Agronomic characteristics of five different urban waste digestates

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Abstract

The use of digestate in agriculture is an efficient way to recycle materials and to decrease the use of mineral fertilizers. The agronomic characteristics of the digestates can promote plant growth and soil properties after digestate fertilization but also harmful effects can arise due to digestate quality, e.g. pH, organic matter and heavy metal content. The objective of this study was to evaluate the differences and similarities in agronomic characteristics and the value of five urban waste digestates from different biogas plants treating either food waste, organic fraction of organic solid waste or a mixture of waste-activated sludge and vegetable waste. The digestate agronomic characteristics were studied with chemical analyses and the availability of nutrients was also assessed with growth experiments and soil mineralization tests. All studied urban digestates produced 5–30% higher ryegrass yields compared to a control mineral fertilizer with a similar inorganic nitrogen concentration, while the feedstock source affected the agronomic value. Food waste and organic fraction of municipal solid

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waste digestates were characterized by high agronomic value due to the availability of nutrients and low heavy metal load. Waste-activated sludge as part of the feedstock mixture, however, increased the heavy metal content and reduced nitrogen availability to the plant, thus reducing the fertilizer value of the digestate.

Keywords

Anaerobic digestion; digestate; fertilizer value; nutrients; heavy metals; plant growth

1. Introduction

Anaerobic digestion is a widely used technique for the treatment of various organic waste materials to produce energy in the form of biogas and nutrient-rich residue, digestate. In Europe the total digestate production in 2010 was 56 Mtonnes per year of which 80–97% was used in agriculture (Saveyn & Eder, 2014). The use of digestate in agriculture has been acknowledged as an efficient way to mitigate greenhouse gas emissions through material recycling, avoidance of mineral fertilizers and improvement of soil properties as reported in several life cycle analyses (Bernstad & la Cour Jansen, 2011, Boldrin et al., 2011, Evangelisti et al., 2014). However, proper digestate management, processing and spreading techniques are needed to avoid potential acidification and eutrophication impacts due to increased nutrient leaching (Abdullahi et al., 2008, Alburquerque et al., 2012a, Bernstad & la Cour Jansen, 2011, Boldrin et al., 2011, Haraldsen et al., 2011) which is dependent on the local soil quality and meteorological conditions as well as digestate characteristics (Evangelisti et al., 2014).

The digestate agronomic characteristics, including organic matter content and quality and plant-available nutrients as well as possibly harmful properties, e.g. heavy metals and pathogens, define the effect on soils and plants (Abubaker et al., 2012, Nkoa, 2014, Teglia et al., 2011), i.e. the agronomic value of the digestate. Anaerobic digestion typically converts most of the feedstock's organic material into biogas while the nutrients of the feedstock are conserved in the digestate (Odlare et al., 2011) in more inorganic and soluble forms (Tambone et al. 2010). The soluble ammonium nitrogen increases the short-term effect of nitrogen in soils enhancing plant growth shortly after fertilization (Abubaker et al., 2012, Gutser et al., 2005). The organic matter in the digestate increases the soil carbon balance (Odlare et al., 2008, 2011) that leads to enhanced microbial processes (Abubaker et al., 2012, Odlare et al., 2008) and enzymatic activity (Galvez et al., 2012), which further increases the long-term nutrient release in soils (Abubaker et al., 2012, Odlare et al., 2008). In addition, digestate has also been reported to increase germination and plant root growth (Maunuksela et al., 2012) and soil quality by increasing water balance and soil structure (Abubaker et al., 2012). As a result, the application of the same amount of plant-available nutrients in digestates compared to mineral fertilizers has been found to produce similar and even increased crop yields compared to mineral fertilizers (Abubaker et al., 2012, Haraldsen et al., 2011, Svensson et al., 2004, Walsh et al., 2012). The amount of digestate applied to land in the EU is defined according to the national legislation which outlines the limits for nitrogen and phosphorus use per hectare. For example, in Finland the limits in cereal and grass fertilization are 170 kg/ha for organic nitrogen, 130–250 kg/ha for soluble nitrogen and 4–52 kg/ha for phosphorus depending on the plant type, yield, geographical location, soil type and phosphorus content of the soil (Government Decree No 1250/2014 on the restriction of certain discharges from agriculture or horticulture, MAVI, 2014).

Excess application of digestate can lead to harmful effects on plants and soils due to, e.g., the quantity and quality of organic matter or the impurities, including heavy metals, organic contaminants or pathogens (Alburquerque et al., 2012b, Govasmark et al., 2011). High organic matter content, depending on its composition, can lead to excess microbial activity and immobilization of nitrogen (Alburquerque et al., 2012a, Gutser et al., 2005) as well as phytotoxicity (Abdullahi et al., 2008). Feedstocks of urban biogas plants, e.g. sewage sludge and biowastes, may contain heavy metals

(Kupper et al., 2014, Odlare et al., 2008), which are concentrated in the digestate due to the mass reduction during anaerobic digestion (Govasmark et al., 2011), and possibly accumulated in the soils or in the food chain after digestate use (Otabbong et al., 1997, Zhu et al., 2014). Altogether, the characterization of the digestate organic matter, nutrient and heavy metal contents and their effects on plants and soils, i.e. the agronomic characteristics, are essential in order to plan digestate management and to control the positive and negative environmental effects of digestate fertilization.

The recent research on the use of digestates in agriculture has focused largely on digestates from agricultural feedstocks such as manure, plant biomass and a mixture of agro-industrial products and manure (e.g. Alburquerque et al., 2012a, 2012b, Fouda et al., 2013, Galvez et al. 2012, Grigatti et al., 2011, Gunnarsson et al., 2010). Furthermore, some studies have reported the effect of digestates originating from urban feedstocks, e.g. of different food and household wastes and sewage sludge, on the crop growth and nitrogen uptake (Abubaker et al., 2012, Haraldsen et al., 2011, Odlare et al., 2011, Rigby & Smith, 2014, Svensson et al., 2004) and on soil quality (Abubaker et al., 2012, Odlare et al., 2008, 2011, Rigby & Smith, 2013). As the focus of these studies is mainly on the growth response of crops, the digestate heavy metal and organic matter content are thoroughly reported only in a limited amount of studies with urban waste digestates (Abubaker et al., 2012, Tambone et al., 2010). Additionally, to the authors' knowledge there are only a few digestate fertilization/quality studies, which take the feedstock composition and origin into consideration when evaluating the fertilizer value (Tambone et al. 2009, 2010) and where the digestion process parameters are considered (Alburquerque et al. 2012b, Tambone et al. 2009). The digestate characteristics are known to be affected by the characteristics of the feedstock (Abubaker et al., 2012, Tambone et al., 2010) as well as the anaerobic digestion process; the reactor type and process parameters (Zirkler et al., 2014). In addition, the feedstock composition can also vary depending on, e.g., waste collection regulations (Saveyn & Eder, 2014) and pretreatment prior to anaerobic digestion, which may significantly affect the digestate composition (Tampio et al., 2014). However, urban feedstocks, especially food waste and household waste, have been found to have rather uniform characteristics despite temporal or geographical differences (Davidsson et al., 2007, Valorgas, 2011).

