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Utilization of Food Waste via Anaerobic Digestion

From Feedstock to Biogas and Fertilizers

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Abstract

Food waste is a renewable resource that can be utilized as both energy and nutrients through anaerobic digestion to increase nutrient recycling and fertilizer self-sufficiency and promote the mitigation of greenhouse gas emissions. Anaerobic digestion of food wastes has, however, faced challenges due to the waste's characteristics, e.g., high protein content, which is why the organic loading rates with food waste digestion are usually kept low to achieve a stable process. The digestate produced during digestion contains all of the nutrients from the food waste feedstock and can be used as a fertilizer in agriculture, where the availability of nutrients, the stability of organic matter, and biosecurity define its agronomic value. In this thesis, the aim was to analyze the potential of using anaerobic digestion for food waste utilization. The anaerobic digestion of food waste, feedstock pretreatment, and processing and utilization of the digestate for fertilizer use were studied.

This study shows the potential of food waste as feedstock for anaerobic digestion without dilution, with a total solids content of 20–25%. A high organic loading rate of 6 kgVS/m^3d (VS, volatile solids) was achieved with methane yields 400–430 m^3/kgVS in continuous food waste digestion while the optimum loading rate was 3 kgVS/m^3d, yielding around 480 m^3/kgVS of methane. Trace element supplementation enabled a stable long-term operation and gradual increase of loading rates without the accumulation of acids. The autoclave pretreatment (160°C and 6.2 bars) of the food waste affected the characteristics – and subsequently, the anaerobic digestion performance, where the formation of protein-based hardly biodegradable compounds led to a 10% lower methane yield during digestion, decreased hydrogen sulfide content in the biogas, and 50% decreased ammonium nitrogen concentration within the digestate. The decreased availability of proteins and hydrogen sulfide formation due to the pretreatment reduce the risk of ammonia inhibition during anaerobic digestion and enable easier biogas cleaning and security.

The food waste digestates shows potential as a nutrient source in crop fertilization independently and after post-treatment. The studied digestates were considered suitable for fertilizer use, as they showed good agronomic value in terms of nutrient content and usability, as well as biosecurity. Food waste digestates produced around 5 to 30% higher ryegrass yield compared with a mineral fertilizer in pot experiments, and the majority (50–70%) of the nitrogen and phosphorus were in the soluble and plant-available forms. The integration of anaerobic digestion and digestate post-treatment technologies enabled the processing of the digestate liquid into concentrated nutrient products rich in nitrogen and potassium. With the combination different processing technologies such as evaporation, stripping, and reverse osmosis, nutrient products with optimal composition can be produced to correspond with the fertilizer demand. Overall, due to the high energy potential of the food waste, the integration of the

anaerobic digestion with heat-demanding digestate liquid post-treatment processes (e.g., stripping and/or evaporation) was possible.

In conclusion, anaerobic digestion has high potential for the utilization of food waste, as food waste produces high methane yields in optimized conditions. The food waste digestate was also shown to be a suitable nutrient (especially nitrogen) source in crop fertilization independently and after post-treatment.

Tiivistelmä

Ruokajätteet ovat uusiutuva resurssi, joita voidaan hyödyntää biokaasuprosessissa tuottaen sekä energiaa että ravinteita, lisätä ravinteiden kierrätystä ja omavaraisuutta sekä vähentää kasvihuonekaasupäästöjä. Ruokajätteen käyttö biokaasuprosessin raaka-aineena voi kuitenkin olla haasteellista korkean proteiinipitoisuuden vuoksi, mikä on vaikuttanut siihen, että orgaanisen aineksen kuormitus pidetään reaktoreissa usein melko matalana stabiilin prosessin saavuttamiseksi. Biokaasuprosessissa syntyvä käsittelyjäännös sisältää kaikki ruokajätteen sisältämät ravinteet, jotka voidaan hyödyntää maataloudessa lannoitteena, jossa sekä ravinteiden saatavuus, orgaanisen aineksen stabiilisuus sekä turvallisuus määrittelevät käsittelyjäännöksen lannoitearvon. Tässä väitöstutkimuksessa tavoitteena oli analysoida biokaasuprosessin potentiaalia ruokajätteen käsittelyssä. Työssä tutkittiin sekä ruokajätteen biokaasuprosessia, syötemateriaalin esikäsittelyä sekä muodostuvan käsittelyjäännöksen prosessointia ja käsittelyä lannoitteeksi maatalouteen.

Tuloksena tässä tutkimuksessa saatiin näyttöä ruokajätteen potentiaalista biokaasuprosessin raaka-aineena sellaisenaan, ilman laimennusta, kun jätteen kuiva-ainepitoisuus oli 20–25 %. Laimentamattomalla ruokajätteellä oli mahdollista saavuutta korkea kuormitus (6 kgVS/m^3d) ja metaanisaanto (400–430 m^3/kgVS) jatkuvatoimisessa biokaasuprosessissa, jossa optimikuormitus oli 3 kgVS/m^3d metaanisaannolla 480 kgVS/m^3d. Hivenaineiden lisäys prosessiin mahdollisti pitkäaikaisen stabiilin prosessin, sekä asteittaisen kuormituksen noston ilman happojen kertymistä prosessiin. Esikäsittelynä ruokajätteen autoklavointi (160 °C ja 6.2 bar) vaikutti jätteen koostumukseen ja sitä kautta myös biokaasuprosessiin, jossa heikosti biohajoavien proteiinipohjaisten yhdisteiden muodostuminen johti 10 % alhaisempaan metaanisaantoon biokaasuprosessissa, alentuneeseen rikkivedyn määrään biokaasussa sekä 50 % alhaisempaan ammoniumtyppikonsentraatioon käsittelyjäännöksessä. Esikäsittelyn aikaansaama alentunut proteiinien saatavuus biokaasuprosessin hajottajamikrobeille sekä alentunut rikkivetypitoisuus biokaasussa vähentävät riskiä ammoniumtypestä aiheutuvalle inhibitiolle ja mahdollistavat sekä helpomman että turvallisemman kaasunpuhdistuksen ja -käsittelyn.

Ruokajäteperäiset käsittelyjäännökset osoittivat potentiaalia ravinteiden lähteenä viljelyskasvien lannoituksessa sekä sellaisenaan että jatkokäsittelyprosessien jälkeen. Tutkitut käsittelyjäännökset soveltuivat lannoitekäyttöön niiden hyvän lannoitearvon vuoksi, mikä perustui jäännösten ravinnepitoisuuksiin, ravinteiden käyttökelpoisuuteen sekä tuotteiden turvallisuuteen. Ruokajäteperäiset käsittelyjäännökset tuottivat mineraalilannoitetta 5–30 % korkeamman nurmisadon astiakokeissa, ja suurin osa (50 – 80 %) jäännösten sisältämästä typestä ja fosforista oli liukoisessa ja kasveille käyttökelpoisessa muodossa. Biokaasuprosessin ja käsittelyjäännöksen nestejakeen jatkokäsittelyteknologioiden integrointi mahdollisti nestejakeen prosessoinnin konsentroiduiksi,

runsaasti typpeä ja kaliumia sisältäviksi, ravinnetuotteiksi. Erilaisten käsittelyteknologioiden, esimerkiksi haihdutuksen, strippauksen ja käänteisosmoosin, yhdistelmillä voidaan tuottaa optimaalisen koostumuksen omaavia ravinnetuotteita vastaamaan lannoitteiden tarvetta. Yleisesti ottaen ruokajätteen korkea energiapotentiaalin vuoksi biokaasulaitoksen ja käsittelyjäännöksen nestejakeen jatkokäsittelyprosessien yhdistäminen on mahdollista myös silloin, kun kyseessä ovat paljon lämpöä kuluttavat käsittelyprosessit, kuten strippaus ja haihdutus.

Johtopäätöksenä voidaan todeta, että biokaasuprosessilla on merkittävä potentiaali ruokajätteen käsittelyprosessina, koska ruokajäte optimoiduissa olosuhteissa tuottaa korkean metaanisaannon. Ruokajäteperäinen käsittelyjäännös soveltuu ravinteiden, etenkin typen, lähteeksi viljelyskasvien lannoitukseen sekä sellaisenaan että jäännöksen jatkokäsittelyn jälkeen.

Preface

The experimental work for this thesis was mainly carried out at the Natural Resources Institute Finland (Luke, previously MTT Agrifood Research Finland) within the EU FP7 funded VALORGAS project during the years 2011–2013. The last part of this thesis was written with funding from the Fortum Foundation, and it was mainly completed at the Department of Chemistry and Bioengineering at the Tampere University of Technology (TUT). I wish to thank Luke/MTT, TUT, the Fortum Foundation, and Maa- ja vesitekniikan tuki ry, for the funding and support in my studies and travels for international conferences. I wish to thank my past and present team members, co-workers and superiors, especially Ilkka Sipilä, Teija Paavola and Saija Rasi, at the Luke/MTT for making my studies and this thesis possible.

I am grateful to my supervisor, Prof. Jukka Rintala, for his guidance and active support during the years, and for introducing me to the world of anaerobic digestion. I want to thank all my co-authors – Satu Ervasti, Teija Paavola, Sonia Heaven, Charles Banks, Tapio Salo, and Sanna Marttinen – for their valuable collaboration, comments, and advice during the projects and preparation of the manuscripts. Additionally, Dr. Hélène Carrére and Prof. Fabrizio Adani are acknowledged for the pre-examination of this thesis. The TUT-group – Sanna, Viljami, Tiina, Marja, Susanna, Maarit and Outi – also deserve my thanks for the peer support with my manuscripts. I am very grateful to the Luke/MTT laboratory personnel, especially the Animale-building lab in Jokioinen, for their excellent analysis services and help with all kinds of practical issues in the lab. My sincerest thanks goes also to Teemu Ala-Kleme and Johanna Nikama for their help with the reactor experiments, to Mirva Ceder for the hygiene analyses, and also to Lucia Blasco and Minna Kahala for teaching me about microbiology research.

For the endless hours in the lab, scientific (and not-so-scientific) conversations, and precious carpooling help, I wish to thank Satu Ervasti. I probably still owe you some gas money for the trips from various places to Jokioinen. For all the most memorable trips and events, as well as coffee/lunch sessions, I want to thank the "youngsters" from Luke/MTT: Satu, Petra Tuunainen, Erja Koivunen, Laura Ventto, Heidi Leskinen, Piia Kairenius, Mikko Räsänen and Mari Kukkola. Without you, the years in Jokioinen would have felt far too long.

I want to thank my family for their support and trust during these years of work and study. Also, my friends Päivi, Suvi, and Niina deserve my thanks; you provided the most needed refreshing non-academic moments. Last but not the least, I want to express my gratitude to Eero; my language- and IT-support, co-PhD student, and love.

Tampere, July 2016 Elina Tampio

Contents

Abbreviations

a	Annually, yearly
AD	Anaerobic digestion
ADF	Acid detergent fiber
BMP	Biochemical methane potential
C, C_{tot}, C_{org}	Carbon, total carbon, organic carbon
CH_4	Methane
CHP	Combined heat and power
CO_2	Carbon dioxide
COD	Chemical oxygen demand
DM	Dry matter
FAN	Free ammonia nitrogen
FM	Fresh matter
FW	Food waste
H_2	Hydrogen gas
H_2S	Hydrogen sulfide
H_2SO_4	Sulfuric acid
HCO_3^-	Bicarbonate ion
HRT	Hydraulic retention time
K, K_{tot}	Potassium, total potassium
KW	Kitchen waste
kWh	Kilowatt hour
MSW	Municipal solid waste
MWh	Megawatt hour
N, N_{tot}, N_{org}, N_2	Nitrogen, total nitrogen, organic nitrogen, nitrogen gas
NaOH	Natrium hydroxide
NDF	Neutral detergent fiber
NH_3	Ammonia
NH_4-N, NH_4^+	Ammonium nitrogen, ammonium ion
$(NH_4)_2SO_4$	Ammonium sulfate
NO_3-N	Nitrate nitrogen

NUE	Nitrogen utilization efficiency
OFMSW	Organic fraction of municipal solid waste
OLR	Organic loading rate
P, P_{tot}	Phosphorus, total phosphorus
PO_4^{3-}, PO_4-P	Phosphate, phosphate phosphorus
RMP	Residual methane potential
RO	Reverse osmosis
S	Sulphur
SCOD	Soluble chemical oxygen demand
SO_4^{2-}	Sulfate
STR	Stirred tank reactor
TAN	Total ammonia nitrogen
TE	Trace element
TKN	Total Kjeldahl nitrogen
TS	Total solids
TVFAs	Total volatile fatty acids
VFA	Volatile fatty acid
VS	Volatile solids
VW	Vegetable waste
WAS	Waste activated sludge

List of Publications

This thesis is based on the following original papers, which will be referred to in the text by their Roman numerals I–IV.

I. Tampio, E., Ervasti, S., Paavola, T., Heaven, S., Banks, C., Rintala, J. 2014. Anaerobic digestion of autoclaved and untreated food waste. Waste Management 34, 370–377.

II. Tampio, E., Ervasti, S., Rintala, J. 2015. Characteristics and agronomic usability of digestates from laboratory digesters treating food waste and autoclaved food waste. Journal of Cleaner Production 94, 86–92.

III. Tampio, E., Salo, T., Rintala, J. 2016. Agronomic characteristics of five different urban waste digestates. Journal of Environmental Management 169, 293–302.

IV. Tampio, E., Marttinen, S., Rintala, J. 2016. Liquid fertilizer products from anaerobic digestion of food waste: mass, nutrient and energy balance of four digestate liquid treatment systems. Journal of Cleaner Production 125, 22–32.

Author's contribution

The author planned all the experiments together with co-authors. The author analyzed the results and wrote the first drafts of all of the publications and finalized them with co-authors. In papers I–II, the author conducted the experimental work together with co-author S. Ervasti. In paper III, the author analyzed the results from growth experiments and nitrogen mineralization together with co-author T. Salo. The calculations and data collection in paper IV were solely executed by the author.

1 Introduction

The world's population is ever increasing, and it has been estimated that the global population will exceed 9–11 billion by the end of this century (UNEP, 2015). The growing population increases the need for food and subsequently, energy and fertilizers for food production (Gustavsson et al., 2011). Energy is still mainly produced from fossil fuels, which account for 81% of the primary energy consumed globally (IEA, 2015). In total, the fertilizer industry consumes 1.2% of the world's energy, and the majority of that is used in the production of ammonia (NH_3) through Haber-Bosch synthesis (Swaminathan and Sukalac, 2004). Compared with nitrogen (N), the manufacturing of phosphorus (P) consumes less energy, but its availability in mineral deposits has been estimated to decrease over time, which will increase the P prize and open possibilities for recycled fertilizers (reviewed in Weikard, 2016). Alternative and more sustainable sources for industrial fertilizers are biomasses, e.g., food waste (FW), into which the atmospheric N and mineral P are concentrated. Biomasses provide a more economical option for the world's growing fertilizer need (Scholz and Wellmer, 2013, Weikard, 2016), as the use of the biomass nutrients increases nutrient recycling and fertilizer self-sufficiency and promotes the mitigation of greenhouse gas emissions.

Globally, around 2 billion tons of municipal solid waste are formed annually, of which 34–53% is organic waste that consists mainly of FW (UNEP, 2015). The large quantities of FW produced need to be sustainably managed to prevent impact on human health and the environment. The uncontrolled degradation of FWs in landfills produces methane (CH_4) and carbon dioxide (CO_2) and promotes the leaching of nutrients, which may affect the eutrophication of water bodies. To prevent these impacts, actions and treatment options are studied and applied. FW management, including priorities for recycling and treatment, are addressed in the EU in the Waste Framework Directive (2008/98/EC, European Parliament and the Council, 2008), which aims to prevent the landfilling of the FW (further regulated in Landfill Directive, 99/31/EC, European Council, 1999) and obligates member states to carry out source separation and safe treatment of organic wastes. The European Commission aims to reduce the amount of FW by at least 30% by the year 2025, concentrating especially on the waste generation in households, manufacturing, retail, and food services sectors through national biowaste

prevention strategies (European Commission, 2014). A more ambitious goal has been set by the UN, which aims to halve the per capita global FW production at the retail and consumer levels and reduce food losses in production and supply chains, including post-harvest losses, by 2030 (UN, 2014).

