pp 209–242. Academic Press, San Diego.
- Tan, K.H. 1996. *Soil Sampling, Preparation, and Analysis*. Marcel Dekker, New York.
- Tran, T.M., L.S. Jensen, and J. Luxhøi. 2012. Turnover of manure  $^{15}\text{N}$ -labelled ammonium during composting and soil application as affected by lime and superphosphate addition. *Soil Sci. Soc. Am. J.* 77: 190–201.
- Ueno, H., and S. Yamamuro. 2001. Fate of nitrogen derived from  $^{15}\text{N}$ -labeled plant residues and composts in rice-planted paddy soil Hideo. *Soil Sci. Plant Nutr.* 47(4): 747–754.
- Westerman, R.L., and L.T. Kurtz. 1971. Isotopic and Nonisotopic Estimations of Fertilizer Nitrogen Uptake by Sudangrass in Field Experiments. *Soil Sci. Soc. Am. J.* 38: 107–109.
- Witter, E., and J. Lopez-Real. 1988. Nitrogen losses during the composting of sewage sludge, and the effectiveness of clay soil, zeolite, and compost in adsorbing the volatilized ammonia. *Biol. Wastes* 23(4): 279–294.
- Wolf, D.C., J.O. Legg, and T.W. Boutton. 1994. Isotopic Methods for the Study of Soil Organic Matter Dynamics. In: Weaver, R.W., Angle, S., Bottomly, P., Bezdicek, D., Smith, S., Tabatabai, A. and Wollum, A., Eds., *Methods of Soil Analysis. Microbiological and Biochemical Properties*, SSSA, Madison. Part 2: 865–906.
- Yevdokimov, I. V, A. Gattinger, F. Buegger, M. Schlöter, and J.C. Munch. 2012. Changes in the Structure and Activity of a Soil Microbial Community Caused by Inorganic Nitrogen Fertilization. 81(6): 803–809.

## Chapter 7. Conclusion

Several steps can be taken to understand the dynamics of SS compost N in the plant-soil system. The first approach is to make high-quality compost using a simple aeration method under different seasonal temperature and aerobic conditions. Better results of composting were achieved in the summer than in the winter with more woodchips being used suggesting a strategy for controlling of temperature and aeration. The compost temperatures were maintained above 55°C for three consecutive days when SS was mixed with woodchips in ratios of 1:1 and 1:0.5 (v/v). The self-made composts had pH values of 5.7–6.9, C/N ratios of 6.5–7.3, TN of 44.1–50.6 g kg<sup>-1</sup>, FAAN of 0.3–0.6 g kg<sup>-1</sup> and NH<sub>4</sub>-N/NO<sub>3</sub>-N of 1.0–8.0, which were similar to the commercial composts in Japan. The overall decrease in free amino acid-N at the end of the process corresponded to the stability of other physicochemical properties that can be regarded as an indicator of compost maturity.

The second avenue is to study the availability of N from compost after soil application as affected by chemical N fertiliser (CF) and different carbon sources from plant residues like bamboo stem, wood for building and rice straw. It should be noted that nitrification was predominant in soil amended with compost. It is not surprising that the higher supplement of CF to compost resulted in an increase in soil N mineralisation, whereas the addition of plant residues caused a significant decrease in soil N mineralisation due to a temporary immobilisation of nitrate nitrogen. The good quality of added carbon, i.e. bamboo stem and rice straw, is likely the important factor to control the soil N mineralisation. The results from pot-culture experiments were consistent with those in the incubation studies, where total N uptake by plant was positively correlated with the net N mineralisation rates ( $r^2 = 0.42\text{--}0.74$ ), that may explain for the availability of N from compost and CF. However, the mineralisation-immobilisation turnover processes can occur simultaneously, making it more difficult to estimate the contribution of N from each source. In addition, the results of pot-culture experiments indicated that the lowering ratios of FAAN/TN, C/N and NH<sub>4</sub>-N/NO<sub>3</sub>-N in the composts compared to those of SS were found to correspond to the increase in plant yield that may reduce the phytotoxicity in the raw material.

In the third approach, the use of the <sup>15</sup>N-labelled compost (0.639 atom%) as a tracer established three N dynamic models, called control (CTRL), non-rice straw addition (NRSA) and rice straw addition (RSA), by which the plant N uptake, N retention in soil and N loss from different N sources were determined. For CTRL, where compost or CF was applied singly to the soil, N uptake from CF was 1.6 times higher than from compost, but half of total applied N was lost by N leaching or moving to lower soil layer. For NRSA, the combination of 1:1 ratio of N from compost and CF can reduce the N leaching by 2.5 folds while maintaining similar N uptake to CF alone, in which 56% total N uptake was derived from compost. The incorporation of RS into compost and CF as shown in the RSA resulted in the highest

retention of N in the surface soil.