The objective of this study was to evaluate the differences and similarities in the agronomic characteristics of different urban waste digestates and to evaluate the agronomic value of these digestates. The agronomic characteristics were studied by (I) analyzing the digestate quality, including pH, organic and heavy metal content of digestates, and reflecting on the results within the context of the European digestate quality criteria and (II) analyzing the fertilizer value with chemical analyses of nutrients, soil nitrogen mineralization test and short-term ryegrass growth experiments. The aim was also to compare the effect of feedstock composition and digestion processes on the digestate agronomic characteristics by taking into consideration the pretreatment of the feedstock. Studied materials originated from anaerobic digesters from different European countries treating food waste (FW), organic fraction of organic solid waste (OFMSW) and a mixture of waste-activated sludge and vegetable waste (VWAS).

2. Materials and methods

2.1. Origin of materials

This study evaluated the agronomic characteristics of five digestates of which three originated from digesters fed with a source-segregated domestic food waste (FW), one from a digester fed with an organic fraction of municipal solid waste (OFMSW) and one from a digester fed with a mixture of waste-activated sludge and vegetable waste (mixture referred as VWAS, Figure 1, Table 1). The respective feedstocks were characterized as well except VWAS, which was not available.

Two food wastes and digestates originated from laboratory stirred tank reactors. Reactors were fed with FW collected from Ludlow, UK, where the FWs were either macerated with a S52/010 Waste Disposer (IMC Limited, UK) (feedstock and digestate referred as FW1) or autoclaved with a double-auger autoclave (160 °C and 6.2 bars, AeroThermal Group Ltd, UK) and macerated (FW2). Both Ludlow feedstocks were frozen (-20 °C) and sent to Natural Resources Institute Finland, to produce the FW1 and FW2 digestates, which were combined samples from two parallel reactors (a more detailed description of both digestates is provided in Tampio et al., 2014). Digestates were stored frozen (-20 °C), and were thawed before analysis. The third FW feedstock and digestate (FW3) were obtained from a sub-commercial-scale anaerobic digester from Greenfinch, UK. OFMSW feedstock and digestate originated from an anaerobic digestion plant in Lisbon, Portugal, treating source-segregated OFMSW from the Lisbon area. The VWAS mixture, which consisted of vegetable waste and waste-activated sludge, was from a pilot digester treating wastes from Treviso, Italy (Table 1).

The feedstocks and digestates from the UK, Portugal and Italy (excluding FW1 and FW2) were sent in frozen form to a laboratory at Natural Resources Institute Finland, where the samples were thawed and stored approximately one week at 4 °C. Prior to analyses feedstock samples were macerated with a Retch Grindomix GM300 knife mill (Retch Gmbh, Germany). From OFMSW feedstock the non-biodegradable material (plastic cups, plastic bags, etc.) was manually removed before analyses of the water soluble nutrients and carbon.

2.2. Nitrogen mineralization

Nitrogen mineralization tests were run to study the effect of digestate applications on soil inorganic nitrogen concentrations. The 48-day mineralization was tested in triplicate at 20 °C according to ISO 14238 (ISO, 2012) with digestates and control soil, where no fertilizer was added. Incubation soil (7% clay, 6% silt and 87% sand; soil organic C 1.8% and pH_w 5.1) was collected from the 0–15 cm top layer of a cultivated agricultural soil in Jokioinen, Finland. The aim was to add digestate to have 20 mg total Kjeldahl nitrogen (TKN) /100 g soil, and thus based on pre-samples 2.2–8.6 g fresh matter (FM) of different digestates were added resulting in 17–31 mg TKN/100 g soil based on analyzed samples. Soil from individual pots was sampled after 0, 4, 20 and 48 days following the start of incubation and was then frozen (-20 °C). After incubation all soil samples were thawed and 100 g moist soil was extracted with 250 ml 2 M KCl and analyzed for ammonium nitrogen (NH_4-N) and nitrate (NO_3-N). Soil inorganic N concentrations were compared against the incubated control soil.

2.3. Growth experiments

The plant availability of the nitrogen in digestates was studied via a pot experiment using the same soil as in the mineralization test. The growth of Italian ryegrass (cv. Fabio) was studied in triplicate treatments with each of the digestate and control applications. The aimed digestate addition was 1500 mg TKN/5 L sandy soil, the amount of which was calculated according to the digestate pre-samples (data not shown). However, the actual applied nitrogen addition varied from 1280 to 2390 mg/pot within digestates when calculated using the nitrogen concentrations of digestates used in the establishment of the pot experiment. Control treatments were mineral fertilizer (NH_4NO_3) applications of 0 to 2000 mg N into the pot at 500 mg N intervals. Sufficient levels of P (500 mg P/pot), K (1500 mg K/pot) and other nutrients (Mg, S, B, Cu, Mn, Mo and Zn) were applied to each pot to maintain N as the only responsive nutrient. Eleven grams of limestone was mixed into the soil of each pot to control pH and add Ca. A half gram of ryegrass seeds were evenly placed on the surface of the experimental soil in each pot. Ryegrass was grown under a glass roof outdoors at ambient air temperature for the first 110 days and for days 110–160 in a greenhouse (14 hours light in 16°C and 10 hours dark in 14°C). The grass was harvested at 30, 60 and 160 days after the start of the experiment. When harvested, ryegrass was cut leaving 2 cm-high stubble, fresh weight was measured and samples were dried at 60 °C after which dry weight (DW) was determined. Samples were milled before analyzing the TKN concentrations.

2.4. Chemical analyses

Total and volatile solids (TS and VS) were determined according to SFS 3008 (Finnish Standard Association, 1990). pH was determined using a VWR pH100 pH-analyzer (VWR International). For analysis of soluble chemical oxygen demand (SCOD) feedstock samples were diluted to 1:10 with distilled water, and agitated for 1 hour. Diluted feedstock and digestate samples were centrifuged (2493 × g, 15 min) after which the supernatant was further centrifuged (16168 × g, 10 min) and stored in a freezer, then thawed before analysis according to SFS 5504 (Finnish Standard Association, 2002a). Total COD was measured by the open reflux, titrimetric method used by the University of Southampton (modified slightly from the Vienna standard method). VFAs (volatile fatty acids: acetic, propionic, iso-butyric, n-butyric, iso-valeric, valeric and caproic acids) were analyzed using a HP 6890 gas chromatograph as described in Tampio et al. (2014). TKN was analyzed by a standard method (AOAC, 1990) using a Foss Kjeltec 2400 Analyzer Unit (Foss Tecator AB, Sweden), with Cu as a catalyst and NH_4-N determined according to McCullough (1967). After N mineralization experiments NH_4-N and NO_3-N from 2 M KCl extracts were analyzed with a Lachat autoanalyzer (Quikchem 8000, Zellweger Analytics, Inc., Milwaukee, WI, USA). Total-C was analyzed using Duma's method according to the manufacturer's instructions with a Leco CN-2000 Elemental Analyzer (Leco Corp., USA).