Actions for the treatment management of FWs are prioritized with the waste hierarchy (Figure 1, Waste Framework Directive (2008/98/EC, European Parliament and the Council, 2008). The first step in the process of reducing the effects of FW treatment is the reduction of FW volume, i.e., the direct prevention of waste generation. The next two steps are the use and processing of FW as new food products for people or animals. However, regional regulations may prevent the use of FW as animal feed, as is the situation in the EU, where the protection against e.g., bovine spongiform encephalopathy is ensured with legislation (European Commission, 2015b). FW is also encouraged to be processed into value-added chemicals for the production of, for example, products for the pharmaceutical industry (Mirabella et al., 2014) as well as bio-plastics through acidogenic fermentation and microbial polymer synthetization (Lee et al., 2014).

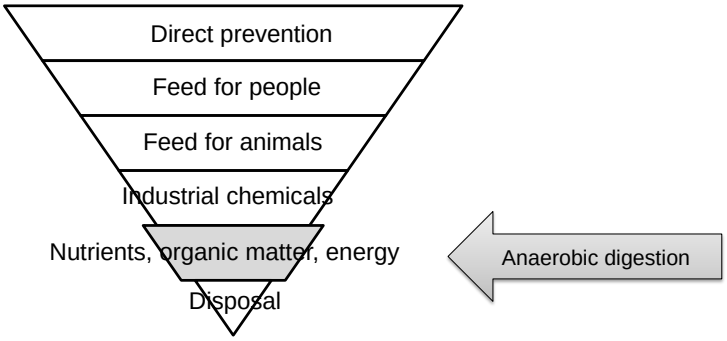

Figure 1. The food waste management hierarchy, which defines the steps for FW treatment. The step including AD treatment is indicated with an arrow (modified from UNEP, 2015).

If the above-mentioned waste hierarchy options are not possible, the FW can be processed, e.g., with anaerobic digestion (AD) or composting. The AD treatment of biodegradable wastes such as FW recovers renewable energy in the form of CH_4 for use in combined heat and power plants (CHP), in vehicles, and for grid injection; it also allows the recycling of nutrients through application of digestion residues, i.e., digestates, in crop production. With composting, the FW is degraded in aerobic conditions, producing mainly CO_2 and water. The residual composted material can be used as a soil amendment, but part of its nitrogen content is volatilized during the process and should be further captured to ensure recycling. In 2012, 90% of the FW treated in Europe (EU28) was processed biologically with both AD and composting according to the Eurostat waste treatment statistics. However, about 5% of FW was still landfilled, while around 5% was incinerated with energy recovery (Eurostat, 2015b). Of the biologically treated waste majority, around 95% (European Commission, 2008) is still composted, while the use of AD as a treatment for FWs and other organic wastes has increased in Europe with a current reported capacity of around 8 Mt (De Baere and Mattheeuws, 2012).

2 Background

2.1 Food waste generation

It is estimated that globally, one-third of the food produced for consumption becomes waste during its production, processing, distribution, and consumption (European Commission, 2014, Gustavsson et al., 2011). Therefore, FW is generally categorized as part of the food that becomes wasted during its journey from the farm to the fork (Figure 2) (European Commission, 2010). In this study, the term FW is used to describe the organic waste generated in the consumption stage, e.g., in households and restaurants. FW from households can be classified as a part of biowaste, which also consists of biodegradable waste from gardens (European Commission, 2015a).

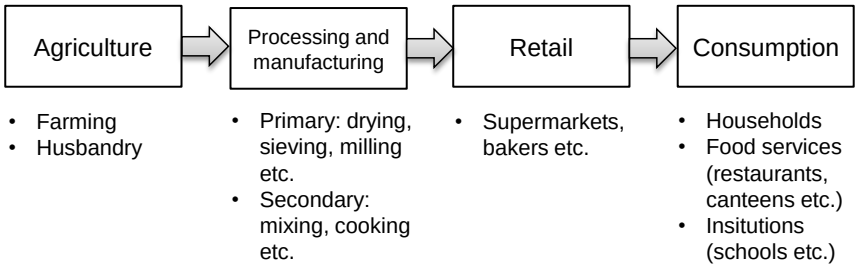

Figure 2. Food waste generation during the food production chain (adapted from Papargyropoulou et al., 2014).

The generation of FW in different countries is dependent on the income level of the consumers, but the overall amount of FW generated is somewhat similar in both low- and high-income countries. In low-income-level countries most of the FW (80%) is generated primarily during farming and transportation, while in high-income countries, consumers and retail sectors are responsible for around 80% of the FW generation (UNEP, 2015). The FW generation in developing countries is due to inadequate storage and transportation systems for food products, along with poor market situations, while developed economies have set high standards for food products (cosmetic standards, best before dates,

etc.), which, along with the relatively cheap price, increases FW amounts (Gustavsson et al., 2011, UNEP, 2015).

The total FW quantity produced each year in Europe (EU27) has been estimated to be around 90 Mt (180 kg per capita), of which an estimated 38 Mt (76 kg per capita) is generated in households (based on data from the year 2006, European Commission, 2010). As a comparison, in the US, 36 Mt (120 kg per capita) of residential and commercial FW was produced in 2011 (US EPA, 2013). The EU estimates an increase in the FW amounts to 120 Mt by 2020 (European Commission, 2016), which is mainly due to increased FW generation in households (Figure 3). The amount of household FW has almost doubled from 2004 to 2012, and FW from food manufacturing and agriculture shows a decreasing trend (based on the Eurostat values of animal and mixed food waste and vegetal wastes in EU28, Eurostat, 2015a).

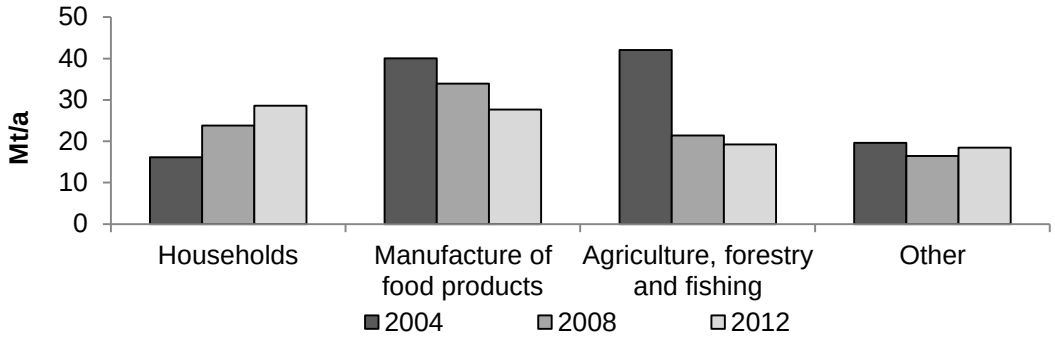

Figure 3. Generation of food waste in different stages of production during 2004–2012 in EU28 countries (env_wasgen, W091-W092, Eurostat, 2015a).

2.1.1 Composition

FW usually consists of different raw and cooked food materials, beverages, and pet food that are generated and discarded during manufacturing, distribution, retail, and food services, as well as in households (European Commission, 2010, Lebersorger and Schneider, 2011). The FW produced in households can be divided into unavoidable and avoidable FW. Unavoidable FW consists of, e.g., fruit and vegetable peels, meat trimmings, bones, shells, coffee filters and grounds, and tea bags, which cannot be eaten. Conversely, avoidable FW is the part of waste that has been edible but is wasted due to, e.g., too-large quantities of food prepared and purchased, which leads to disposal of leftovers or as a result of food spoiling (Lebersorger and Schneider, 2011, WRAP, 2009). However, the ratio between unavoidable and avoidable FW is dependent on consumer habits and cultural differences where, e.g., bread crusts and potato/apple peels can be classified into either group depending on eating habits (Lebersorger and Schneider, 2011, Schott et al., 2013) or can be classified into the "possibly avoidable" group (WRAP, 2009).

In Europe, the FW generated in households is mostly avoidable waste. Around 45% of food is wasted as whole or only partly consumed products, mainly due to poor planning with grocery shopping, while around 10–15% is wasted as leftovers (Langley et al., 2010, Lebersorger and Schneider, 2011), which can be seen as avoidable FW. In the preparation stage, the unavoidable FW consisting of peels, trimmings, etc. has been reported to comprise 33% (Langley et al., 2010) or 44% (Lebersorger and Schneider, 2011) of the total FW in households. All in all, the total amount of avoidable FW has been reported to vary from 34% to over 60% of the total FW generated in households in Europe (Langley et al., 2010, Lebersorger and Schneider, 2011, Schott et al., 2013, WRAP, 2009). Depending on the classifications and study areas, avoidable FW shares of as high as 80% have been reported (Vanham et al., 2015, WRAP, 2009). For example, in Finland and Sweden, the avoidable FW amount in households has been estimated to be 20–30 kg (Silvennoinen et al., 2014) and 30 kg per capita per year (Schott et al., 2013), while the avoidable FW amount in households has also been reported to be as high as 97 kg per capita on average in Europe (Vanham et al., 2015).

The composition of FW is dependent on the source of the waste (Zhang et al., 2007), e.g., the stage of the FW production chain. Composition of FW originating from households and restaurants is also affected by the eating habits and diets of consumers – varying both seasonally and geographically – as well as the consumer's social status, income level, and environmental awareness (Hansen et al., 2007a). The main constituents of FWs are carbohydrates, for example, fruits and vegetables (around 50% of FW), with lower contents of protein and fat-containing dairy, meat, and fish, and other carbohydrates such as cereals (in total, 10–50% of FW, Table 1). However, prepared meals are reported to constitute 3 to 20% of FW, representing a fraction of waste that could be totally avoided by proper meal and shopping planning (Silvennoinen et al., 2014, WRAP, 2009). Over 90% of the produced FW could be also avoided in the categories of rice/cereal products, dairy, and other foods, while the fruit and vegetable, meat, and drink categories all include 20–50% of unavoidable material that cannot be consumed (Table 1).

2.1.2 Collection and characteristics

The collection of FW in Europe is usually executed regionally as separate collection (source separated FW) or as mixed with other municipal solid waste (MSW). The source separated FW in households and restaurants/canteens is collected into a separate fraction than the other municipal wastes. Usually in households, FW is collected into its own recycling bin, which is lined with either biodegradable plastic, plastic or paper, bags (Al Seadi et al., 2013, Bernstad et al., 2013). Depending on the local waste collection regulations, both pet litter and yard and garden waste can be included in FW collection (Lebersorger and Schneider, 2011, Saveyn and Eder, 2014), which affects the chemical characteristics, e.g., the TS content of the FW (Hansen et al., 2007a). If not separated at home, the FW can be mixed with other fractions as mixed-MSW, while fractions such as metals and glass are collected separately.

Table 1. Household food waste (FW) composition in selected European countries. Results are based on studies where FW composition was studied by either by hand-sorting the FW from household waste bins (hand-sorting) or by FW weighing by consumers when FW was formed (consumer diary). The results are percentages (%) of the total FW amount. The amount of avoidable FW is estimated and is presented as % of the FW type.

Food waste class	Detailed class	Finland[a]	Portugal[a]	Italy[a]	UK[a]	Greece[b]	Austria[c,d]	Finland[e,d]	UK[f]	Average (% of FW generated)	Avoidable (% of FW generated in each class)[g]
		Hand-sorting						Consumer diary			
Fruits and vegetables	Fruit and vegetable waste	25.2	-	29	48.2–53.8	54–71	-	-	-	30–60	47–88
	Fruit and vegetables (whole)	6.4	-	1.8	7.1–12.2	-	-	-	-		
	Fruit	-	17.8	-	-	-	8.6	-	30		
	Vegetables	-	31.2	-	-	-	17.7	19	23		
	Fruits and berries	-		-	-	-	-	13	-		
	Salads	-	0.6	-	-	-	-	-	-		
Pasta/rice, bread, bakery, cereals	Pasta/rice, cereals	0.3	-	6.4	-	0	1.9	4	-	5–20	>90
	Pasta, rice	-	-	-	0.3–1.5	-	-	-	-		
	Cereals	-	-	-	0.3–0.4	-	-	-	-		
	Bread and bakery	2.7	2.6	1.4	10.1–13.3	1.5–8.8	-	13	-		
	Bread and cereals	-	-	-	-	-	-	-	16		
	Baking ingredients and cereals	-	-	-	-	-	1.5	-	-		
	Confectionery and snacks	0.2	0.3	0	-	-	0.9	-	-		
	Confectionery and desserts	1.9	-	-	-	-	11.7	-	-		
	Cakes, desserts, confectionary, snacks -		-	-	0.1–0.5	0–1.4					
Meat, fish, eggs	Meat and fish	2.7	6.1	2.1	3.6–10.9	2.4–4.5	10.9	7	-	2–10	50–65
	Meat	-	-	-	-	-	-	-	2		
	Fish	-	-	-	-	-	-	-	1		
	Bones	0.4	0	1.1	2.9–8.9	-	-	-	-		
	Eggs	-	-	-	0.6–1.2	-	0.6	-	-		
	Egg shells	1	-	0.7	-	-	-	-	-		
Dairy products	Dairy, milk	0.4	0.6	0	0.3–0.6	-	7.5	17	-	0.5–10	>90
	Cheese	-	-	-	-	-	4.6	-	-		
	Milk, cheese, eggs	-	-	-	-	0.3–1	-	-	10		

Drinks	Drinks (coffee grounds, tea bags, etc.)	19.5	0.1	0	6.2–10.4	0.3–0.4	1.6	-	9	1–15	60
Meals	Mixed meals	4.4	24	0.7	-	0.2–1.5	-	-	-	3–20	>90
	Prepared meals	-	-	-	-	-	2.9	18	-		
	Convenience and take-out food	-	-	-	-	-	2.6	6	-		
	Sandwiches	-	-	-	-	-	1.9	-	-		
Other food	Other food	5.7	-	3.6	0.3–2.3	-	-	-	10	1–7	>90
	Dried food, powders	-	0.2	-	-	-	-	-	-		
	Jam	-	-	-	-	-	1.7	-	-		
	Sauces	-	-	-	-	-	1.4	-	-		
	Spices and herbs	-	0	0	-	-	1.2	-	-		
	Spreads and similar delicatessen	-	-	-	-	-	0.8	-	-		
	Pet food	-	-	-	-	-	0.5	-	-		
Other biodegradable	Other biowaste	-	-	-	0.8–1.6	23–30	4.2	3	-	1–30	-
	Biodegradable bags	1.6	-	3.7	1.9–3	-	-	-	-		
	Garden waste	7.2	0.8	15.2	-	-	-	-	-		
	Paper and cards	17.5	6.4	13.8	-	-	-	-	-		
Other	Undefined	-	-	12.8	-	-	-	-	-	-	-
	Stones, seeds, etc.	-	-	4.8	-	-	-	-	-		
	Contaminants	2.8	9.3	3	0–0.4	-	-	-	-		

[a]Valorgas, 2010a, [b]Malamis et al., 2015, [c]Lebersorger and Schneider, 2011, [d]based on composition of avoidable food waste generated in households, [e]Silvennoinen et al., 2014, [f]Langley et al., 2010, [g]according to WRAP, 2009

-, not available

In waste treatment plants, the pretreatment methods for FW are, for example, the separation of unwanted waste fractions, e.g., plastic, glass, and metals, which are usually due to the negligence of the consumers (Jank et al., 2015). Contaminants in FW decrease the quality and potentially increase the heavy metal and organic contaminant concentrations. Source separated FW usually undergoes mechanical separation and homogenization processes prior to subsequent treatment, e.g., in AD (Bernstad et al., 2013, Davidsson et al., 2007, Hansen et al., 2007a, Hansen et al., 2007b). The treatment of source separated FW with different pretreatment methods (shredders, screens, and magnets) thus affects the chemical characteristics and amount of organic matter to be further utilized (Hansen et al., 2007b, Jank et al., 2015). However, the differences in the characteristics between FWs can also be minor, depending on the applied treatment processes (Davidsson et al., 2007). If the waste is collected as mixed-MSW, the organic fraction (organic fraction of municipal solid waste, OFMSW) is separated using mechanical separation (Al Seadi et al., 2013, Tchobanoglous et al., 1993). The separation produces mechanically recovered OFMSW whose characteristics, however, differ from source separated FW, e.g., with higher TS and VS content as well as C/N ratio (Table 2, Zhang et al., 2012). Mechanically recovered OFMSW also contains more impurities and heavy metals, for example, than source separated FW (Zhang et al., 2012). The contamination of other waste fractions is likely (Al Seadi et al., 2013), which is why source separation of FW is more promoted and favored.