Overall, these results suggest a simple aerobic composting technique for recycling of SS and checking the compost quality based on the changes in temperature, nitrogen availability and plant growth bioassays. The tropical climate appears to be more favorable to maintain optimum temperature during composting process. The N mineralisation from composts can be controlled by combining with CF and/or bioavailable carbon sources by which their C/N ratios were modified. The appropriate incorporation of rice straw and compost into paddy soils is of particular interest in Vietnam where the farmer used to face up to disposal of crop residues and the burning of them is strictly regulated in agricultural sites. Labeling the compost with  $^{15}\text{N}$  permitted more accurate estimation of plant N uptake, N retention in soil and N leaching/loss in plant–soil system. However, further in situ experiments using  $^{15}\text{N}$ -labelled composts are required to use the sludge feedstock of Vietnam and evaluate the effectiveness of these models to be adapted to local conditions.



## Appendix A

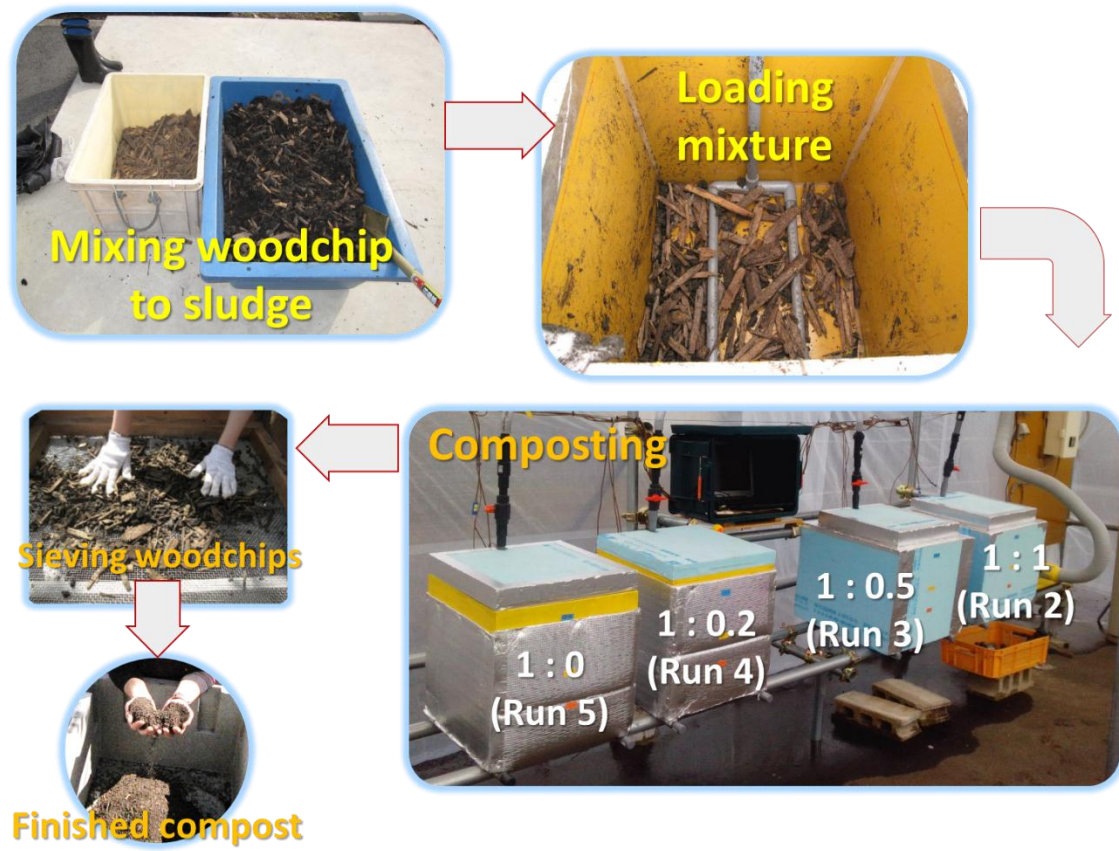


Fig. A1. An overview of aerobic composting process

Summer



Winter



Fig. A2. View of composting systems in summer (left) and winter (right), from outside (above) and inside (below) of reactors