Soluble nutrients (N_{tot}, P_{tot}, K_{tot}) were analyzed from 1:5 water extractions according to SFS-EN 13652 (Finnish Standard Association, 2002b). Samples were shaken for 1 h and filtered through a cellulose filter (pore size ~ 8 µm). The concentrations of NH_4-N, NO_3-N and phosphate phosphorus (PO_4-P) were analyzed with a Lachat autoanalyzer. Soluble total N in water extractions was measured with a Lachat autoanalyzer after oxidation of organic N into NO_3-N in an autoclave with peroxodisulfate. Soluble total P and K from water extracts were measured with inductively coupled plasma emission spectrometry (Perkin Elmer Optima 8300, USA).

The measurement of phosphorus availability was based on modified Hedley fractionation (Sharpley & Moyer, 2000, Ylivainio et al., 2008), where the fertilizer product was extracted sequentially with water, 0.5 M $NaHCO_3$, 0.1 M NaOH and 1 M HCl at a ratio of 1:60. First inorganic P was determined from the extract and then total P concentration was measured after digestion with peroxidase in an autoclave as described in Ylivainio et al. (2008). Organic P concentration was calculated as the difference between total and inorganic P.

Samples for heavy metal (Pb, Ni, Cd, As, Cu, Zn and Cr) analyses were first dried in 60 °C and then digested in aqua regia according to SFS ISO 11466 (Finnish Standard Association 2007). Approximately 1.0 g of sample was boiled in 9.35 ml of aqua regia for 2 hours, transferred into a 100 ml volumetric flask and filtered. After digestion Cu, Cr, Zn and Ni were determined with inductively coupled plasma emission spectrometry (Thermo Jarrell Ash IRIS Advantage, Thermo Scientific, USA), and As, Cd, Pb, with graphite furnace atomic absorption spectrometry using a Varian AA280Z (Varian Inc., USA). Hg was measured based on cold vapor atomic absorption spectrometry using Varian M-6000A Mercury Analyzer (Varian Inc., USA).

2.5. Calculations

The organic N (N_{org}) in the digestates was calculated from the difference between TKN and the sum of mineral nitrogen (NH_4-N + NO_3-N). The dissolved organic nitrogen (DON) was calculated as the difference between 1/5 water extractable N_{tot} and the sum of NH_4-N and NO_3-N.

The apparent nitrogen utilization efficiency (NUE) of plants was calculated according to the following equation (Gunnarsson et al., 2010):

$$NUE (\%) = (N_{uptake} – N_{control}) / N_{added} \times 100$$

where N_{uptake} refers to the N uptake per pot (mgN/pot) with each studied digestate, $N_{control}$ to the N uptake per pot of the unfertilized control (mgN/pot) and N_{added} to the amount of added N per pot as tot-N (mgN/pot). The NUE was calculated for both NH_4-N and TKN.

3. Results and discussion

3.1. Digestate quality

3.1.1. Digestate pH, solids and organic matter

The pH, solids and organic material concentrations of the digestates and feedstocks were assessed to evaluate the effect of digestate on soil quality and plant growth (Table 2). All digestates were neutral or slightly alkaline (pH 6.7–8.4), which is typical for food and green waste digestates (reviewed by Teglia et al., 2011). The neutral pH supports the use of digestates in agriculture, while the use of alkaline digestates could increase, e.g., NH_4-N volatilization from soil during spreading depending on the temperature (Nkoa, 2014) and the acidic digestates can decrease soil pH and enhance the heavy metal mobilization in soils (Otabbong et al,. 1997). Subsequently, the effect of digestate pH on soil is dependent on soil characteristics (Alvarenga et al., 2015, Makádi et al., 2012), thus, in a 4-year fertilization study the soil initial pH of 5.4–5.7 was not affected after application of household- and restaurant waste-based urban digestate (Odlare et al., 2008).

The FW and OFMSW feedstocks had rather similar TS (230-290 g/kg) and VS (210–260 g/kg), but these characteristics were not reflected in the digestates (Table 2). The FW digestates (FW1 and FW2) had solid (TS) and organic matter (VS) concentrations over 50–80 g/kgFM, which were higher than in the FW3-, OFMSW- and VWAS-based digestates (10–30 g/kgFM), where the lower TS concentrations were most likely related to internal water additions/recirculation in the biogas plants from which the digestates (FW3, OFMSW, VWAS) originated. The high TS and VS in FW1 and FW2 digestates could also be partly explained by the lower degradation during anaerobic digestion (VS degradation 70–78% in FW1 and FW2, over 90% in FW3 and OFMSW), probably due to the lower hydraulic retention time and higher organic loading rate (47–58 days, 4 kgVS/m^{3}d) in reactors fed with FW1 and FW2 than with FW3 (26 days, 3.3 kgVS/m^{3}d) and OFMSW (24 days, 2.4 kgVS/m^{3}d) feedstocks. Overall, the results support the fact that the digestate TS concentration is dependent on the reactor configuration (e.g. wet/dry process) and process parameters (loading rate, retention time) (Teglia et al., 2011) despite the uniform characteristics of the feedstocks. It is also likely that the actual organic composition of the digestate feedstocks was different, which was not reflected in the TS and VS concentrations.

The studied digestates were considered suitable for agricultural use as the VS concentrations fulfilled the minimum level for organic matter content introduced in the European proposal for digestate quality (15 %TS, Saveyn & Eder, 2014). Digestates also had similar concentration of solids (20–80 gTS/FM) and organic matter (12–64 gVS/FM, Table 2) as has been studied with various digestates in field- and laboratory-scale fertilization experiments, where the plant growth or soil response were considered good (TS 17–120 g/kg, VS 9–66 g/kg) (Abubaker et al., 2012, Alburquerque et al., 2012a, 2012b, Fouda et al., 2013, Rigby & Smith, 2013). As digestate fertilization adds organic matter to soil, the microbiological activity, mineralization and subsequently the availability of nutrients are increased (Galvez et al., 2005, Gutser et al., 2005, Odlare et al., 2008, 2011). Thus, excessive amounts of organic matter can lead to imbalanced microbial function and nitrogen immobilization (Alburquerque et al., 2012a, Gutser et al., 2005) and to phytotoxicity due to organic acids (Abdullahi et al., 2008) i.e. affect digestate stability (defined as the amount of easily degradable organic matter).