Municipal FWs have been found to have rather uniform characteristics despite temporal or geographical differences, while different eating habits affect FW composition, (Davidsson et al., 2007, Hansen et al., 2007b, Valorgas, 2010a). In Table 2, FW characteristics from Europe, Asia, and North America are presented based on literature, where the overall characteristics of different FW samples are somewhat similar. Depending on the collection and possible separation treatments, the FW usually has a total solids (TS) content of around 20–30%, of which 90–95% is considered organic (VS, volatile solids, Table 2). Around 50% of the VS in FWs consist of carbohydrates (fruits, vegetables, bread, and cereals), while both proteins and fats contribute to 10–30% of the organic matter. Furthermore, the relatively high protein content affects the TKN (Total Kjeldahl nitrogen) content of the FW, which can vary from 3 to 14 g/kgFM (fresh matter) with an of average 5–7 g/kgFM (Table 2). The carbohydrates in FW consist of different sugars, e.g., starch, cellulose and hemicellulose (Alibardi and Cossu, 2015), as well as lignin, the building materials of plant cell walls (Hendriks and Zeeman, 2009). In FW, the cellulose and hemicellulose content is around 4–10% of the organic matter content; thus, it is highly dependent on the FW composition, while the lignin content can vary from 1.6 to over 25%VS (Tanimu et al., 2015, Vavouraki et al., 2014, Zhang et al., 2015b, Zhang et al., 2012). The high variation within the lignin content between FWs is mainly due to the heterogeneity of the FW (Papadimitriou, 2010) but is also a results of the complex nature of lignin and different analyzing methods, which increase the deviation (Hatfield and Fukushima, 2005).

Table 2. Food waste characteristics in different studies from Europe, Asia, and North America.

FW type	Country of origin	TS (%)	VS (%)	TKN (g/kgFM)	P-tot (%TS)	K-tot (%TS)	N (%TS)	C (%TS)	H (%TS)	O (%TS)	Fats, lipids (%VS)	Proteins (%VS)	Carbo-hydrates (%VS)	Reference
FW	EU	23.7	21.6	4	-	-	-	-	-	-	17.2	10.6	71.5	Ariunbaatar et al., 2015
FW	EU	22.2	21.1	4.7	-	-	-	-	-	-	9.2	14.3	76.5	Ariunbaatar et al., 2014b
FW	Spain	86.8	78.5	12.8	0.3	-	-	45.1	-	-	-	-	-	Forster-Carneiro et al., 2008
ss-FW	Denmark Sweden	17-37	14-34	-	0.3-0.6	0.8-1.3	2.2-3.1	45-52	6.4-7.8	-	8.1-16.6	8.1-16.6	29-55	Davidsson et al., 2007
ss-FW	UK	23.7-28.6	21.7-24	7.4-8.1	0.3-0.6	0.9-1.4	2.8-3.4	48.3-51.3	5.5-6.7	29.8-34.7	14.8-15.7	18.3-23.5	-	Valorgas, 2010a
ss-FW	Finland	27.0	24.9	6.5	0.3	1	2.5	49.4	-	-	15.6	16.2	-	Valorgas, 2010a
ss-FW	Italy	24.4-27.5	20.2-23.6	7.0-7.2	0.3	1	2.6	47.2	-	-	20.2	18.6	-	Valorgas, 2010a
ss-FW	Portugal	6.3-6.3	5.0	2.7-4.4	0.4-0.9	2.9	5.7	54.8	-	-	31.4	55.4	-	Valorgas, 2010a
ss-FW	UK	23.7	21.7	8.1	0.5	1.4	3.4	47.6	7.0	33.3	15.1	23.5	45.3	Zhang et al., 2012
ss-FW	USA	30.9	26.4	-	0.5	0.9	3.2	46.8	-	-	-	-	-	Zhang et al., 2007
h-FW	UK	23.7	22	7.4	-	-	3	52.3	6.9	-	-	-	-	Yirong et al., 2015
r-h-FW	Malaysia	-	-	-	0.7	0.5	2	33.5	-	-	27.1	-	-	Tanimu et al., 2015
r-FW	Greece	18.5	17.4	5.0	0.7	-	-	-	-	-	7.6	9.1	51.8[a]	Vavouraki et al., 2014
r-FW	Spain	6.4	6	-	-	-	1.3	-	-	-	0.7	7.8	-	Cuetos et al., 2010
r-FW	USA	19.6	18.7	-	-	-	-	-	-	-	-	-	-	Grimberg et al., 2015
r-FW	USA	23.6	22.9	-	-	-	-	-	-	-	-	-	-	Grimberg et al., 2015
r-FW	China	23.8	21.2	-	-	-	2.7	50.3	7.1	29.1	-	-	-	Zhang et al., 2015a
r-FW	China	19.9	18.04	-	-	-	2.8	48.7	7.3	32.6	24.2	16.8	59.2	Liu et al., 2012
r-FW	Korea	18.1	17.1	5.4	0.8		3.5	46.7	6.4	36.4	13.6	19.2	65.3	Zhang et al., 2011
OFMSW	Spain	81	42.6	34	0.1	-	-	30.5	-	-	-	-	-	Forster-Carneiro et al., 2008
sh-OFMSW	Spain	81.9	43.4	22	0.1	-	-	30.7	-	-	-	-	-	Forster-Carneiro et al., 2008
mr-OFMSW	UK	52.8	33.6	14.4	0.2	0.4	1.3	33	4.8	22.2	6.9	13	34	Zhang et al., 2012

Food waste (FW), organic fraction of municipal solid waste (OFMSW), source separated (ss), restaurant (r), household (h), mechanically recovered (mr), shredded (sh)
[a]Total sugars
-, not available

2.2 Anaerobic digestion

2.2.1 Principles of anaerobic digestion

Microbiology

AD is a synergistic process carried out by micro-organism consortiums consisting of both bacteria and archaea, which function under the absence of oxygen. During AD, the complex biomolecules of the feedstock are degraded into low molecular weight compounds: CO_2 and CH_4. The four stages of the AD – hydrolysis, acidogenesis, acetogenesis and methanogensis – function in symbiosis, producing substrates for the subsequent process stages (Figure 4, Jain et al., 2015, Merlin Christy et al., 2014). In the first stage, hydrolysis, the insoluble macromolecules, proteins, long-chain carbohydrates, and fats are degraded into smaller compounds such as short-chain sugars, amino acids, and long-chain fatty acids (Angelidaki and Sanders, 2004). Hydrolytic micro-organisms produce extracellular enzymes, such as cellulase, cellobiase, xylanase, amylase, protease, and lipase, which hydrolyze compounds with high molecular weight (reviewed in Jain et al., 2015, Merlin Christy et al., 2014). In the second stage, acidogenesis, the hydrolyzed products are further broken down by fermentative bacteria into different organic acids (volatile fatty acids, VFAs), hydrogen (H_2), CO_2 and organic compounds, e.g., ethanol (Merlin Christy et al., 2014). During the degradation of amino acids, inorganic ammonia (NH_3) is also formed, which converts into water-soluble ammonium nitrogen (NH_4^+, NH_4-N) in pH- and temperature-dependent conditions (Kayhanian, 1999). In the third stage of the AD, acetogenesis, the VFAs and other intermediates from the previous stage are converted by bacteria to acetate, CO_2 and H_2, where the synergy between hydrogen-converting methanogens prevents the accumulation of intermediate compounds, such as VFAs (reviewed in Merlin Christy et al., 2014).

During methanogenesis, methanogenic micro-organisms, i.e., archaea, transform intermediates (acetate and H_2) into CH_4 and CO_2. Acetoclastic methanogens degrade the acetate to produce CO_2 and CH_4 while H_2 is simultaneously converted by hydrogenotrophic methanogens by the reduction of CO_2 (Merlin Christy et al., 2014). Typically, 70% of the CH_4 during AD is produced through acetoclastic methanogensis and around 30% through hydrogenotrophic methanogenesis (Jain et al., 2015). However, the process conditions, e.g., high ammonia concentrations during the digestion, have been reported to change this balance toward the hydrogenotrophic pathway, where the acetate is degraded by syntrophic acetate oxidizers (Banks et al., 2012, Karlsson et al., 2012). Additionally, sulfate-reducing micro-organisms can compete with methanogens and form hydrogen sulfide (H_2S) through the microbial reduction of sulfate (SO_4^{2-}) with H_2 and acetate (Barrera et al., 2013). Another pathway to the formation of H_2S is the degradation of proteins into amino acids and further to sulfides (Figure 4, Möller and Müller, 2012).

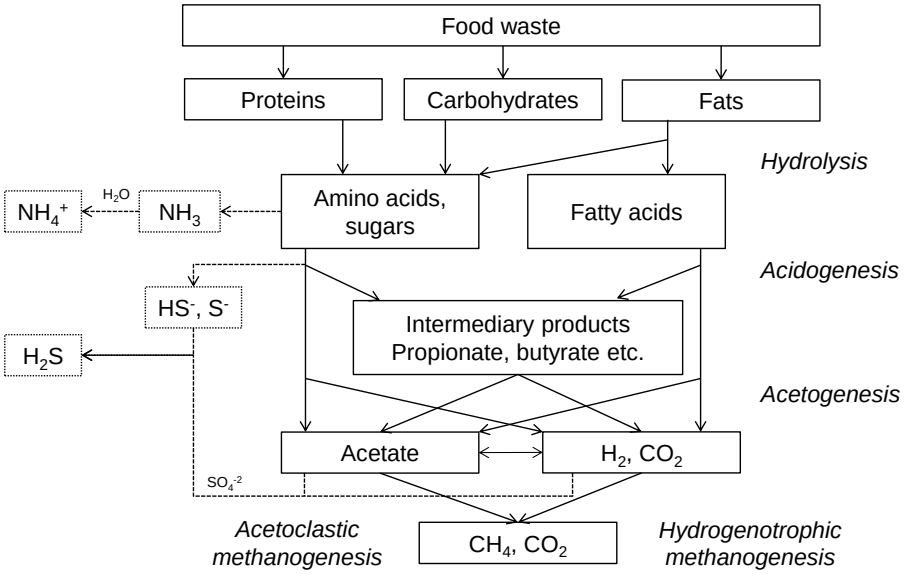

Figure 4. Schematic diagram of the degradation pathways of, e.g., food waste into biogas, hydrogen sulfide and ammonium nitrogen during AD (modified from Merlin Christy et al., 2014 according to Barrera et al., 2013, Kayhanian, 1999).

Theoretically, AD produces biogas consisting of 50% CO_2 and 50% CH_4; thus, the composition of the feedstock affects the ratio. Typically, biogas contains 50–70% CH_4 and the highest yield can be achieved with materials with high fat and protein content – for example FW – due to the high amount of reduced carbon within these molecules (Table 3, Angelidaki and Sanders, 2004). Furthermore, other gas components can be formed during AD, e.g., H_2S, H_2, N_2 and water vapor (Rasi et al., 2010, Tchobanoglous et al., 2013), which affect the biogas composition due to substrate composition, microbial consortia and digester conditions (Angelidaki and Sanders, 2004, Jain et al., 2015, Möller and Müller, 2012).

Table 3. Theoretical methane yield and composition of organic substrates (Angelidaki and Sanders, 2004).

Substrate	Composition	CH_4 yield (dm^3CH_4/kgVS)	CH_4 (%)
Carbohydrate	$(C_6H_{10}O_5)_n$	415	50
Protein	$C_5H_7NO_2$	496	50
Fat	$C_{57}H_{104}O_6$	1014	70

The theoretical methane production of, for example, food waste can be calculated based on i) the component composition of the substrate (as presented in Table 3), ii) the elemental composition using Buswell's equation, or iii) the chemical oxygen demand (COD) of the substrate (Nielfa et al., 2015). Buswell's equation is based on the elemental composition (C, H, O, N) (reviewed in e.g. Angelidaki and Sanders, 2004); therefore, it gives slightly different results than the component-based estimation. The COD estimation is also reported to give higher values compared to the other two methods (Nielfa et al., 2015). Furthermore, when the theoretical values are compared with the experimental values, the

theoretical values tend to overestimate the actual value achieved with lab experiments due to the composition of the studied material. The feedstock contains not only easily and readily degradable matter (sugars, proteins, fats) but also inert and non-biodegradable materials, e.g., lignin, an organic compound with a complex nature making it impossible (Angelidaki and Sanders, 2004) or difficult to be degraded anaerobically (Hatfield and Fukushima, 2005, Hendriks and Zeeman, 2009). The accessibility of the organic compounds is also essential, where the biodegradability of, for example, cellulose decreases if it is incorporated into lignocellulosic complexes (Hendriks and Zeeman, 2009).

AD conditions (OLR, HRT, pH, temperature)

The optimal conditions for AD are defined by the microbial requirements. As the growth rates of different micro-organisms in the different stages of AD vary, the balance between degradative reactions is essential. To maintain balanced and efficient digestion, the equilibrium between microbial populations is kept steady with substrate supply and accessibility through particle size, organic loading rate (OLR), hydraulic retention time (HRT), and other process parameters (e.g., pH and temperature) (Jain et al., 2015). The optimum pH for AD is between 6 and 8.5 – this is where the methanogens are considered most sensitive toward changes in pH (Ferrer et al., 2010, reviewed in Jain et al., 2015). The pH of the process is affected by the production of acids during acidogenesis and the conversion of acids during methanogenesis (Kayhanian, 1999, Qiao et al., 2013). Also, the buffering capacity, arising from the concentrations of, e.g., HCO_3^- and NH_4^+, affects digester pH (Qiao et al., 2013). However, the instabilities in digestion can affect the microbial synergies and lead to excess production of acids, decreasing the pH.

The process temperature affects the microbial activity directly, and it is therefore an important factor in successful AD (Jain et al., 2015); the applied temperatures are dependent on the requirements of the microbial consortia, especially methanogenic archaea (Ferrer et al., 2010). The two most significant temperature zones used for AD are mesophilic (25–40°C) and thermophilic (50–65°C) zones. Thus, psychrophilic digestion is also possible in temperatures under 20°C (Angelidaki and Sanders, 2004, Jain et al., 2015). Mesophilic micro-organisms have a slower growth rate compared to thermophiles, so they have been regarded as more resistant to changes in process parameters, e.g., temperature and pH, than thermophiles (reviewed in Ferrer et al., 2010). The growth rate also affects the applied HRT; in mesophilic digesters, HRT is kept in the minimum of 15 days, while thermophilic reactors operate at 55°C with HRT of 5 to 8 days in minimum (reviewed in Ferrer et al., 2010, Tchobanoglous et al., 2013). The shorter HRT increases the rate of thermophilic digestion as well as loading capacity and decreases the volume of digesters improving the economics of the process. Additionally, higher CH_4 yields are possible due to an increase inthe biochemical reaction rates with the increasing temperature compared with the mesophilic process (Ferrer et al., 2010, Tchobanoglous et al., 2013).

Inhibition and trace element supplementation

The inhibition of AD refers to a decrease in the growth and activity of the micro-organisms (reviewed in Rajagopal et al., 2013). The relatively high protein content within the substrate increases the potential of ammonia inhibition during AD, as the ammonia is released during hydrolysis through the deamination of nitrogenous compounds (proteins, phospholipids) (Kayhanian, 1999). High ammonia nitrogen concentrations have been reported to lead to process instability, and even failure, and decreased CH_4 production due to the accumulation of VFAs. The inhibition has been reported to arise in varying concentrations depending on the substrate material and process conditions (reviewed in Rajagopal et al., 2013). The ammonia inhibition has also been proposed to be dependent on the pH buffer capacity within the reactor, which is dependent on the NH_4-N concentrations (Prochazka et al., 2012). However, the major part of ammonia inhibition is not considered to arise from the ionized form of ammonium (NH_4^+) but from unionized free ammonia nitrogen (FAN, NH_3) (Chen et al., 2008, Rajagopal et al., 2013). The ratio between NH_4^+ and NH_3 in digesters is dependent on both the temperature and pH, where the NH_3 shows increasing concentrations and inhibition along with these process parameters. The permeability of the NH_3 to the cell membrane is the foremost cause of inhibition for the methanogenic micro-organisms (Gallert and Winter, 1997), which decreases the intracellular pH and subsequently causes imbalance and inhibition within the cell homeostasis (reviewed in Prochazka et al., 2012, Rajagopal et al., 2013). Another explanation for the ammonia inhibition is the direct inhibition of specific methanogenic enzyme reactions (reviewed in Rajagopal et al., 2013).