Incubation period: 20 days



Fig. A3. View of an incubation study





Fig. A4. View of the pot-culture experiment



Fig. A5. Visible response of Komatsuna to composts and sludges

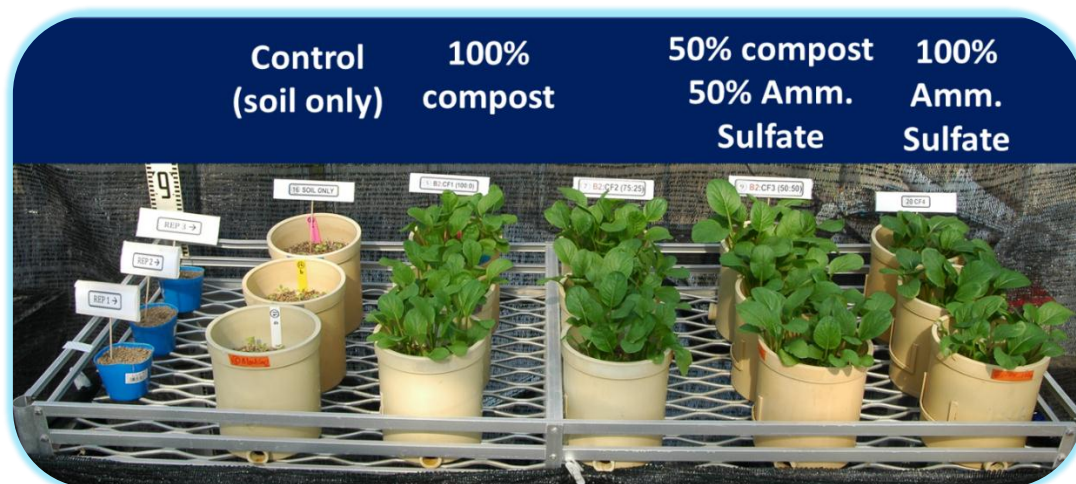


Fig. A6. Visible response of Komatsuna to compost and ammonium sulfate at 38 days after sowing

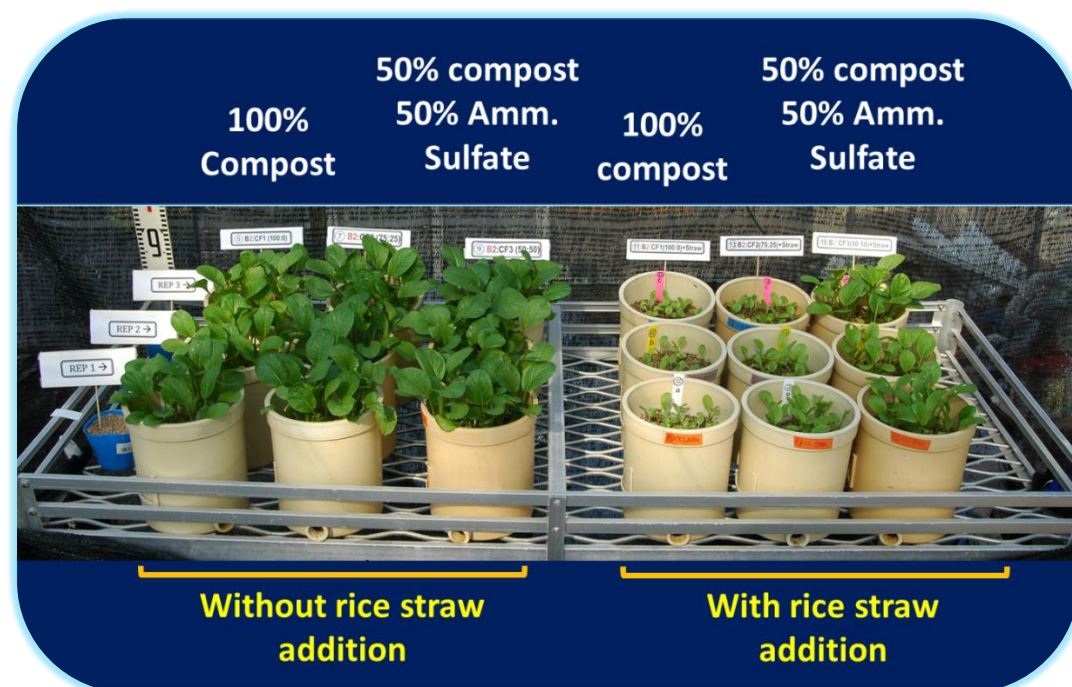


Fig. A7. Visible response of Komatsuna to compost and ammonium sulfate with and without rice straw addition