The FW3, OFMSW and VWAS digestates were considered stable due to the lower carbon concentration compared to FW1 and FW2 which had 50–80% higher COD, VS and C_{tot} concentrations (Table 2). All three FW digestates were characterized with higher SCOD concentrations (11–19 g/kg) compared to OFMSW and VWAS digestates (7–8.5 g/kg). The VFAs accounted for 28 and 45% of SCOD in FW1 and FW3, 52% in VWAS and the low share of 8% in FW2 and 5% in OFMSW digestates, suggesting that the share was not feedstock dependent. In terms of VFA concentration, only FW2 and OFMSW were considered stable, as the VFA_{tot} was under the limit of 1500 mg/l, which is proposed for digestate fertilizer use within the end-of-waste criteria (Saveyn & Eder, 2014). The limit value for digestate VFAs in agricultural use in the UK (0.43 gCOD/gVS, BSI, 2010) was, however, not exceeded with any of the studied digestates. Although a high concentration of fatty acids can contribute to the phytotoxic effects (Abdullahi et al., 2008), the VFAs are also reported to act as a carbon source for soil micro-organisms and to degrade fast after application to soils (Kirchmann & Lundvall, 1993). The non-VFA-SCOD found in digestates was most likely related to, e.g., undegraded carbohydrates and also for other acids such as humic acids (Scaglia et al., 2015, Zheng et al., 2014), which have been recently proposed to act as bio-stimulants enhancing plant growth (Scaglia et al., 2015). Additionally, humic acids are related to the stability of digestates (Zheng et al., 2014) along with the other stable molecules, lignin and long-chain proteins (Tambone et al., 2009).

3.1.2. Heavy metal content

Digestate heavy metal contents (mg/kgTS) were studied from dried samples and compared with the EU legislative limits for digestate application (Table 3). VWAS digestate had the highest content of heavy metals and was the only one to exceed the limits within European legislation concerning Hg, Cu and Zn. VWAS digestate most likely reflected the heavy metal content of the feedstock mixture, especially the waste-activated sludge, as the vegetable waste usually contains heavy metals in similar contents as FW feedstocks (Table 3). Compared to VWAS, FW and OFMSW digestates had a lower content of heavy metals reflecting the content in the feedstocks. Heavy metal contents between FW and OFMSW digestates were fairly similar in Hg (0.1–0.3 mg/kgTS) and Cr (8–13 mg/kgTS), while OFMSW had a slightly increased content of Pb, Cd, As, Cu, Zn, and low content of Ni (7 mg/kgTS in OFMSW, 16–42 mg/kgTS on FW digestates). Considering the feedstocks, the content of Pb was over tenfold in the autoclaved FW2 feedstock compared to the FW1 feedstock and 1.53 times higher with Cu, Zn and Cr, apparently due to residues from the autoclaving apparatus during the pre-treatment of the food waste, thus, the increases in Cu and Zn were not reflected in FW2 digestate.

The heavy metal contents (mg/kgTS) increased and concentrated from feedstocks to digestates due to the reduction of solids content during digestion. Overall, the contents of heavy metals in the digestates were similar to those reported with different sewage sludge and organic waste digestates (Table 3). However, due to the feedstock characteristics VWAS digestate showed increased heavy metal content exceeding the legislative limit and thereby preventing its use in agriculture as such, as the heavy metals can cause effects in soils and plants. For example, Cu and Zn are reported to bind with organic compounds and immobilize in soils (Otabbong et al., 1997, Zhu et al. 2014), and the fertilization with sewage sludge has been reported to increase the accumulation of Cd, Zn, Pb and Cu in plants (Otabbong et al. 1997), the effects of which are dependent on the chemical properties, such as solubility of metals, and by soil characteristics, such as pH.

The actual amount of heavy metals ending up in the soils depends on the amounts of digestate used. For example, with digestate fertilization at a rate of 170 kgTKN/ha/year the mass of the studied digestates varies from 20 to 80 tons per hectare depending on the TS and nitrogen content. Subsequently, the volume of heavy metals applied to the soil is dependent on the applied digestate amounts. The calculated heavy metal volumes per hectare (g/ha/year, Table 3) showed increased heavy metal loads with VWAS digestate, which, due to low TKN content and TS, requires large application volumes to meet the fertilization goal (170 kgTKN/ha). With FW and OFMSW digestates the heavy metal loads were remarkably lower, and FW digestates showed the least environmental contamination of the studied urban digestates.

3.2. Fertilizer value

3.2.1. Digestate nutrient concentrations

The concentration of nutrients and the solubility of phosphorus were analyzed to evaluate the fertilizer value of the digestates. Overall, FW and OFMSW digestates had higher concentrations of nitrogen and potassium and lower phosphorus concentrations and C/N ratio when compared to the VWAS digestate. FW and OFMSW digestates (except FW2 digestate) had total, mineral and soluble nitrogen concentrations over 3 g/kgFM due to the high initial total nitrogen concentrations in FW and OFMSW feedstocks (around 6–8 g/kgFM, Table 2). In FW1 digestate the NH_4-N/TKN ratio was low (50%) compared to FW3 and OFMSW (71–82%) digestates and was caused by the decreased organic matter degradation, as was observed during the material characterization. FW and OFMSW digestates had the C/N ratios (1.5–3.3) and concentrations of total nitrogen (4.5–8.7 g/kgFM) and potassium (2–3 g/kgFM) typical for these types of digestates and similar to a mixture of 80% OFMSW +20% pig slurry (Gutser et al., 2005, Tambone et al., 2010). However, phosphorus concentrations in FW and OFMSW digestates were low (0.1–0.3 g/kgFM) compared to 0.8–1.1 g/kgFM in the OFMSW + pig slurry digestate in Tambone et al. (2010). The pretreated FW2 digestate showed remarkably low NH_4-N and soluble total nitrogen concentration (<3 g/kgFM) and NH_4-N/TKN ratio (20%) caused by the autoclaving treatment which has been shown to decrease protein degradation during anaerobic digestion (Tampio et al., 2014).

VWAS digestate had low TKN and NH_4-N (around 2 g/kgFM, Table 2) due to the low nitrogen concentration in the feedstock mixture, as both vegetable waste and waste-activated sludge have low total nitrogen concentrations (1.5 gTKN/kgFM in Shen et al., 2013 and 1.7 gTKN/kgFM in Cavinato et al., 2013, respectively). The TKN, C/N ratio (around 6) and low potassium concentrations (0.6 g/kgFM) in VWAS digestate were comparable with municipal (Tambone et al., 2011) and industrial wastewater treatment sludge digestates (Alburquerque et al., 2012a). VWAS digestate had the soluble phosphorous content of 0.35 g/kgFM, where the phosphate phosphorus (PO_4-P) accounted for 100% of the P_{tot} in 1/5 water extractions indicating good plant availability of P (Teglia et al., 2011) and was most likely due to the high P content of the waste-activated sludge, as reported by Odlare et al. (2008) and Zirkler et al. (2014).

FW and OFMSW digestates were considered to have the highest fertilizer value compared to VWAS digestate as the nitrogen availability in the soil after spreading is dependent on the plant available NH_4-N concentration and the NH_4-N/TKN ratio (Fouda et al., 2013, Teglia et al., 2011). The high fertilizer value was also supported by the ratio between C and organic N (C/N_{org}), which was 8, indicating high N release in soils (Gutser et al., 2005). The VWAS digestate had a C/N_{org} ratio of 29 suggesting a lower N release.