The diffusion of ammonia through the cell membrane is dependent on the physiology of the micro-organisms, and the species most vulnerable to ammonia inhibition is methanogens, but resistance varies greatly between species (Rajagopal et al., 2013). In certain conditions (temperature 35, pH 7.7, FAN level 200 mg/l), acetogenic bacteria has also been proposed to be more sensitive to inhibition than methanogens (Kayhanian, 1999). From the methanogens, the acetoclastic micro-organisms are, however, more sensitive, and it is known that the high NH_4-N concentration within digesters alters the CH_4 formation pathway toward the hydrogenotrophic route, as the hydrogenotrophes are more resistant (Banks et al., 2012). Moreover, the acclimation of the microbial population toward the high NH_4-N concentrations is possible; however, whether the acclimation time only allows new, more resistant micro-organisms to grow or truly acclimates the present populations is still unknown (Rajagopal et al., 2013). Besides acclimation, controlling the C/N ratio inside digesters and diluting feedstock can be used to decrease nitrogen, and subsequently, the NH_4-N concentrations, and avoid inhibition (Kayhanian, 1999, Rajagopal et al., 2013). In AD, the suitable C/N ratio is proposed to be around 25 (Chen et al., 2008, Jain et al., 2015), as N is also needed as a nutrient for microbial growth (Prochazka et al., 2012).

The accumulation of VFAs in the digester is the result of an imbalance within the microbial relationships between H_2 producing and converting micro-organisms or the VFA utilizing populations (Ferrer et al., 2010), which can be due to, e.g., ammonia inhibition. Also, insufficient retention times can affect the VFA degradation (Ferrer et al., 2010) of especially propionate degrading micro-organisms due to their relatively long growth times (Qiao et al., 2013), which leads to the accumulation of propionate and lowers digester pH. Imbalanced VFA production has been connected with trace element (TE) deficiencies, affecting the process stability and lowering CH_4 production (Banks et al., 2012, Zhang and Jahng, 2012). TEs play a role in the synthesis of the coenzymes and cofactors in the methanogenic pathway, where the shortage of TEs disturbs the enzymatic reactions in methanogenesis and decreases CH_4 formation (Zhang et al., 2015a). Different TEs are required by different enzymes and coenzymes, and the CH_4 formation pathway affects the need for certain TEs (Banks et al., 2012). However, too high concentrations of TEs can be inhibitory to methanogens (Zhang et al., 2015a).

The TEs are not necessarily in bioavailable ionic form but can also occur as precipitates, which lowers the bioavailability of the elements (Zhang et al., 2015a) and increases the amount of supplemented TEs. The dosage of TEs should be properly managed to avoid environmental load during digestate fertilization, as many of the TEs are also classified as heavy metals. One proposed solution is to use, e.g., chelating agents, which increases the bioavailability of metals and simultaneously reduces the amount of TE supplementation needed (Zhang et al., 2015a).

2.2.2 Feedstock pretreatment

Different feedstock pretreatment methods based on either mechanical grinding or heat and pressure can be applied to improve the CH_4 production of the AD by accelerating the hydrolysis and acidogenesis steps through solubilization (Izumi et al., 2010). Heat-based pretreatments enhance hydrolyzation, accelerate degradation and increase CH_4 production, as the heat disintegrates molecules and chemical bonds that increase biodegradability. Additionally, thermal pretreatments are reviewed to enhance the digestate handling by improved dewatering and decreased viscosity (Liu et al., 2012, Zhou et al., 2013).

Thermal and hydrothermal pretreatments have been widely studied as a means of hydrolyzing recalcitrant components, e.g., fibers, in a wide range of wastes to make them easier to degrade (Papadimitriou, 2010, Ren et al., 2006). One hydrothermal treatment is autoclaving, where water is used as a reagent at increased temperature and pressure, to hydrolyze and solubilize sugars, starches, proteins, and hemicellulose (Papadimitriou, 2010, Ren et al., 2006). Materials pretreated by autoclaving under various conditions have shown increased CH_4 production in batch tests; digested swine slurry autoclaved at 120°C showed a 115% increase in CH_4 yield (Menardo et al., 2011), and autoclaving of waste activated sludge (WAS) increased CH_4 yield in continuous tests by 12% and 25% after autoclaving at 135°C and 190°C, respectively (Bougrier et al., 2007). However, the CH_4 content

was lower than control when dewatered pig manure was thermally pretreated in 110–150°C (Rafique et al., 2010), as thermal treatments have been observed to decrease CH_4 yields with high protein-containing feedstocks (Bougrier et al., 2008, Cuetos et al., 2010). The decrease in the degradation capacity of micro-organisms has been proposed to be related to the formation of melanoidins or Maillard compounds, which are formed through reactions between sugars and amino acids (Bougrier et al., 2008, Liu et al., 2012, Monlau et al., 2013). These compounds are reported to be toxic (Cuetos et al., 2010) and difficult or even impossible to be degraded in AD (Bougrier et al., 2008). Maillard compounds start to form at temperatures above 100°C depending on the composition of proteins and carbohydrates in the feedstock and the retention time (reviewed in Ariunbaatar et al., 2014a). Additionally, other toxic and inhibitory compounds, such as furanic and phenolic compounds, are formed during the high-temperature pretreatment of lignin-containing materials (Monlau et al., 2014).

2.2.3 Agricultural use of digestate

Anaerobic digestate, i.e., the residue produced in AD, is a mixture of partially degraded organic matter from the digester feedstock, microbial biomass and inorganic compounds, i.e., nutrients (Alburquerque et al., 2012b). The organic compounds in the anaerobic digester degrade during the microbiological processes, but all the nutrients are conserved, which increases the agronomic value of the produced digestate (Tambone et al., 2010). In Europe, the total digestate production from all digested biomasses in 2010 was 56 Mt per year, of which 80–97% was used in agriculture (Saveyn and Eder, 2014). When used as fertilizer, the nutrients in FW digestate can be returned to agriculture to close the nutrient cycle and thereby reduce the need for mineral fertilizers. In several life cycle analyses, 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 (Bernstad and 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 and la Cour Jansen, 2011, Boldrin et al., 2011, Haraldsen et al., 2011), which is dependent on the local soil quality (Rigby and Smith, 2013) and meteorological conditions, as well as digestate characteristics (Evangelisti et al., 2014).

Agronomic value

The digestate's characteristics and quality define its effect on plant growth, soils, and environment. The agronomic value of the digestate is dependent on four main characteristics: i) nutritional value to plants and organic matter content, ii) the content of contaminants (plastic, glass, metals), iii) the content of toxic and inhibitory compounds, e.g., heavy metals, and iv) the hygienic quality (Abubaker et al., 2012, Nkoa, 2014, Teglia et al., 2011). AD converts most of the organic material of the feedstock 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). Organic N – bound to the proteins

within the FW – are degraded, and NH_4-N is formed (Kayhanian, 1999), while P from the proteins and phospholipids degrades into its mineral form, phosphates (PO_4^{3-}). Plant availability of N and P is dependent on the amount of soluble nutrient forms within digestates (reviewed in Möller and Müller, 2012).

The N availability to plants after fertilization is dependent on the amount of mineral N within the digestate, where the balance between organic and inorganic forms is especially essential (e.g. NH_4-N concentration and NH_4-N/TKN ratio, Fouda et al., 2013, Teglia et al., 2011). NH_4-N/TKN ratios over 50% are usually considered indicative of good fertilizer effect, while digestates with lower NH_4-N/TKN ratios are more suitable for soil amendments (reviewed in Nkoa, 2014, Teglia et al., 2011). Soluble NH_4-N increases the short-term effect of N in soils enhancing plant growth shortly after fertilization (Abubaker et al., 2012, Gutser et al., 2005), as the NH_4-N is also considered as the limiting factor in plant growth (Odlare et al., 2011). Also, the C/N – more specifically, the C/N_{org} ratio – affects the release of N in the soil after fertilization, where a low C/N_{org} ratio promotes N release and availability (Gutser et al., 2005).

The organic matter in the digestate increases the soil carbon balance, which leads to enhanced microbial processes (Abubaker et al., 2012, Odlare et al., 2008) and enzymatic activity (Galvez et al., 2012). This further increases mineralization and long-term nutrient release in soils (Abubaker et al., 2012, Galvez et al., 2012, Odlare et al., 2008, Odlare et al., 2011). As it follows, there is a minimum value for the organic matter content (VS 15% of TS) in digestates within the upcoming European regulations concerning digestate use as a fertilizer in agriculture (Saveyn and Eder, 2014). However, a VS content that is too high, depending on its composition, can lead to imbalanced microbial activity and immobilization of N (Alburquerque et al., 2012b, Gutser et al., 2005) as well as phytotoxicity due to organic acids (Abdullahi et al., 2008). Organic acids, e.g., VFAs, are also reported to act as a carbon source for soil micro-organisms and be rapidly degraded after digestate application (Kirchmann and Lundvall, 1993). In the EU proposal for digestate fertilization, the maximum value for the digestate VFA concentration is 1500 mg/l (Saveyn and Eder, 2014). Besides VFAs, other acids also constitute the soluble organic matter within digestates, e.g., humic acids. These acids are proposed to act as bio-stimulants for plant growth (Scaglia et al., 2015) and are related to digestate stability (Zheng et al., 2014) along with other stable molecules, e.g., lignin and long-chain proteins (Tambone et al., 2009). The organic acid concentrations within digestates are also related to digestate pH, which can decrease soil pH and enhance heavy metal mobilization in acidic conditions (Otabbong et al., 1997), while alkaline conditions induce the volatilization of NH_4-N during and after digestate spreading (Nkoa, 2014). However, the effect of the digestate pH on soils is highly dependent on the soil's characteristics (Alvarenga et al., 2015).

Excess application of digestate can lead to harmful effects on plants and soils due to, e.g., the quantity and quality of the organic matter or any impurities, including heavy metals, organic contaminants, or

pathogens (Alburquerque et al., 2012b, Govasmark et al., 2011). Feedstocks of urban biogas plants, e.g., FWs and other 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 AD (Govasmark et al., 2011) and possibly accumulate in the soils or the food chain after digestate use (Otabbong et al., 1997, Zhu et al., 2014). In the EU, the heavy metal load in digestates/composts is regulated by national legislation, and a new quality standard proposal will set limits for heavy metals (Zn, Cu, Ni, Cd, Pb, Hg, Cr) and organic contaminants (polychlorinated biphenyls, polycyclic aromatic hydrocarbons, polychlorinated dibenzodioxins and –furans, and perfluorocarbons) (Saveyn and Eder, 2014).

Digestate post-treatment

The post-treatment of digestates may be used if the digestates have unbalanced nutrient ratios for plant growth (Camilleri-Rumbau et al., 2015), which leads to the need for additional mineral fertilizer supplementation (Svensson et al., 2004). Raw digestates, depending on the feedstock, digester type, and operational parameters, can be relatively diluted, which increases the spreading amounts to achieve the desired fertilization level. The transportation of large quantities of water is inefficient, and large digestate volumes increase the transportation costs, especially over longer distances (Rehl and Müller, 2011, Teglia et al., 2011). Different digestate processing methods can be applied to tackle the high moisture and uneven nutrient content. Digestate post-treatment can be divided into processes where i) the nutrient concentration of the material is increased in comparison with the original digestate, or where ii) the aim is to produce separate nutrient containing mineral fertilizer-like material. The nutrient concentration can be increased, e.g., by the solid-liquid separation of the digestate. Separation with either screw press, belt press, or decanter centrifuges transfers most of the digestate volume into the liquid fraction, along with the water-soluble nutrients N and potassium (K) (Table 4, Hjorth et al., 2010). The solid fraction contains most of the digestate TS and P bound to the solid particles, and the decreased volume makes the solid digestate easier to handle and transport. The solid fraction can be also further dried, pelletized (reviewed in Möller and Müller, 2012), or composted (Teglia et al., 2011) to increase transportability and marketing value. The efficiency of the solid-liquid separation processes and the separation of P into the solid fraction is usually enhanced by adding chemicals or organic polymers to neutralize the particle charges, which affect the agglomeration capability of the particles (reviewed in Hjorth et al., 2010, Sheets et al., 2015).

The liquid digestate containing the majority of digestate N and K (Table 4) has, however, high water content and volume, as well as low nutrient concentrations (Camilleri-Rumbau et al., 2015, Hjorth et al., 2010, Zarebska et al., 2015). This complicates its usability in agriculture (Camilleri-Rumbau et al., 2015, Hjorth et al., 2010) by increasing application volumes and transportation costs (Chiumenti et al., 2013). In liquid digestates, the N is reported to be mainly (45–80%) in the soluble NH_4-N form (Möller and Müller, 2012), which is easily volatilized during liquid spreading (Nkoa, 2014). The digestate liquid can be further processed to remove water and simultaneously concentrate nutrients,

which have been reported to lower the environmental impact of digestate use and enable transportation to areas with nutrient deficits (Rehl and Müller, 2011). The combination of solid-liquid separation and digestate liquid treatment technologies is able to produce fertilizer products with optimal composition (Hjorth et al., 2010) to match the nutrient requirements of crops and achieve better control of the applied fertilizer.

Table 4. Mass and nutrient separation efficiencies from solid-liquid separation with decanter centrifuge and digestate liquid treatment with ammonia stripping, evaporation, and reverse osmosis.

Separation efficiency, %						Material	Scale	Reference
Mass	TS	N	NH_4-N	P	K			
Solid-liquid separation with decanter centrifuge; mass and nutrient separation % in liquid fraction								
-	31–46	69–76	-	9–48	-	D	full	Møller et al., 2002
91	36	72	89	10	99	DL_{pig}	full	Ledda et al., 2013
76	17	48	81	4	77	DL_{cow}	full	Ledda et al., 2013
22.5	-	87	-	19	33.5	M_{pig}	full	Melse and Verdoes, 2005
-	38–67	71–87	-	34–40	-	M_{pig}	full	Møller et al., 2002
-	35–45	51–73	-	18–22	-	M_{cow}	full	Møller et al., 2002
75–95	5-66	46–99	72–92	9–52	-	$M_{pig, cow}$	full	reviewed in Hjorth et al., 2010
Stripping; nutrient separation % in ammonium sulfate								
-	-	-	97	-	-	D	lab	Liu et al., 2015
-	-	-	>96	-	-	D	lab	Bonmatí and Flotats, 2003a
-	-	-	>80	-	-	D, M_{pig}	lab	Laureni et al., 2013
-	-	65–80	80–92.2	-	-	DL	pilot	Guštin and Marinšek-Logar, 2011
-	-	65–76	-	-	-	DL	full	Morales et al., 2013
-	-	94	-	-	-	U	pilot	Antonini et al., 2011
-	-	-	92	-	-	U	lab	Basakcilardan-Kabakci et al., 2007
-	-	-	65–98.8	-	-	M_{pig}	lab	Bonmatí and Flotats, 2003a
-	-	-	95	-	-	-	-	Flotats et al., 2011
Evaporation; mass and nutrient separation % in concentrate								
-	-	80–84	-	84–96	90–99	D	lab	Bonmatí and Flotats, 2003b
20.2	-	99.2	-	-	-	D	pilot	Chiumenti et al., 2013
10	-	-	-	-	-	U	-	Maurer et al., 2003
-	-	95	-	100	99	U	lab	Ek et al., 2006
-	-	95	-	100	100	S	lab	Ek et al., 2006
15–20	-	98	-	-	-	-	-	Flotats et al., 2011
Reverse osmosis; mass and nutrient separation % in retentate								
28	86–100	99.7	99.6	72	99.5	DL	full	Ledda et al., 2013
29	97–100	97	97	100	99	DL	full	Ledda et al., 2013
-	-	95	-	90	99	U	lab	Ek et al., 2006
-	-	90	-	92	97	S	lab	Ek et al., 2006
-	-	-	99.5	-	-	-	-	Ek et al., 2006
-	92.3	-	66	-	-	M_{pig}[a]	lab	Mondor et al., 2008

Digestates (D), digestate liquids (DL), manures (M), urine (U), and sewage reject water (S)
[a]after electrodialysis treatment
-, not available

The techniques for the digestate liquid treatment are, e.g., ammonia stripping and evaporation, which are able to produce nitrogen-rich liquid fertilizers. These technologies are already in full-scale use in AD plants across Europe, concentrated mainly in plants treating sewage sludge (e.g. Boehler et al., 2015, Flotats et al., 2011, Fuchs and Drosg, 2013). Additionally, with struvite precipitation, a solid fertilizer product can be produced from liquid streams (Ek et al., 2006, Mehta et al., 2015), and membrane technologies can be applied solely for digestate liquid treatment or as a combination with other treatments (reviewed in Zarebska et al., 2015). In ammonia stripping, the NH_4-N-rich liquid stream is stripped in increased pH and temperature, where the NH_4-N is transformed to NH_3 and around 65–99% of the soluble N can be further recovered, e.g., with H_2SO_4 in the form of ammonium sulfate ($(NH_4)_2SO_4$) during acid scrubbing (Table 4) (Fricke et al., 2007). The most used applications for ammonia stripping are packed-bed reactors, which provide a large contact area for air and liquid and ensure the rapid shift from liquid NH_4-N to gaseous NH_3 (reviewed in Guštin and Marinšek-Logar, 2011, Sheets et al., 2015). The produced ammonium sulfate can be further processed and used as mineral N fertilizer (Sheets et al., 2015); thus, the residue fraction still contains the remaining nutrients – K, P, and organic forms of N, which are not stripped as ammonia.

The evaporation process is based on the volume reduction of digestate liquid by heating (Fricke et al., 2007), which can be done in vacuum conditions to decrease the need for heat energy (Bonmatí and Flotats, 2003b, Chiumenti et al., 2013). With this technology, 80–98% of the digestate liquid N and nearly 100% of K and P is collected within the nutrient-rich concentrate (Table 4), and the residual fraction is collected as condensed water. Evaporator applications with either falling films or horizontal spraying films have been implemented in biogas plants (Fricke et al., 2007), and the NH_4-N volatilization is prevented with acid additions to conserve the N in the concentrate. Usually, the condensate water produced from evaporation (as well as the P and K containing stripping residue from stripping) are either discarded to wastewater treatment or post-treated (Bonmatí and Flotats, 2003b, Guštin and Marinšek-Logar, 2011, Ledda et al., 2013). Reverse osmosis in particular can be used to separate NH_4-N from liquid streams, where the liquid must be pretreated to remove solids to prevent membrane fouling (Sheets et al., 2015). Reverse osmosis is a membrane treatment along with microfiltration, ultrafiltration and nanofiltration (Camilleri-Rumbau et al., 2015, Mehta et al., 2015, Zarebska et al., 2015). These technologies are based on selective separation, e.g., of nutrients in semipermeable membranes. The permeability of nutrients is based on molecule size, reactivity and pressure (Mehta et al., 2015), and pH, which can be controlled to maintain a suitable NH_4^+/NH_3 ratio and increase the N recovery rate of the process (reviewed in Fricke et al., 2007, Sheets et al., 2015).

The digestate liquid treatment processes are both energy (and especially heat) consuming processes and require some amounts of chemicals, e.g., H_2SO_4 and/or NaOH (Fricke et al., 2007, Sheets et al., 2015), where for example, evaporation has been reported to consume300–350 kWh per ton of water evaporated (Fuchs and Drosg, 2013). The nutrient concentration methods, stripping, evaporation, and membrane technologies may also be able to concentrate unwanted heavy metals and salts, which

possibly hinder the use of these concentrated, nutrient-rich fractions in agriculture (reviewed in Mehta et al., 2015).

2.3 Food waste as feedstock for AD and digestate production

2.3.1 Anaerobic digestion of FW

The AD of FW has been studied due to its composition and characteristics, which lead to high CH_4 yields. In batch tests, FW feedstocks have produced 300–570 dm^3/kgVS of CH_4, while theoretical BMP (biochemical methane potential) estimations show the BMP values to be as high as 800 dm^3/kgVS (Table 5). The characteristics of the FW affect the CH_4 yields, where around 20–30% of the VS consist of fats and proteins, which yield higher amounts of CH_4 than carbohydrates (see Table 3, Table 5). However, the decrease in the measured BMPs compared with the theoretical estimations is related to the composition of undegradable and inert material, e.g. lignin, which lowers CH_4 production (Angelidaki and Sanders, 2004, Hendriks and Zeeman, 2009).

Table 5. The organic composition, measured methane yields in batch assays, and theoretical methane production of food wastes.

FW type	TS (%)	VS (%TS)	Fat (%VS)	Protein (%VS)	Carbo-hydrate (%VS)	$BMP_{measured}$ (dm^3/kgVS)	$BMP_{theoretical}$ (dm^3/kgVS)	Reference
Synthetic FW	23.7	91.1	17.2	10.6	71.5	414	-	Ariunbaatar et al., 2015
Synthetic FW	22.2	95.0	9.2	14.3	76.5	426	-	Ariunbaatar et al., 2014b
ss-FW	23.7	91.4	15.1	23.5	45.3	445–456	547[a] 494[b]	Zhang et al., 2012
ss-FW	17–37	81–92	8.1–16.6	8.1–16.6	29–55	298–573	583–834[a] 495–545[b]	Davidsson et al., 2007
ss-FW	23–33	83–93	12–15	13–15	-	428–487	530[a]	Hansen et al., 2007b
ss-FW	33	86.0	11.2	13.8	45.6	399	442[b]	Schott et al., 2013
ss-FW	30.9	85.3	-	-	-	435	-	Zhang et al., 2007
r-FW	23.8	89.2	-	-	-	372	618[a]	Zhang et al., 2015a
r-FW	18.1	94.5	13.6	19.2	65.3	480	-	Zhang et al., 2011
r-FW	19.9	90.5	24.2	16.8	59.1	538	-	Liu et al., 2012

Food waste (FW), source separated (ss), restaurant (r)
[a]theoretical BMP based on element composition (C, H, N, O) according to Buswell's equation
[b]theoretical BMP based on component composition (fat, protein, carbohydrate)
-, not available

In continuous meso- and thermophilic AD, FWs have yielded around 350–500 dm^3/kgVS of CH_4. In full-scale operation, high OLRs and shortened HRTs are pursued to decrease the AD plant size and to improve energy efficiency through increased CH_4 production (Ferrer et al., 2010, Qiao et al., 2013). However, in the mesophilic digestion of FW, OLRs are usually maintained as relatively low, because the digestion of FW at higher OLRs has often proven unstable (Table 6). For example, after OLR increases to 2 and 3 kgVS/m^3d, FW digesters have started to accumulate VFAs, which has led to ceased CH_4 production (Banks et al., 2008, Zhang and Jahng, 2012). In some studies, inhibition has been observed at higher OLRs of around 5–6 kgVS/m^3d (Nagao et al., 2012, Zhang et al., 2015a). The unstable operation has been attributed to ammonia inhibition resulting from high protein content (Gallert and Winter, 1997), which is often indicated by the accumulation of VFAs (Banks et al., 2012).

Especially in FW digesters operating long-term, the imbalanced VFA production has been connected with TE deficiencies, which affect the process stability and lower CH_4 production, (Banks et al., 2008, Banks et al., 2012, Facchin et al., 2013, Yirong et al., 2015, Zhang et al., 2011, Zhang and Jahng, 2012, Zhang et al., 2012). After the introduction of TE supplementation to the digesters, stable operation on higher OLRs (around 7 kgVS/m^3d) has been possible (Table 6). At the beginning of the digestion, the background concentration of TEs from the inoculum provides sufficient TE levels for FW digestion (Facchin et al., 2013), but the effect of inoculum has been reported to decrease over time (Zhang et al., 2015a) due to the washout of the TEs. The most widely studied TEs in FW digestion studies are iron (Fe), copper (Cu), zinc (Zn), manganese (Mn), cobalt (Co), molybdenum (Mo), nickel (Ni), selenium (Se), tungsten (W), and boron (B), of which especially Fe (Zhang and Jahng, 2012), Mo, W (Facchin et al., 2013), Co (Banks et al., 2012, Facchin et al., 2013) and Se (Banks et al., 2012, Zhang et al., 2015b) have been reported to stabilize VFA concentrations and increase the CH_4 production of mesophilic digesters treating FW. In addition, the combination of TEs has been reported to show synergistic effects on the digestion of FW (Banks et al., 2012, Facchin et al., 2013, Zhang et al., 2011, Zhang et al., 2015a).

Table 6. The operational parameters and possible inhibition and trace element (TE) additions in different laboratory food waste digesters.

FW type	OLR ($kgVS/m^3d$)	HRT (d)	CH_4 yield ($dm^3/kgVS$)	VS reduction (%)	TAN in reactor (g/L)	Inhibition observed	TE additions	Reference
Mesophilic digestion								
r-FW	2.19	30	-	75–80	-	yes, after 70 days	no	Zhang and Jahng, 2012
r-FW	2.19–6.6	20–30	350–450	75–80	1.3–2.7	no	Co, Mo, Ni, Fe	Zhang and Jahng, 2012
FW	3.7–5.5	8	420	85–89	n.a.	yes, after day 50 (OLR 5.5)	no	Nagao et al., 2012
FW	3.7–12.9	16	420–450	84–93	-	yes, after OLR 9.2	no	Nagao et al., 2012
r-FW	2–6	15–30	390–420 (OLR 2–4) 360–0 (OLR 6)	30–70	-	yes, after day 45 (OLR 6)	no	Zhang et al., 2015a
r-FW	2–7	15–31	460–500	70–80	-	no	mixtures of Fe, Co, Mo, Ni + chelating agent	Zhang et al., 2015a
ss-FW	2–5	38–95	-	-	5–6	yes, after OLR 3	no	Banks et al., 2012
ss-FW	2–5	38–95	-	-	5	no	mixtures of Al, B, Co, Cu, Fe, Mn, Ni, Zn, Mo, Se, W	Banks et al., 2012
FW	3.8	-	380	96	-	no	no	Grimberg et al., 2015
KW+DAS+VFR	10.3–12.9	8–10	500–800	60–64	-	yes	no	Guo et al., 2014
diluted FW	9.4	19	-	64	1	yes	no	Gallert and Winter, 1997
Thermophilic digestion								
diluted FW	9.4	19	-	65	1.3	yes	no	Gallert and Winter, 1997
ss-FW	1–4	-	430	-	2–6	yes, after day 70	no	Yirong et al., 2015
ss-FW	1–4	-	430	-	2–6	yes	mixtures of Al, B, Co, Cu, Fe, Mn, Ni, Zn, Mo, Se, W	Yirong et al., 2015
KW	0.5–3[b]	18–36	-	-	-	yes, after day 80	no	Ma et al., 2011
ss-FWs	2.8	-	275–410	81	-	no	no	Davidsson et al., 2007

Food waste (FW), source separated (ss), restaurant (r), kitchen waste (KW), vegetable/fruit residue (VFR), dewatered activated sludge (DAS),
[a]pilot experiment, [b]gCOD/L d, [c]biogas productivity (L/L/d)
-, not available

2.3.2 Pretreatment of food waste

Prior to utilization, e.g., in AD plant FW undergoes pretreatment steps, which usually seek to improve the FW quality and digestion process through separation of unwanted materials, particle size reduction, and solubilization (Bernstad et al., 2013, Carlsson et al., 2012). The reduction of particle size increases the surface area available for microbial degradation (reviewed in Ariunbaatar et al., 2014a, Izumi et al., 2010); thus, excess size reduction has been shown to negatively affect the AD of FW by decreasing solubility and by VFA accumulation, which subsequently led to decreased CH_4 production (Izumi et al., 2010). Although the separation of unwanted materials, such as plastic, glass, and metals from FW is a common practice in full-scale digestion plants, the separation process can reduce some FW and its nutrients and organics, which reduces the potential CH_4 yield of the FW (Bernstad et al., 2013, Davidsson et al., 2007).

Heat-based pretreatments are recommended within national regulations for the hygienization of organic wastes, where the thermal treatment leads to pathogen removal. According to the EU's Animal By-Products Regulation (1096/2009/EC, European Parliament and the Council, 2009), materials from category 3, such as FW, must be hygienized (70°C, 1 h), while category 2 materials (e.g., Salmonella-contaminated slaughterhouse materials) must be pressure sterilized (>133°C, 20 min) before digestion to reduce the risk to public and animal health. Low-temperature hygienization is commonplace at AD plants to pretreat FW, but thermal and hydrothermal treatments have also been used as pretreatments before AD to enhance the solubilization and biodegradability of, e.g., FWs and mixed biowastes in higher temperatures (Lissens et al., 2004, Ren et al., 2006, Sawayama et al., 1997). Thermal treatments of FW with autoclaves in temperatures 120–200°C have been previously studied, where increased CH_4 yields are observed in the lower temperature range. Recently, pretreatment temperatures from 140 to 175°C have been observed to lower the biodegradability and CH_4 conversion of FW (Table 7). This decreased CH_4 production is connected with the formation of Maillard compounds, as the FW includes a high proportion of amino acids containing proteins and easily biodegradable carbohydrates. Additionally, due to these characteristics, the formation of Maillard compounds in FW has been proposed to occur in lower temperatures compared with sludges (Guo et al., 2014, Liu et al., 2012), where the formation of Maillard-like compounds has been reported in higher temperatures (190°C, Bougrier et al., 2008).

Table 7. The effect of thermal and hydrothermal pretreatment on the AD of food wastes in temperatures 120–200°C.

Temper-ature (°C)	Material	Pressu-re (bar)	Time (min)	Change in CH_4 production (%)	Apparatus/ technique	AD type	Reference
120	sFW	-	60	+13.5	oven	batch	Ariunbaatar et al., 2014b
120	KW	-	30	+10[a]	autoclave	batch	Ma et al., 2011
140	sFW	-	60	+1	oven	batch	Ariunbaatar et al., 2014b
140	sFW	-	30	+4	oven	batch	Ariunbaatar et al., 2014b
150	sFW	-	30	+3.6	oven	batch	Ariunbaatar et al., 2014b
140–160	KW	-	50–70	-0.2	thermal hydrolysis	batch	Li and Jin, 2015
170–175	KW+ VFR	5	60	+3	thermal hydrolysis	batch	Zhou et al., 2013
175	KW	-	60	-7.9	oil bath	batch	Liu et al., 2012
175	VFR	-	60	-11.9	oil bath	batch	Liu et al., 2012
175	KW+VFR +DAS	-	60	-3.6[a]	thermal hydrolysis	conti-nuous	Guo et al., 2014
175	sFW	40	60	+30	autoclave	batch	Sawayama et al., 1997
185	FW	12	10	+7	autoclave	batch	Lissens et al., 2004
160–200	FW	40	60	+6	thermal hydrolysis	batch	Schieder et al., 2000

Food waste (FW), kitchen waste (KW), vegetable/fruit residue (VFR), dewatered activated sludge (DAS), synthetic (s)
[a]biogas production
-, not available

2.3.3 Food waste digestate

Agronomic value

The agronomic value of FW digestate, like all digestates, can be divided into four components: i) nutritional value to plants and organic matter content, ii) content of contaminants, iii) content of, e.g., heavy metals, and iv) hygienic quality (Abubaker et al., 2012, Nkoa, 2014, Teglia et al., 2011). The digestate nutrient content is dependent on the substrate (Abubaker et al., 2012, Haraldsen et al., 2011, Tambone et al., 2010), and with FW feedstock, the amount of plant nutrients is usually high due to the waste's characteristics. Additionally, the digestate characteristics are also dependent on the AD process; the reactor type and process parameters (Trzcinski and Stuckey, 2011, Zirkler et al., 2014) affect the TS content, for example (Table 8). The TS content in FW digestates from wet-type AD reactors is usually around 6%, while in liquid-type digestates and solid digestates from different solid-liquid separation processes, the TS content is around 2% and 25%, respectively (Table 8, Hjorth et al., 2010). The TS content of the digestate affects the transportation needs and costs, especially over longer distances (Rehl and Müller, 2011, Teglia et al., 2011). With FWs, the AD plants are usually located far from agricultural lands where the nutrients could be utilized (Babson et al., 2013), which increases the transportation distance of the digestate nutrients.