The availability of phosphorus for plant growth is dependent on the solubility which was analyzed with Hedley fractionation, where 50–70% of the P in FW and VWAS digestates was considered as plant available (water and $NaHCO_3$ extractable, Figure 2). OFMSW digestate showed a lower P solubility of 30% indicating a difference in the digestate composition compared to FW digestates, which was however not detected in any other characterization analysis. The P fractionation of OFMSW and waste water sludge-based digestates were also studied by García-Albacete et al. (2012), where the $NaHCO_3$ extractable Olsen-P was similar (0.1–0.4%) as in studied digestates (0.04–0.2%). Because not all of the total P in digestates is considered to be plant available, the solubility of P should be measured to avoid the overestimation of P availability from the digestates. For example, previous life cycle analyses have overestimated the P substitution by assuming that 100% of mineral fertilizer P is able to be substituted with digestates (Boldrin et al., 2011, Bernstad & la Cour Jansen 2011). Thus, in some studies the more accurate P substitution rate of 50% is applied (Evangelisti et al., 2014).

As the FW and OFMSW were characterized as being rich in N and poor in P, and the VWAS digestate had a relatively low concentration of both nutrients, reduced fertilizer value and the need for additional mineral fertilizer supplements can be expected due to uneven and potentially deficient N and P ratios (Svensson et al., 2004). The low NH_4-N in VWAS and FW2 digestates also supported their use as soil amendments rather than as source of nutrients (Teglia et al., 2011).

3.2.2. Nitrogen mineralization in soil

The transformation of digestate organic nitrogen into mineral forms in soil was studied via mineralization experiments (Table 4, Figure 3) with different digestate nitrogen application rates from 171 to 318 mgTKN/kg soil. Application of dissolved organic N (DON) of 1:5 water extractions was 27–64 mg/kg and this proportion of organic N can be considered most easily mineralized. In the beginning of the mineralization experiment the soil NO_3-N concentration was low and the predominant form of soil inorganic nitrogen was NH_4-N from the digestates. Nitrification of NH_4-N to NO_3-N happened at a fast rate in all digestate applications after a 4-day adaptation/immobilization period. After 48 days the mineralization of organic N was of the same magnitude (around 30 mgN/kg) as all other digestates except the FW3 digestate (2 mgN/kg, Table 4, Figure 3).

Considering the low N mineralization with the FW3 digestate, the digestate responded to its readily mineralized N concentration, while the organic N application was 25–60% lower than with other digestates. Other studied digestates had lower initial NH_4-N concentrations and 15–30% of their organic N mineralized during the incubation. FW3 digestate did not show notable differences in ryegrass growth experiments, indicating that the increase of mineralized N in soil was not vital for plant growth (Gunnarsson et al., 2010), when the initial NH_4-N concentration was high. In addition, the low mineralization can be attributed to the availability of organic nitrogen (Abubaker et al., 2015, Rigby & Smith, 2013), which was low due to the variation in the digestate application volumes.

The net N mineralization started soon after a short adaptation/immobilization period due to the easily degradable material, and no further nitrogen immobilization was detected which is reported to lead to a good growth response (Gutser et al., 2005). The low initial NH_4-N in FW2 digestate was due to the feedstock pretreatment where the nitrogen-containing molecules have been previously reported to transform into recalcitrant and hardly degradable Maillard compounds (Tampio et al., 2014), and therefore, low mineralization and growth responses were anticipated. However, the N mineralization with FW2 digestate was on the same level as in the other studied digestates indicating that the soil microbes were still, to some extent, able to transform the rather recalcitrant nitrogen. With VWAS digestate the observed high C/N_{org} ratio and the low NUE during the growth experiment indicated low N release and availability which were reflected by 50% decreased mineralization of N_{org} compared to the other studied digestates in the mineralization test. This difference was connected with the composition of the waste-activated sludge feedstock which led to a low nitrogen concentration in the VWAS digestate.

3.2.3. Ryegrass growth and nitrogen uptake

The plant growth and nitrogen uptake in pot experiments were studied with Italian ryegrass (cv. Fabio) in order to compare the nitrogen fertilizer value of the digestates (Table 5, Figure 4). Depending on the applied nitrogen amount, digestate applications produced ryegrass yields of 38–60 gDW/pot, which were 5–30% higher than the control with similar inorganic N concentration. FW1 and FW2 digestates had 20- 30% higher yields compared to the control and high NH_4-N utilization efficiencies ($NUE_{NH4\text{-}N}$) >90% were observed because soluble nitrogen was fully used for plant growth. However, with FW3, OFMSW and VWAS digestates the increase in the ryegrass yield was more moderate (5–10%) compared to the control, and NUEs were between 74 and 82% indicating that the soluble N was not fully available for plant growth. During the growth experiment 30–50% of the TKN was utilized by the ryegrass from all studied digestates.

The improved ryegrass growth response was compared to the mineral fertilizer control, which indicated that the nutrient composition, especially nitrogen availability, was sufficient for plant growth in the studied digestates. The ammonium nitrogen level of the digestate applications was comparable to ammonium nitrate level of the controls, and part of DON was also mineralized and increased ryegrass growth. The result is supported by previous studies, where the FW- and OFMSW-based digestates have been reported to increase the crop biomass yield compared to digestates originating from other feedstocks (Abubaker et al., 2012, Haraldsen et al., 2011, Svensson et al., 2004) and increased or similar yields as mineral fertilizers (Haraldsen et al., 2011, Walsh et al., 2012). In comparison, in a long-term (4 years) field-scale fertilization study, digestates produced 88% of the yield of mineral fertilizers (Odlare et al., 2011), and equal yields to mineral fertilizers were achieved when digestates were supplemented with mineral fertilizers (Odlare et al. 2008).

During the growth experiment the NUE_{NH4-N}, calculated from the applied NH_4-N, showed high values (>75%, Table 5) for all digestates indicating that the ryegrass was able to use the mineral N of the digestates, as previously reported (NUE 90–95%, Gunnarsson et al., 2010, Grigatti et al., 2011). The NUE_{TKN} values, calculated according to the applied TKN, were between 40–50% with FW- and OFMSW-based digestates (except FW2) and around 33% with VWAS and FW2 digestates. Considerably higher NUE_{TKN} values (44–85%) have been previously reported with pig slurry (Grigatti et al., 2011) and a mixture of pig slurry and agro-industrial wastes (Gunnarsson et al., 2010, Alburquerque et al., 2012a), while the average NUE_{TKN} for mineral fertilizers was around 60% (Gutser et al. 2005), as also shown in the present study. The relatively low NUEs found in this study (33%) with FW2 and VWAS digestates indicated that the TKN still consisted of recalcitrant N, which was not plant available and fully mineralizable (Gunnarsson et al., 2010). These results were supported by previous findings with FW2 feedstock, where the feedstock pretreatment transformed nitrogen into a recalcitrant form, reflected in the low NH_4-N concentration and reduced soil mineralization capacity. However, with VWAS the characteristics of waste-activated sludge most likely affected the digestate TKN composition, its uptake efficiency and high C/N_{org} ratio lowering N release. Thus, VWAS digestate produced similar growth response as FW and OFMSW digestates, and no effect of the uneven N and P concentrations between digestates (see chapter 3.2.1) were observed on ryegrass growth in the short-term experiment.

4. Conclusions

Overall, the studied urban digestates originating from FW, OFMSW and VWAS had potentially favorable agronomic characteristics and produced 5–30% higher ryegrass yields compared to the control mineral fertilizer with a similar inorganic nitrogen concentration, while the feedstock source played a major role in material characterization. FW and OFMSW digestates (except FW2) reflected their feedstock composition and showed rather similar nutrient concentrations, soil N mineralization, ryegrass growth and heavy metal content and were, as follows, characterized with high agronomic value. The VWAS digestate showed decreased nitrogen availability due to lower nitrogen concentration of the feedstock which led to decreased fertilizer value. In addition, VWAS digestate increased the risk for soil contamination due to high content of heavy metals, which also exceeded the limits within European legislation and thus, prevents its use in agriculture as such. However, the temperature and pressure pretreatment of the FW2 feedstock reduced the digestate nitrogen availability and promoted its use as a soil amendment rather than a fertilizer.

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Table 1. Origin and background information of the studied feedstocks and digestates. FW=Food waste, OFMSW=organic fraction of municipal solid waste, VWAS= mixture of vegetable waste and waste-activated sludge, HRT=hydraulic retention time, OLR=organic loading rate.

Feedstock/Digestate	Scale	Temperature	Phase	HRT (d)	OLR
FW1	Laboratory	Mesophilic	1	58	4.0[b]
FW2[a]	Laboratory	Mesophilic	1	47	4.0[b]
FW3	Sub-commercial	Mesophilic	1	26	3.3[b]
OFMSW	Full scale	Thermophilic	2	24	3.7[c]
VWAS	Pilot	Thermophilic	1	16	3.8[c]

[a]Feedstock pretreated with autoclave (160 °C, 6.2 bar)

[b]kgVS/m^3day

[c]kgCOD/m^3day

Table 2. Feedstock and digestate characteristics.

Material	*Feedstocks*				*Digestates*				
Sample	FW1	FW2	FW3	OFMSW	FW1	FW2	FW3	OFMSW	VWAS
pH, solids and organic matter									
pH	5.5	5.4	5.0	4.7	8.0	7.6	8.3	8.3	7.6
TS (g/kgFM)	247.0	226.4	255.1	287	68.1	78.8	19.9	32.2	34.2
VS (g/kgFM)	229.9	209	232.8	264.3	50.2	63.7	12.3	18.9	23.9
VS/TS (%)	93.1	92.3	91.3	92.1	73.6	80.9	61.7	58.7	69.9
SCOD (g/kgFM)	114.6	104.2	132.9	69.9	15.4	18.5	11.2	7.3	8.4
COD (g/kgFM)	364.4	361.2	444	412.5	77.1	100.3	21.8	30.6	26.7
SCOD/COD (%)	31.4	28.8	29.9	17.0	20.0	18.4	51.4	23.9	31.5
VFA_{tot} (g/kgFM)	3.1	2.2	4.9	5.5	3.3	1.1	4.1	0.3	3.4
VFA_{tot} (gCOD/kgFM)	3.5	2.3	5.4	5.9	4.3	1.5	5.0	0.4	4.4
Nutrients									
C_{tot} (g/kgFM)	N/A	N/A	N/A	N/A	26.9	25.9	6.8	10.3	13.5
C/N	N/A	N/A	N/A	N/A	3.1	3.3	1.5	2.3	6.1
TKN (g/kgFM)	7.8	7.3	8.2	5.7	8.7	7.8	4.7	4.5	2.2
NH_4-N (g/kgFM)	0.5	0.4	0.6	0.3	4.5	1.7	3.9	3.2	1.7
NH_4-N/TKN (%)	6.7	5.0	7.2	5.4	52.0	21.3	82.1	71.1	78.6
1:5 water soluble nutrients									
N_{tot} (g/kgFM)	N/A	N/A	N/A	N/A	6.0	3.0	4.4	4.0	2.2
NH_4-N (g/kgFM)	N/A	N/A	N/A	N/A	4.4	1.9	3.3	2.8	1.6
NO_3-N (g/kgFM)	N/A	N/A	N/A	N/A	0.013	0.011	0.011	0.007	0.003
PO_4-P (g/kgFM)	N/A	N/A	N/A	N/A	0.27	0.14	0.06	0.13	0.35
P_{tot} (g/kgFM)	N/A	N/A	N/A	N/A	0.33	0.19	0.11	0.15	0.35
K_{tot} (g/kgFM)	N/A	N/A	N/A	N/A	3.2	2.5	1.9	1.9	0.6

N/A, not available

Table 3. Heavy metals in the studied digestates and their feedstocks, regulatory framework concerning heavy metal limits in European countries, literature data and heavy metal load after digestate application.

Heavy metals	Pb	Ni	Hg	Cd	As	Cu	Cr	Zn
Feedstocks (mg/kgTS)								
FW1	0.2	0.6	0.06	0.06	0.5	4.9	1.1	28.2
FW2	2.2	0.5	0.08	0.05	0.5	8.4	3.3	37.8
FW3	0.7	1	0.08	0.06	0.4	5.7	1.8	29.4
OFMSW	0.5	0.8	0.05	0.02	0.2	9.6	1.3	93.3
Digestates (mg/kgTS)								
FW1	2.1	17.8	0.1	0.2	0.7	25.6	9.8	116
FW2	5.6	16.6	0.2	0.1	0.4	22.4	11.9	94.6
FW3	5.6	42.4	0.1	0.3	1	21.7	7.5	175
OFMSW	11.7	6.7	0.3	1.5	3.3	58.7	13	401
VWAS	98	22.3	1.8	1.1	2.6	626.5	32.9	1006
Regulatory limit values for digestate use (mg/kgTS)								
Uk[a]	200	50	1	1.5	-	200	100	400
Finland[b]	100	100	1	1.5	25	600	300	1500
EU proposal[c]	120	50	1	1.5	-	200	100	600
Feedstock in the literature (mg/kgTS)								
Vegetable waste[d]	<1–22	<1–10	N/A	<0.5–1	N/A	<1–18	1–7	3–97
Sewage sludge[e]	40–144	N/A	N/A	6–32	N/A	700–1570	N/A	321–487
Digestate in the literature (mg/kgTS)								
Sewage sludge[f]	4–30	13–37	N/A	0.3–1.7	N/A	50–1000	N/A	200–1300
Biowaste, green waste, industrial waste[g]	5–282	5–41	N/A	0–0.46	N/A	21–161	7.4–54	60–340
Household waste[h]	4.1–6.1	5.5–7.9	0.05–0.13	0.4–0.6	N/A	44–67	6.7–15.4	227–381
Heavy metal load after digestate spreading (g/ha/year)[i]								
FW1	2.8	23.8	0.2	0.3	0.9	34.3	155.1	13.1
FW2	9.6	28.6	0.3	0.2	0.8	38.4	162.5	20.4
FW3	4.1	30.6	0.1	0.2	0.7	15.6	126.2	5.4
OFMSW	14.2	8.2	0.4	1.8	4.0	71.6	488.9	15.8
VWAS	259.1	58.8	4.8	2.9	6.8	1655.7	2658.6	86.9