The nutrient content in FW digestates varies depending on the FW type, digestion process, and TS content, where the TKN content is usually between 2 and 7 kg/kgFM, P_{tot} content ranges from 0.5–1 g/kgFM, and K_{tot} is around 1 g/kgFM. The NH_4-N/TKN ratio in FW digestates is over 50% in general, which indicates increased fertilizer potential (reviewed in Nkoa, 2014, Teglia et al., 2011). The FW digestate can also contain contaminants (e.g. glass and metals) that are correlated with FW quality and sorting processes (Bernstad and la Cour Jansen, 2012). The inorganic impurities in the digestate, such as heavy metals, can originate from food production, when soils, crops and animal feeds already contain certain levels of heavy metals. During household waste collection heavy metal-containing batteries, metal containers, etc. can end up in the food waste stream if they are not removed during waste pretreatment. Subsequently, the amount of heavy metals ending up in soils is dependent on the application volumes of digestates, which are dependent on the TS and nutrient concentrations.

The hygienic quality of the FW digestates is dependent on the pathogens; the species usually found in the FW feedstock are, e.g., *Salmonella, Listeria, Escherichia coli, Campylobacter, Mycobacteria, Clostridia* and *Yersinia* (reviewed in Sahlström, 2003). The most abundant pathogens are usually enterococci and coliforms, whose concentration is around 10^4–10^5 cfu/g in FW (Sahlström et al., 2008). The inactivation pathogen in thermophilic processes (50–55°C) is calculated in hours but with mesophilic temperatures (30–38°C) in days (reviewed Sahlström, 2003). Thus, according to the European Animal By-Products Regulation, when food waste is used as a digester feedstock, pre- or post-hygienization is required to prevent risk against human and animal health (1069/2009/EC, European Parliament and the Council, 2009). Within the legislation, the threshold value for *E. coli* or Enterococcaceae is 1000 cfu/g and no Salmonella in a 25 g sample.

Table 8. Food waste digestate characteristics reported in the literature.

Feedstock	pH	TS (%)	VS (%TS)	C/N	TKN (g/kgFM)	NH_4-N (g/kgFM)	NH_4-N /TKN (%)	P_{tot} (g/kgFM)	K_{tot} (g/kgFM)	Reference
Whole digestates										
FW	7.9–8.2	4.8–5.4	66.4–69.3	2.3–2.7	7.6–8.1	4.4–4.7	54–62	-	-	Pezzolla et al., 2012
FW	8.3	18.4	75	15.5	28	2.0	7	0.2	-	Forster-Carneiro et al., 2008
sh-OFMSW	8.9	7.8	47.4	6.1	45	1.9	4	0.005	-	Forster-Carneiro et al., 2008
OFMSW	8.5	3.7	61.6	7.6	47	1.2	3	0.004	-	Forster-Carneiro et al., 2008
60% ss-h-FW, 40% slaughterhouse waste	7.9	6.1	-	7.1	7.9	5.3	67	0.9	1.6	Abubaker et al., 2012, Abubaker et al., 2015
66% ss-h-FW, 24% silage, 10% grease tap sludge	8.7	5.9	-	12.1	5.3	3.3	62	0.4	3.7	Abubaker et al., 2012, Abubaker et al., 2015
24% ss-h-FW, 43% food processing waste, 33% slaughterhouse waste	8	1.7	-	11	2.6	2.0	77	0.2	1.1	Abubaker et al., 2012, Abubaker et al., 2015
Liquid digestates										
ss-h-FW	8.02	1.45	-		2.2	1.5	68	0.2	1.1	Haraldsen et al., 2011
ss-h-FW		0.7	-		1.3	0.4	30	0.06	1.3	Haraldsen et al., 2011
FW	4.4	17.1	86	14	6.0	0.5	8	0.7	0.9	Rigby and Smith, 2013
FW+abattoir+farm waste	8.1–8.3	5.0–5.2	67.1–72.7	-	5.5–7.2	2.8–3.9	39–71	0.2–0.4	1.5–2.7	Rigby and Smith, 2014
ss-FW	-	5.8	71.2	-	6.5	3.8	58	0.7	2.7	Zhang et al., 2012
mr-OFMSW	-	6.6	49.9	-	3.2	1.5	47	0.3	1.1	Zhang et al., 2012
Solid digestates										
OFMSW	8.8	28.9	42.3	11	6.7	1.0	15	1.3	1.5	Rigby and Smith, 2013
OFMSW	7.3–7.6	34–37.2	52.3–56.6	-	5.1–6.3	0.7–1.2	11–24	1.0–1.5	1.1–1.8	Rigby and Smith, 2014
ss-FW	-	14.7	82.6	-	8.0	3.5	44	1.5	2.6	Zhang et al., 2012
mr-OFMSW	-	35	60.5	-	5.7	1.7	30	1.2	1.4	Zhang et al., 2012

Food waste (FW), organic fraction of municipal solid waste (OFMSW), source separated (ss), household (h), mechanically recovered (mr)
-, not available

Fertilizer use

The application of the same amount of plant-available nutrients in FW-based digestates compared to mineral fertilizers has been found to produce similar and even up to 40% increased crop yields (Table 9, Abubaker et al., 2012, Furukawa and Hasegawa, 2006, Haraldsen et al., 2011, Rigby and Smith, 2014). These effects are due to both nutrient content – especially the amount of mineralized N (Fouda et al., 2013, Teglia et al., 2011) – and the introduction of organic matter to soils, increasing the soil structure and microbiology (Odlare et al., 2008, Odlare et al., 2011). Some studies have, however, reported 10–25% lower crop yields with FW digestates compared to mineral fertilizers (Odlare et al., 2008, Svensson et al., 2004), as the plant-available N concentrations have not been able to compete with the mineral fertilizers. However, the digestate produced from FW could be used with mineral fertilizer supplementation to match the crop nutrient demand (Nicoletto et al., 2014, Svensson et al., 2004), or it could be post-treated to produce concentrated fertilizer products for agriculture with optimal nutrient ratios for plant growth (Hjorth et al., 2010).

The results from various FW digestate fertilization experiments are, however, affected by the experiment conditions, e.g., soil quality, which can lead to different N mineralization in soils (Rigby and Smith, 2013), and further reduce the availability of nutrients. Digestate fertilization could have alternative effects on plants, e.g., the plant's nutritional composition. Fertilization with fruit and distillery waste-based digestates has been reported to decrease the nitrate (NO_3-N) content in vegetables, especially in lettuce (Nicoletto et al., 2014), where the predominant form of N in digestates is NH_4-N, which does not accumulate into plants as the NO_3-N form in the mineral fertilizers does.

Table 9. The effect of food waste digestate fertilization on crops in field- and pot-scale experiments.

Digestate origin	Plant/crop	Type of experiment	Effect of digestate fertilization	Reference
Slaughterhouse waste + FW	Spring wheat	Pot experiment	1–40% higher biomass yield compared with mineral fertilizer, lower yield compared with pig slurry[b]	Abubaker et al., 2012
FW+abattoir+ farm waste[a]	Ryegrass	2-year field trial	13–23% increased dry matter yield and plant N uptake compared with mineral control[c], effective source of available N	Rigby and Smith, 2014
FW[a]	Barley	Pot experiment	Equivalent grain yield with mineral NPK fertilizer[b]	Haraldsen et al., 2011
FW	Spinach and Komatsuna	Field trial	Comparable yield and N uptake compared with mineral fertilizer	Furukawa and Hasegawa, 2006
OFMSW	Spinach	Field trial	21% increased biomass yield and plant growth compared with control with no fertilization	Islam et al., 2016
FW	Crop rotation, barley and oats	4-year field trial	7–26% lower N yields and 19% lower crop yields compared with mineral fertilizer control. Similar crop yields were achieved with digestate supplemented with mineral fertilizer[b]. Digestates introduced more plant-available N and promoted soil microbial activity compared to mineral fertilizers and manure	Odlare et al., 2008, Svensson et al., 2004
FW	Crop rotation, barley and oats	8-year field trial	15% lower biomass yield compared with mineral fertilizers. Higher yield compared with unamended and compost-amended plots. Substrate induced respiration, potential ammonium oxidation, and N mineralization were improved[b]	Odlare et al., 2011

Food waste (FW), organic fraction of municipal solid waste (OFMSW)
[a] Liquid digestate, [b]equal N in digestates and mineral fertilizer control, [c]not equal N fertilization rate in digestates and control

3 Objectives

The main aim of this study was to analyze the potential of using anaerobic digestion for FW utilization. To specify the aim, the anaerobic digestion of FW and the processing and utilization of digestate was studied using four sub-objectives (Figure 5):

1. To increase the FW treatment rate in mesophilic AD and achieve stable AD performance at high OLRs (Paper I).

2. To study the effect of autoclave pretreatment on the AD performance (Papers I and II), as well as the digestate quality and agronomic usability (Papers II and III).

3. To evaluate the agricultural usability of different FW digestates (Papers II and III).

4. To evaluate the mass and nutrient flows and energy balance of digestate liquid post-treatment in order to assess the feasibility of producing concentrated nutrient products (Paper IV).

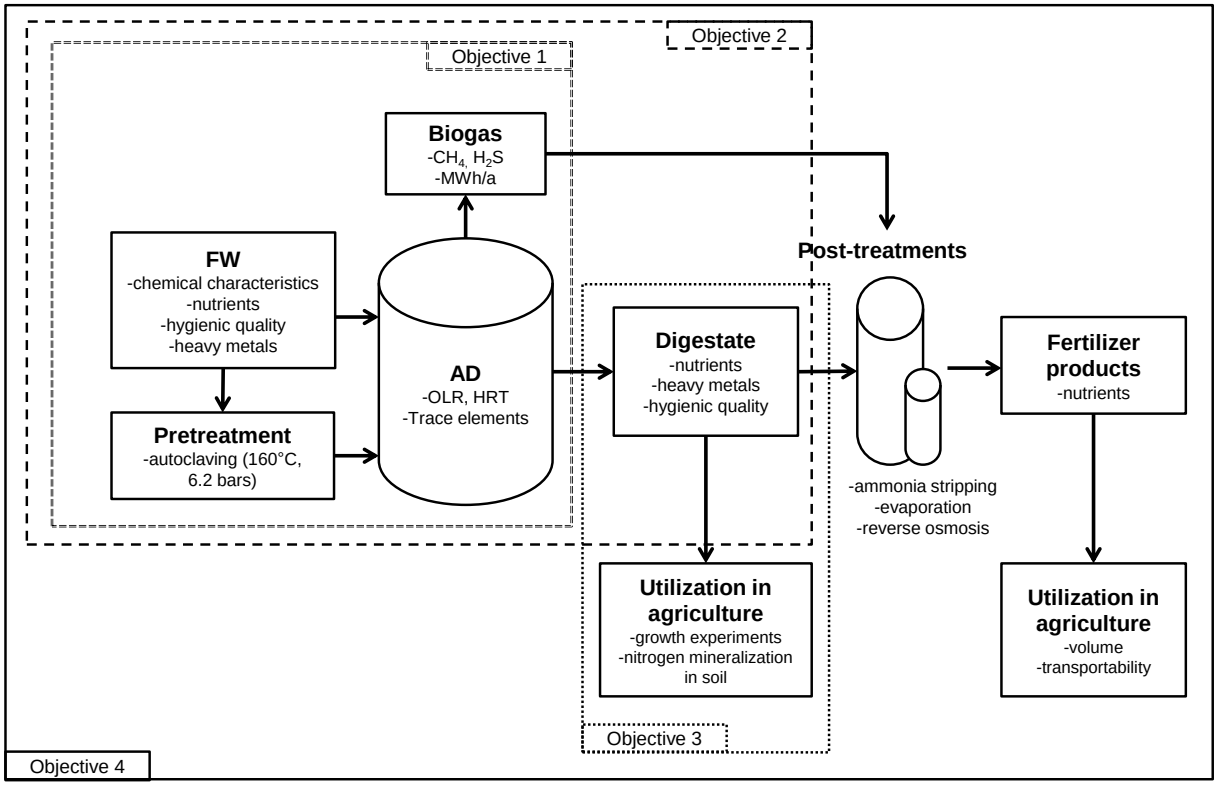

Figure 5. The overview of the thesis objectives.

4 Materials and methods

This study analyzed the utilization FW as a feedstock for AD, the pretreatment with autoclaving, and the quality and usability of the produced digestates. The AD of FW and autoclaved FW was studied with BMP assays in batch mode and continuous experiments in stirred tank reactors (STRs). The characteristics of the feedstocks and the digestate quality were examined with chemical analyses as well as hygiene indicator analyses. The usability of the digestate in agriculture was tested with growth experiments and N mineralization tests. On the whole, the mass, nutrient, and energy balances were calculated for a theoretical full-scale FW digester and the digestate post-treatment processes to assess the feasibility of producing concentrated nutrient products (Table 10).

Table 10. The summary of the thesis objectives, experiments and analyses conducted in each corresponding paper.

Objective	Studied materials	Experiments	Main analyses	Paper
1. Increase of the FW treatment rate in mesophilic AD to achieve stable AD performance at high OLRs	FW	STRs, BMPs	Chemical characteristics	I, II
2. Effect of autoclave pretreatment on the AD performance, digestate quality and agronomic usability	FW	STRs, BMPs, ammonification trials	Chemical characteristics	I, II
3. Agricultural usability of different FW digestates	FW, OFMSW, VWAS	Growth experiments, N mineralization	Chemical characteristics, nutrients, heavy metals, hygienic quality	II, III
4. Mass, nutrient, and energy balance of digestate liquid post-treatment in order to assess the feasibility of producing concentrated nutrient products	FW digestate	Theoretical mass, nutrient and energy balance calculations		IV

Food waste (FW), organic fraction of municipal solid waste (OFMSW), vegetable waste + waste activated sludge (VWAS), stirred tank reactor (STR), biochemical methane potential (BMP)

4.1 Materials

4.1.1 Food wastes and digestates

This study characterized three FWs (FW1–FW3) and one OFMSW, their representative digestates, as well as one digestate with a mixture of vegetable waste (VW) and WAS as feedstock (sample referred to as VWAS). These digestates originated from laboratory stirred tank reactors (Paper I and II) and sub-commercial, full-scale and pilot-scale reactors (Paper III). The studied digestates and their representative FWs/feedstocks are summarized in Table 11. The characteristics, including nutrient content (Papers II, III) and hygienic quality (Paper II), were analyzed from both feedstocks and digestates, while heavy metal content (Paper III) and plant growth and N mineralization (Paper III) were analyzed solely from the digestates. Additionally, digestates FW1 and FW2 were tested considering their residual methane potential (RMP) and the ammonification potential (Paper II).

FW1 and FW2 were based on source separated domestic FW collected from the South Shropshire biowaste digestion plant in Ludlow, UK. Biodegradable bags (used for waste collection) were removed and the FW material was mixed and divided into two equal portions. One portion was left untreated (sample referred to as FW1), while the other portion was pretreated for 45 minutes at 160°C and 6.2 bars in a novel double-auger autoclave (AeroThermal Group Ltd, UK) (sample referred to as FW2). Both portions were then passed through a macerating grinder (S52/010 Waste Disposer, IMC Limited, UK), packed into 35-liter plastic boxes, frozen, and shipped at -20°C to the Natural Resources Institute, Finland, to be used in AD experiments to produce digestates FW1 and FW2. Before the experiments and analyses, the FWs were thawed and stored at 4°C (Papers I, II, and III), while the representative digestates FW1 and FW2 were stored at -20°C, then thawed and stored at 4°C before analyses and experiments.