[a]BSI, 2010, [b]Decree of the Ministry of Agriculture and Forestry No 24/11 on Fertiliser Products, [c]Saveyn & Eder, 2014, [d]Bożym et al., 2015, [e]Otabbong et al., 1997, [f]Zirkler et al., 2014, [g]Kupper et al., 2014, [h]Govasmark et al., 2011, [i]Digestate spreading calculated according to TKN rate of 170kgTKN/ha

N/A, not available

Table 4. Applied nitrogen and mineralization of nitrogen after 48 days incubation.

Digestate	FW1	FW2	FW3	OFMSW	VWAS
Application (g/100g)					
FM	2.2	2.6	4.8	5.1	8.6
Applied (mg/kg)					
TKN	205	171	235	244	318
N_{org}	108	121	77	102	181
DON	36	27	53	64	54
NH_4-N	97	50	158	142	137
NO_3-N	0	0	1	0	0
Mineralization from applied organic N					
mg/kg	36	34	2	29	26
% of DON	100	125	3	45	47
% of N_{org}	33	28	2	28	14

Table 5. Ryegrass yields and N uptake during pot experiments with the studied digestates and control. Ryegrass yield after 3rd harvest and nitrogen uptake and nitrogen uptake efficiency (NUE) after 2nd harvest. NUEs calculated with NH_4-N and TKN.

	Applied (mg/pot)			Yield	N uptake	$NUE_{NH4\text{-}N}$	NUE_{TKN}
Treatment	TKN	$N_{soluble}$	NH_4-N	(gDM/pot)	(mgN/pot)	(%)	(%)
Controls							
N0	0	0	-	18.9 ± 0.6	243.9 ± 9.3	-	
N500	500	500	-	31.8 ± 2.8	582.9± 16.9	68	
N1000	1000	1000	-	50.8 ± 4.6	858.1 ± 31.5	61	
N1500	1500	1500	-	63.1 ± 4.2	1138.1 ± 33.3	60	
N2000	2000	2000	-	77.6 ± 5.2	1440.2 ± 63.6	60	
Digestates							
FW1	1540.8	997.6	727.1	50.5 ± 1.3	895.0 ± 3.5	90	42
FW2	1284.3	580.8	376.2	38.4 ± 3.1	663.8 ± 14.7	112	33
FW3	1763.6	1584.7	1188.4	58.3 ± 5.6	1123.9 ± 67.1	74	50
OFMSW	1832.6	1546.9	1069.7	59.1 ± 4.3	1116. ± 42.2	82	48
VWAS	2390.0	1441.1	1032.9	56.8 ± 2.9	1014.8 ± 13.8	75	32

Figure Captions

Figure 1. The analyzed agronomic characteristics of the studied digestates and feedstocks.

Figure 2. Solubility of phosphorus determined with Hedley fractionation.

Figure 3. Nitrogen mineralization during 48-day incubation tests. Digestates FW1 (a), FW2 (b), FW3 (c), OFMSW (d), VWAS (e).

Figure 4. Ryegrass yield and nitrogen uptake of digestates compared to control treatments. The dotted line represents the control treatments and error bars the standard deviation within control samples.

Fig. 1.