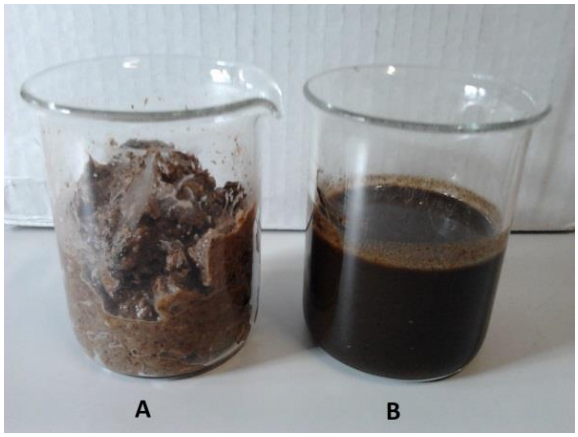

Figure 6. The untreated FW1 (A) and the autoclaved FW2 (B) characterized in this study.

The studied FW3 originated from Greenfinch, UK, and was source separated food waste. Its representative digestate was obtained from a sub-commercial-scale anaerobic digester (Greenfinch, UK). Source separated OFMSW from the Lisbon area, Portugal, was analyzed along with its digestate from a full-scale AD plant (Lisbon, Portugal). The OFMSW feedstock showed rather different composition compared to other FWs, and prior to analyses of water-soluble nutrients and carbon, the non-biodegradable material (plastic cups, plastic bags, etc.) were manually removed. Prior to analyses, all feedstock samples were macerated with a Retch Grindomix GM300 knife mill (Retch Gmbh, Germany). The fifth digestate, VWAS, was obtained from a pilot digester treating a mixture of VW and WAS from Treviso, Italy. The feedstock representative of this digestate was not available for characterization. All waste and digestate samples were sent in frozen form to a laboratory at the Natural Resources Institute, Finland, where the samples were thawed and stored approximately one week at 4°C (Paper III).

Overall, the studied digestates originated from different phases and scales of the AD (Table 11). For FW1 and FW2 digestates, the characterization analyses were done from digestates from OLRs 2–6 kgVS/m^{3}d (Papers II and III), hygiene and RMP analyses from OLRs 4 and 6 kgVS/m^{3}d (Paper II), and growth and mineralization experiments from OLR 4 kgVS/m^{3}d (Paper III).

Table 11. The feedstocks and digestates studied in this thesis from different AD reactors.

Sample	Feedstock	Origin	Digestion parameters					Paper
			Scale	Temperature	Phase	HRT (d)	OLR (kgVS/m^{3}d)	
FW1	ss-FW	UK	Laboratory	Mesophilic	1	117	2.0	I,II
						78	3.0	I,II
						58	4.0	I, II, III
						39	6.0	I,II
FW2[a]	ss-FW	UK	Laboratory	Mesophilic	1	94	2.0	I,II
						63	3.0	I,II
						47	4.0	I, II, III
						31	6.0	I,II
FW3	ss-FW	UK	Sub-commercial	Mesophilic	1	26	3.3	III
OFMSW	OFMSW	Portugal	Full scale	Thermophilic	2	24	3.7[c]	III
VWAS	VW+WAS[b]	Italy	Pilot	Thermophilic	1	16	3.8[c]	III

Food waste (FW), organic fraction of municipal solid waste (OFMSW), vegetable waste + waste activated sludge (VWAS), vegetable waste (VW), waste activated sludge (WAS), source separated (ss)
[a]Feedstock pretreated with autoclave (160°C, 6.2 bar)
[b]Feedstock was not characterized
[c]kgCOD/m^{3}day

4.1.2 Inocula

Different inocula were used in different experiments in this study (Table 12). The STRs were inoculated with digestate from a mesophilic AD digesting mechanically dewatered sewage sludge (Biovakka Suomi Ltd, Turku, Finland) (Paper I). In BMP and RMP assays, the inoculum was taken from an AD treating municipal and industrial biowastes (Envor Biotech Ltd, Forssa, Finland) (Paper I, II). The digestates obtained from the laboratory-scale STRs were used as inocula to test the BMP and RMP with these digestates (Paper II).

Table 12. The characteristics of the mesophilic inocula used in this study in stirred tank reactor (STR), biochemical methane potential (BMP), and residual methane potential (RMP) experiments.

Inoculum	TS (%)	VS (%)	VS/TS (%)	TKN (g/kgFM)	NH_4-N (g/kgFM)	Experiment	Paper
Full-scale sewage sludge digester	7.7	4.3	55.8	4.9	2.4	STR	I
Full-scale biowaste digester	4.5	2.9	63.2	-	-	BMP, RMP	I,II
Laboratory-scale reactor treating FW1	6.6	4.5	67.3	8.0	4.0	BMP, RMP	II
Laboratory-scale reactor treating FW2	8.2	6.7	81.2	7.3	1.2	BMP, RMP	II

-, not available

4.2 Experimental set-up

4.2.1 Anaerobic digestion

Batch assays

Two types of batch assays were executed in this study: BMP and RMP assays (Papers I and II). Batch assays were used to obtain the BMP values for FWs and digestates (Papers I and II), as well as to study the ammonification potential of FW digestates (Paper II). All assays were performed at 37°C using automated testing equipment (Bioprocess Control Ltd, Sweden) (Figure 7). The assays were mixed mechanically (84 rpm) for one minute per hour. From the biogas, CO_2 was absorbed by NaOH before the automated CH_4 gas volume measurement, which was based on liquid displacement. Assays were conducted in 500 ml glass bottles as duplicates or triplicates, each with a total liquid volume of 400 ml (BMPs in Paper I, BMPs and RMPs in Paper II) or 200 ml (RMP assays in Paper I). The inoculum to substrate ratio in BMP assays was 1:1 on a VS basis (Papers I and II), except in the FW2 sample, which was digested in a VS ratio of 1:2 (Paper II). The digestates in the RMP assays were incubated without inoculum. In all BMP assays, distilled water was added to obtain 400 ml liquid volume. If pH was lower than 7.5, it was adjusted to around 8 with 3 M NaOH. $NaHCO_3$ (3 g/l) was used as a buffer in BMPs and RMPs (Paper I). Finally, the contents of all bottles were flushed with N_2 to obtain anaerobic conditions.

Continuous reactor experiments

The continuous AD was studied with samples FW1 and FW2 in four 11-liter stainless steel STRs (Metener Ltd, Finland) (Figure 7), which operated at 37°C with semi-continuous stirring (32 rpm, 5 seconds on and 60 seconds off). Two parallel reactors were fed with FW1 (R1, R2) and two reactors with FW2 (R3, R4). The reactors were manually fed five times a week through an inlet tube, which extended below the digestate surface and was also used for digestate sampling. Digestate overflowed from the reactors by gravity through a u-tube trap to prevent gas escape. The gas volume and CH_4 content were measured through two methods: i) using an automatic system where the produced biogas was collected into a small (~220 ml) gas storage vessel on top of the reactor, and ii) by water displacement in a volume-calibrated cylindrical gas collector, after which the gas was collected in aluminum gas bags (Paper I).

Figure 7. The laboratory stirred tank reactors (left) and BMP devices (right) used in this study.

During the 473 days of experimentation, OLRs were gradually increased from 2 to 6 kgVS/m^3day (Table 13). During days 179–193 reactors R1 and R3 were supplemented once a week with a trace element (TE) solution containing Se (0.2 mg/l) and Co (1.0 mg/l). From day 199 onward, all reactors (R1–R4) were given a weekly supplement of two TE solutions (Banks et al., 2012), one containing cation elements (mg/l) – Al 0.1 , B 0.1, Co 1.0, Cu 0.1, Fe 5.0, Mn 1.0, Ni 1.0, Zn 0.2 – and the other oxyanions (mg/l): Mo 0.2, Se 0.2, and W 0.2. For each kg of digestate removed from the reactors over the one-week period, 1 ml of each of these TE solutions was added.

Table 13. The scheme of increasing OLRs and HRTs in continuous experiments (Paper I).

OLR (kgVS/m^3d)	2	3	4	6
Days	19–150	151–255	256–417	418–473
HRT (d)				
FW1 (untreated FW)	117	78	58	39
FW2 (autoclaved FW)	94	63	47	31

4.2.2 Digestate nitrogen usability in soil and plants

The agricultural usability of the studied digestates was assessed with nitrogen mineralization in soil and plant growth tests. N mineralization tests were conducted to study the effect of digestate applications on soil inorganic N concentrations (Paper III). The 48-day mineralization was tested in triplicates 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 digestates were added to the soil (2.2–8.6 gFM), resulting in N additions of 17–31 mgN/100g soil. 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 NH_4-N and NO_3-N. Soil inorganic N concentrations were compared against the incubated control soil.

The plant availability of the N in the digestates was studied via a pot experiment using the same soil as in the mineralization test (Paper III). The growth of Italian ryegrass (cv. Fabio) was studied in triplicate treatments with each of the digestates and a control. The applied N addition varied from 1280 to 2390 mgN/pot within the digestates. Control treatments were mineral fertilizer (NH_4NO_3) applications of 0 to 2000 mgN into the pot at 500 mg N intervals. Sufficient levels of P (500 mgP/pot), K (1500 mgK/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 were mixed with the soil of each pot to control pH and add Ca. A half gram of ryegrass seeds was evenly placed on the surface of the experimental soil in each pot. The 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 of light at 16°C and 10 hours of dark at 14°C). The grass was harvested at 30, 60, and 160 days after the start of the experiment. When harvested, the ryegrass was cut, leaving 2 cm-high stubble; the fresh weight was measured, and samples were dried at 60°C after which the dry weight (dry matter, DM) was determined. Samples were milled before analyzing the TKN concentrations.

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 (mgN/pot).

4.3 Mass, nutrient, and energy balances of a theoretical AD plant

The mass, nutrient, and energy balance of a theoretical full-scale AD plant digesting FW was calculated to evaluate the digestate liquid post-treatment and assess the feasibility of producing concentrated nutrient products. The aim was also to compare the nutrient concentration and energy efficiency of digestate liquid treatment technologies (Paper IV). Figure 8 presents the applied AD plant system boundaries, including pretreatment, a digester, digestate treatment and biogas upgrading. The formed biogas was assumed to be upgraded in a CHP into heat and electricity to be used in the AD plant, and the excess electricity was to be fed to the power grid (Table 14). The digestate treatment was assumed to include the separation of the digestate into liquid and solid digestates, from which the liquid digestate would be further treated with one of the four treatment systems consisting combinations of ammonia stripping, evaporation and membrane (reverse osmosis, RO) technologies (Table 15).

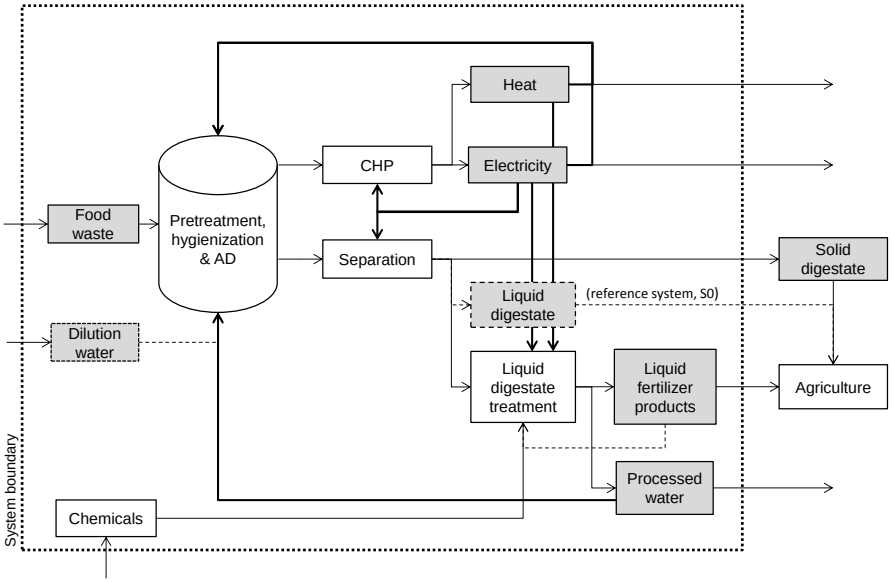

Figure 8. The system boundaries for the theoretical AD plant. Gray boxes represent feedstock/product and white boxes represent studied unit operations.

4.3.1 Anaerobic digestion and digestate liquid treatment

The AD feed was source separated FW with TS 25%, VS 23%, N_{tot} 7.5 kg/tFM, NH_4-N 0.4 kg/tFM, P_{tot} 0.9 kg/tFM, and K_{tot} 2.8 kg/tFM (based on Papers I and II). The FW (60 kt/a) was assumed to be pretreated by shredding/maceration and then hygienized (1 h at 70°C). The amount of thermal energy needed for heating 60 kt of FW during hygienization was calculated with the specific heat capacity of water (4.18 kJ/kg°C). No additional heating was allocated for the heating of FW prior to the digester as the heat energy from the hygienization was assumed to be sufficient for the mesophilic (40°C) digester

(Berglund and Börjesson, 2006, Prapaspongsa et al., 2010). The heating of the dilution water was calculated using the specific heat capacity of water. The references for the calculation of the AD energy consumption are presented in Table 14 in addition to the heat losses from the hygienization and digester units. Pretreatment and hygienization were not considered to affect the FW mass and nutrient content as material was not removed during the pretreatment step. During the maceration step, 40 kt/a of dilution water was added to the FW to achieve a TS of 15%.

Table 14. The literature values used in the calculations concerning the mass, nutrient and energy balance of a theoretical AD plant treating FW.

Process	Value/calculation	Reference
Hygienization & pretreatment		
Heat consumption	FW (60 kt) temperature from 15 to 75°C to maintain the required temperature during hygienization	-
Electricity consumption	37.5 kWh/tFM for hygienization and pretreatment	reviewed in Pöschl et al., 2010
Digester		
Heat consumption	The heat energy from the hygienization was assumed to be sufficient for the mesophilic (40°C) digester. The heating of the dilution water (40 kt/a) from 15 to 40°C was calculated with the specific heat capacity of water	-
Electricity consumption	18 kWh/tFM	reviewed in Berglund and Börjesson, 2006, Pöschl et al., 2010
Heat loss	15% of the heat demand	Rapport et al., 2011, Smyth et al., 2009
CHP unit		
CHP efficiency	38% for electricity and 48% for heat	Bacenetti et al., 2013, Poeschl et al., 2012
Electricity consumption	5% of the energy produced in CHP	Banks et al., 2011, Havukainen et al., 2014, Naegele et al., 2012, Pöschl et al., 2010
Digestate separation		
Electricity consumption	3.5 kWh/tFM digestate	Flotats et al., 2011, Hjorth et al., 2010, Ledda et al., 2013, Møller et al., 2000, Møller et al., 2002
Nutrient separation efficiency	In liquid digestate: 90% mass, 20% TS, 20% VS, 70% N_{tot}, 81% NH_4-N, 10%P_{tot}, 85% K_{tot}	

-, not applicable

The energy content (MWh/a) of the produced biogas was calculated by multiplying the BMP of the FW (450 m^3CH_4/tVS, Papers I and II) with the amount of feedstock VS fed to the reactor. The conversion factor of 1 m^3_{CH4}=10 kWh was used. The mass of the produced digestate was calculated by subtracting the mass of the biogas from the feedstock (60 kt of FW + 40 kt of dilution water). The calculation of the biogas mass was based on biogas composition (60% CH_4, 40% CO_2) and component densities (CH_4 0.72 kg/m^3, CO_2 1.96 kg/m^3). In the digestate, the total nutrient concentrations (N_{tot}, P_{tot}, K_{tot}, kg/tFM) were assumed to be the same as in the feedstock, while the NH_4-N in FW was assumed to increase from 0.4 kg/tFM to 4 kg/tFM after digestion (from Papers I and II).