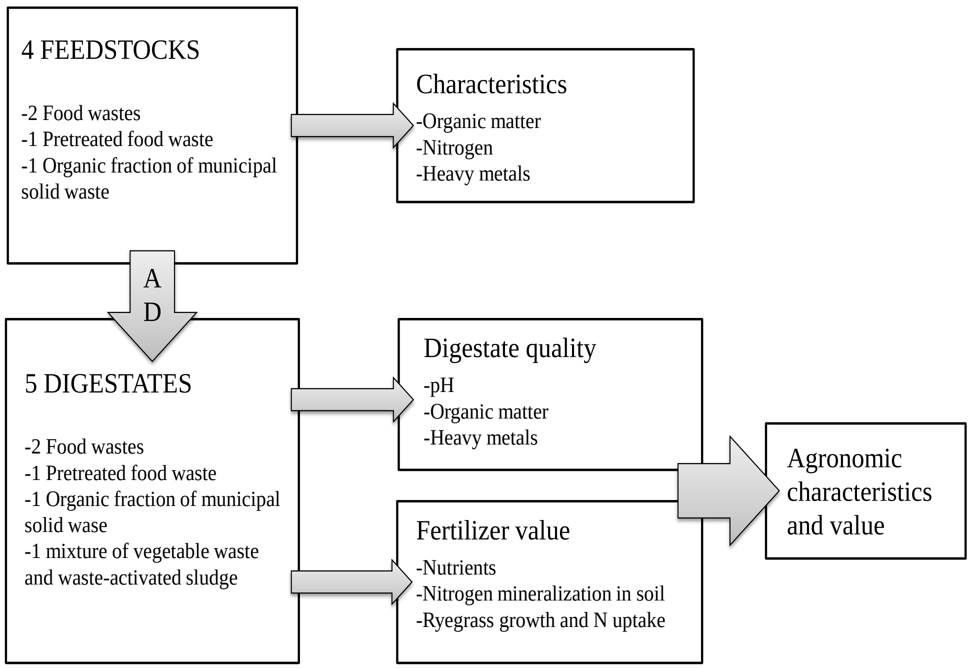
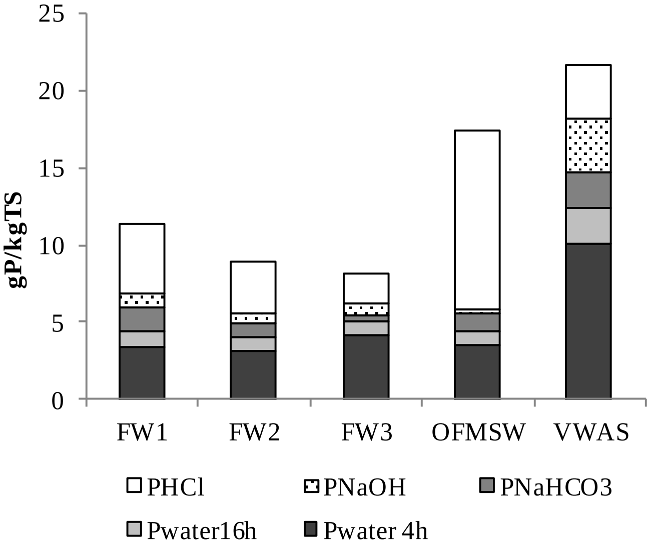
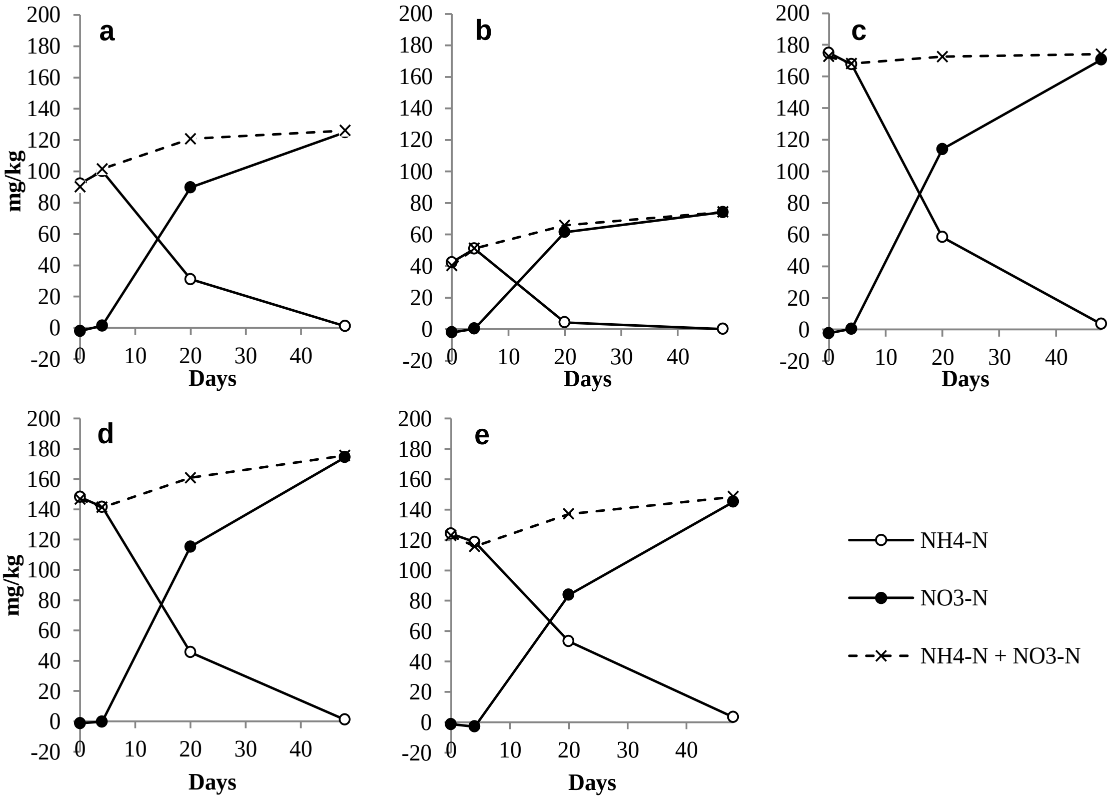

4 FEEDSTOCKS
-2 Food wastes
-1 Pretreated food waste
-1 Organic fraction of municipal solid waste
Characteristics
-Organic matter
-Nitrogen
-Heavy metals
A
D
5 DIGESTATES
-2 Food wastes
-1 Pretreated food waste
-1 Organic fraction of municipal solid wase
-1 mixture of vegetable waste and waste-activated sludge
Digestate quality
-pH
-Organic matter
-Heavy metals
Fertilizer value
-Nutrients
-Nitrogen mineralization in soil
-Ryegrass growth and N uptake
Agronomic characteristics and value

Fig. 2.



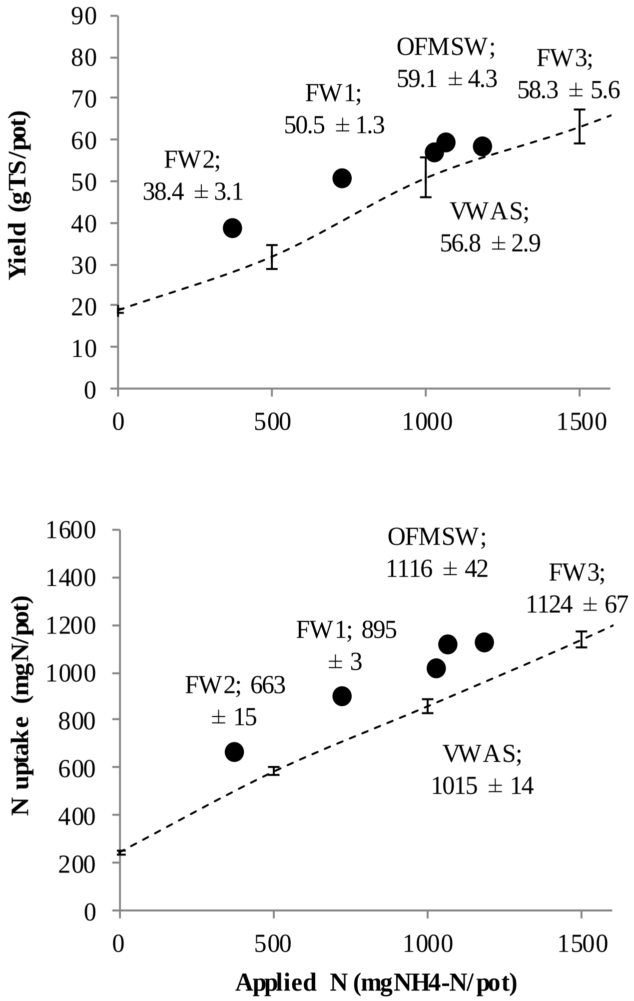
25
20
15
10
5
0
gP/kgTS
FW1
FW2
FW3
OFMSW
VWAS
PHCl
PNaOH
PNaHCO3
Pwater16h
Pwater 4h

Fig. 3.



a
b
c
d
e
mg/kg
Days
200
180
160
140
120
100
80
60
40
20
0
-20
0
10
20
30
40
NH4-N
NO3-N
NH4-N + NO3-N

Fig. 4.



90
80
70
60
50
40
30
20
10
0
Yield (gTS/pot)
FW2;
38.4 ± 3.1
FW1;
50.5 ± 1.3
OFMSW;
59.1 ± 4.3
FW3;
58.3 ± 5.6
VWAS;
56.8 ± 2.9
0
500
1000
1500
1600
1400
1200
1000
800
600
400
200
0
N uptake (mgN/pot)
FW2; 663
± 15
FW1; 895
± 3
OFMSW;
1116 ± 42
FW3;
1124 ± 67
VWAS;
1015 ± 14
0
500
1000
1500
Applied N (mgNH4-N/pot)