After digestion, the FW digestate was assumed to be separated with a decanter centrifuge producing liquid and solid fractions. The liquid fraction was further treated to produce fertilizer products, while the solid fraction was assumed to be used as such in agriculture. Centrifuge separation efficiencies for mass, TS, VS, nutrients (N_{tot}, NH_4-N, P_{tot}, K_{tot}), and electricity consumption were adopted from the literature (Table 14). Four systems (S1-S4) for the digestate liquid treatments along with a control system (S0) with no treatment were:

S0: no treatment
S1: stripping of digestate liquid
S2: stripping and RO of digestate liquid
S3: evaporation and RO of digestate liquid
S4: stripping, evaporation and RO of digestate liquid

It was assumed that the outputs from the different digestate liquid treatment systems were suitable for fertilizer use or processed water suitable for discharging or recirculation within the process. With each treatment system, the consumption of chemicals (NaOH, H_2SO_4, m^3/a) was included in the calculation of the output mass and characteristics. The literature values of the consumption of energy and chemicals, as well as the nutrient recovery efficiencies for each technology, are presented in Table 15. In the treatment system with combined stripping, evaporation, and RO (S4) the heat energy was allocated solely on the stripping, as it was assumed to be sufficient for both treatments (Ervasti et al., 2011).

Ammonia stripping combined with H_2SO_4 scrubbing produces ammonium sulfate (for agricultural use) and stripping residue (for agricultural use/post-treatment). The stripping temperature was 80°C. In the mass and nutrient balance calculations the NH_4-N recovery efficiency was 95% based on literature. $(NH_4)_2SO_4$ was assumed to be a chemically pure product with no TS, VS, P_{tot}, or K_{tot}, while N_{tot} consisted solely of NH_4-N. The stripping was assumed to be executed in atmospheric pressure; thus, no energy consumption for the production of a vacuum was allocated. Energy consumption values for both heat and electricity are presented in Table 15. Consumption of chemicals during stripping (50% NaOH) and scrubbing (93% H_2SO_4) were based on NaOH consumption during a pH increase in urine and the calculated H_2SO_4 consumption (Table 15).

The *evaporation* of either digestate liquid or stripping residue produces concentrate (for agricultural use) and condensate (for post-treatment). During evaporation, the liquid was heated to 80°C and the pH of the digestate liquid was controlled with H_2SO_4 to prevent the volatilization of NH_4^+. The mass and nutrient balance calculations for the evaporation were based on literature values (Table 15), where the TS and VS separation efficiencies in the concentrate were assumed to be 100% and the NH_4-N recovery rate the same as in N_{tot} (80%). The H_2SO_4 (93%) consumption was based on a pH decrease with manure and urine. The energy consumption of evaporation consisted of the heat energy needed to

increase the digestate liquid temperature and electricity consumption, which was based on typical literature values (Table 15).

The *RO treatment* of the stripping residue from stripping or condensate from evaporation produces both retentate (for recirculation within the process) and treated water (used as AD diluting water/discharged). The mass and nutrient balances for the RO treatment were calculated based on the nutrient separation efficiencies and electricity consumption values from the literature (Table 15). The regeneration and/or change of RO membranes were not taken into consideration.

Table 15. The literature values used in the calculations concerning the mass, nutrient, and energy balance of the digestate liquid treatment processes.

Process	Value/calculation	Reference
Stripping		
(NH_4-N) recovery	95 % in the ammonium sulfate	Basakcilardan-Kabakci et al., 2007, Bonmatí and Flotats, 2003a, Flotats et al., 2011, Guštin and Marinšek-Logar, 2011, Laureni et al., 2013, Liu et al., 2015
NH_4-N concentration	40 g/kgFM	Laureni et al., 2013
Heat consumption	Temperature increase from the digester to the stripper (from 40 to 80°C) calculated using the specific heat capacity of water	-
Electricity consumption	2 kWh/kgN	reviewed in van Eekert et al., 2012
NaOH (50%) consumption	20 L/m^3, pH from 9 to 10	Antonini et al., 2011
H_2SO_4 (93%) consumption	Calculated using the molar ratios of H_2SO_4 and $(NH_4)_2SO_4$ and the N concentration of 40 kg/tFM in the ammonium sulfate	-
Evaporation		
Nutrient recovery	In the concentrate: 20% mass, 90% N_{tot}, 100% P_{tot}, 100% K_{tot}	Bonmatí and Flotats, 2003b, Chiumenti et al., 2013, Ek et al., 2006, Flotats et al., 2011, Maurer et al., 2003
Electricity consumption	5 kWh/t liquid digestate	
Heat demand	Digestate liquid temperature from 40 to 80°C calculated using the specific heat capacity of water	-
H_2SO_4 (93%) consumption	0.005 m^3/t, pH from 9 to 6, pH from 7.2 to 5.5	Ek et al., 2006, Sørensen and Eriksen, 2009
RO treatment		
Nutrient recovery	In the retentate: 20% mass, 100% TS, 100% VS, 95% NH_4-N, 95% P_{tot}, 99% K_{tot}	Carretier et al., 2015, Chiumenti et al., 2013, Ek et al., 2006, Flotats et al., 2011, Ledda et al., 2013, Mondor et al., 2008
Electricity consumption	2.5 kWh/t stripping residue	

-, not applicable

4.4 Analyses

The chemical analyses and methods used in this thesis are summarized in Table 16. The CH_4 content from the biogas produced in STRs was determined automatically either by infrared analysis (ExTox Gasmess-Systeme GmbH, Germany) or using a portable Combimass GA-m gas analyzer (Binder Engineering GmbH, Germany) (Paper I). In BMP and RMP assays the volumetric measurement of CH_4 was automated and based on liquid displacement. All CH_4 yields were converted into the standard temperature and pressure conditions (0°C, 100 kPa) according to the ideal gas law using ambient temperature and air pressure (Papers I and II).

Hygienic quality (Paper II) was analyzed using *E. coli*, other coliforms, total coliforms, Enterococcus, sulfite-reducing clostridia and Salmonella as indicator organisms. Analyses of different coliforms were performed using a Harlequin *E. coli* / coliform (LabM) culture medium with 24–48 h incubation times at 37°C (Baylis and Patrick, 1999). Enterococcus were determined with KF streptococcus agar (incubated for 48 h at 44.5°C) according to SFS-EN ISO 7899 (Finnish Standard Association, 2000) and sulfite-reducing clostridia with sulfite-iron agar (incubated anaerobically for 48 h at 37°C) according to SFS-EN 26461 (Finnish Standard Association, 1993). For the qualitative analyses of Salmonella, samples were pre-enriched in buffered peptone water (37°C, 16–20 h) and incubated in Rappaport-Vassiliadis broth (42°C, 24 h). Aliquots from the broth were cultured on Salmonella-selective Rambach and xylose-lysine-decarboxylase agars and incubated at 42°C for 24 h. If growth was observed, colonies were confirmed with triple sugar iron agar, urea-agar, and lysine carboxylase broth (37°C, 24 h) (ISO, 2002).

Table 16. Summary of the chemical analyses and methods used in this thesis.

Analysis	Method and apparatus	Paper
Fresh samples		
pH	VWR pH100 pH-analyzer (VWR International).	I, II, III
TS and VS	SFS 3008 (Finnish Standard Association, 1990).	I, II, III
SCOD	Samples were centrifuged and analyzed according to SFS 5504 (Finnish Standard Association, 2002).	I, II, III
COD	Open reflux, titrimetric method used by the University of Southampton (modified from the Vienna standard method).	III
TVFA	Concentrations of acetic, propionic, iso-butyric, n-butyric, iso-valeric, valeric and caproic acids were determined using an HP 6890 gas chromatograph with an HP 7683 autosampler (Hewlett-Packard, Little Falls, USA) and GC ChemStation Rev. B.03.02 software.	I, III
TKN	Standard method (AOAC 1990) using a Foss Kjeltec 2400 Analyzer Unit (Foss Tecator AB, Höganäs, Sweden), with Cu as a catalyst.	I, II, III
NH_4-N	According to McCullough, 1967).	I, II, III
Soluble-N	Measured as TKN after 1:15 dilution.	II
	Measured from 1:5 water extractions with a Lachat autoanalyzer (Quikchem 8000, Zellweger Analytics, Inc., Milwaukee, WI, USA).	III
Soluble-P, soluble-K	Measured from 1:5 water extractions with inductively coupled plasma emission spectrometry (ICP-OES) (Perkin Elmer Optima 8300, USA).	II, III
Soluble NH_4-N, NO_3-N and PO_4-P	Analyzed with a Lachat autoanalyzer from 2 M KCl extracts.	III
P fractionation	Based on modified Hedley fractionation (Sharpley and Moyer, 2000, Ylivainio et al., 2008). The digestate was extracted sequentially with water, 0.5 M $NaHCO_3$, 0.1 M NaOH, and 1 M HCl at a ratio of 1:60. Inorganic P was determined from the extract, and the P_{tot} concentration was measured after digestion with peroxidase in an autoclave (Ylivainio et al., 2008). Organic P concentration was calculated as the difference between total and inorganic P.	III
Air dried (60°C) samples		
Crude protein	Duma's method with standard methods (AOAC, 1990) using a Leco FP 428 nitrogen analyzer (Leco Corp., St Joseph, USA) and N% multiplying factor 6.25.	II
Crude fat	Analyzed with a Soxcap-Soxtec-Analyzer (AOAC, 1990; Foss Tecator Application Note AN 390).	II
Soluble carbohydrates	Samples were inverted with 1 N HCl and analyzed according to Somogyi, 1945).	II
NDF	According to Van Soest et al., 1991) with a filtering apparatus.	II
ADF	According to Robertson and Van Soest, 1981).	II
Cellulose	Calculated as ADF-Lignin.	II
Hemicellulose	Calculated as NDF-ADF.	II
C_{tot}	Duma's method according to manufacturer's instructions with a Leco CN-2000 Elemental Analyzer.	II, III
P_{tot}, K_{tot}	Samples were digested with HNO_3 (Huang and Schulte, 1985) and analyzed with ICP-OES according to manufacturer's instructions.	II
Ni, Cu, Zn, Cr	Digested in aqua regia according to SFS ISO 11466 (Finnish Standard Association, 2007). After digestion, determined with ICP-OES (Thermo Jarrell Ash IRIS Advantage, Thermo Scientific, USA).	III
As, Cd, Pb	Digested in aqua regia according to SFS ISO 11466 (Finnish Standard Association, 2007). After digestion, determined with graphite furnace atomic absorption spectrometry using a Varian AA280Z (Varian Inc., USA).	III
Hg	Measured based on cold vapor atomic absorption spectrometry using Varian M-6000A Mercury Analyzer (Varian Inc., USA).	III
Fe	According to Huang and Schulte, 1985) with ICP-OES (Thermo Jarrel Ash Iris Advantage, Franklin, USA).	I

5 Results and discussion

5.1 Food waste characteristics

The characteristics of the FW define its potential as biogas plant feedstock. The organic composition of the FW determines, e.g., the CH_4 production potential, while the elemental content affects the AD process stability as well as the characteristics and fertilizer value of the produced digestate. The three studied FWs (FW1–FW3, Table 19, Papers I–III), showed rather similar characteristics, with high TS (20–25%), VS (20–23%), and TKN (7–8 g/kgFM) content, as has been previously reported with similar FWs from the Europe, Asia, and North America (Table 2). The studied FWs had similar organic content (proteins, fats, cellulose, hemicellulose), as has been reported previously for FWs from households and restaurants (Table 2, Tanimu et al., 2015, Vavouraki et al., 2014, Zhang et al., 2015a, Zhang et al., 2012). The high content of proteins and fats (in total, around 35% of the VS content) also led to a relatively high BMP value for FW1 (500 m^3CH_4/kgVS). Previously reported BMP values for FWs have been lower, in the range of 300 to 480 m^3CH_4/kgVS (Table 5). The high BMP value obtained with FW1 is most likely due to the source separation of the waste, which (along with the manual separation of the biodegradable bags) increased the BMP. Additionally, the FW1 was macerated before analysis, which increases the contact area for the hydrolytic micro-organisms and increases CH_4 production (Ariunbaatar et al., 2014a, Izumi et al., 2010). Overall, the OFMSW showed slightly different characteristics (Table 17), e.g., higher TS (29%) and fiber content (cellulose + hemicellulose 44% VS in OFMSW, 10–15% in FWs), which are due to the different treatment methods of this waste fraction, where, for example, the separation of contaminants (glass, metals) is applied.

Table 17. The characteristics of the studied materials.

Parameter	Unit	FW1	FW2	FW3[b]	OFMSW[b]	Paper
General characteristics (n=9–10)						
pH[a]	-	5.1 ± 0.2	5.0 ± 0.1	5.0	4.7	I,II, III
TS	g/kgFM	247.9 ± 4.8	208.5 ± 10.0	255.1	287.0	I,II, III
VS	g/kgFM	230.3 ± 4.5	192.0 ± 8.9	232.8	264.3	I,II, III
VS/TS	%	92.9 ± 0.2	92.1 ± 0.5	91.3	92.1	I,II, III
SCOD	g/kgFM	98.4 ± 11.6	116.4 ± 9.9	132.9	69.9	I,II, III
COD[b]	g/kgFM	364.4	361.2	444.0	412.5	III
TVFA	g/kgFM	3.3 ± 0.4	2.2 ± 0.1	4.9	5.5	I,II, III
TKN	g/kgFM	7.5 ± 0.3	6.9 ± 0.3	8.2	5.7	I,II, III
NH_4-N	g/kgFM	0.4 ± 0.1	0.4 ± 0.1	0.6	0.3	I,II, III
NH_4-N/TKN	%	5.1 ± 1.6	6.0 ± 0.1	7.2	5.4	I,II, III
Density[c]	kg/l	1.064 ± 0.0042	1.063 ± 0.0002	n.d.	n.d.	I
Methane production in batch assays (n=2)						
BMP	m^3CH_4 /kgVS	0.501 ± 0.020	0.445 ± 0.001	n.d.	n.d.	I
Organic characteristics (n=3–4)						
Crude protein	g/kgVS	203.5 ± 16.8	205.4 ± 3.6	209.7[d]	182.0 [d]	II
Crude fat	g/kgVS	131.7 ± 9.1	133.0 ± 7.0	n.d.	n.d.	II
Soluble carbohydrate	g/kgVS	114.1 ± 16.8	56.2 ± 5.4	101.6[d]	18.2[d]	II
Cellulose	g/kgVS	47.9 ± 6.6	57.8 ± 9.0	79.4[d]	342.0[d]	II
Hemicellulose	g/kgVS	52.2 ± 6.3	33.2 ± 7.4	58.1[d]	101.3[d]	II
Lignin	g/kgVS	6.2 ± 7.6	75.5 ± 9.9	5.6[d]	22.9[d]	II
(cel+hemi)/lign	-	16.1	1.2	24.6	19.4	II
Total nutrients (n=3–4)						
C_{tot}[b]	g/kgTS	469.1	486.6	n.d.	n.d.	II
TKN	g/kgTS	30.7 ± 1.7	32.1 ± 1.6	32.1	19.9	II
C/N	-	15.3	15.2	n.d.	n.d.	II
P_{tot}	g/kgTS	3.8 ± 0.1	6.5 ± 1.3	n.d.	n.d.	II
K_{tot}	g/kgTS	11.4 ± 1.6	10.3 ± 0.4	n.d.	n.d.	II
Soluble nutrients (n=3–4)						
Soluble-N	g/kgTS	9.6 ± 0.5	16.3 ± 0.4	n.d.	n.d.	II
Soluble-P	g/kgTS	1.7 ± 0.8	1.7 ± 0.3	n.d.	n.d.	II
Soluble-K[b]	g/kgTS	9.0	9.0	n.d.	n.d.	II
Metals and heavy metals (n=2 for Fe, n=1)						
Fe	g/kgTS	0.13 ± 0.01	22.73 ± 12.5	n.d.	n.d.	I
Ni	mg/kgTS	0.6	0.5	1.0	0.8	III
Cu	mg/kgTS	4.9	8.4	5.7	9.6	III
Zn	mg/kgTS	28.2	37.8	29.4	93.3	III
Cr	mg/kgTS	1.1	3.3	1.8	1.3	III
Pb	mg/kgTS	0.2	2.2	0.7	0.5	III
Cd	mg/kgTS	0.06	0.05	0.06	0.02	III
Hg	mg/kgTS	0.06	0.08	0.08	0.05	III
As	mg/kgTS	0.5	0.5	0.4	0.2	III

[a]n=26, [b]n=1, [c]n=3, [d]Valorgas, 2010b
FW2=autoclaved FW
n.d., not determined

The heavy metal concentration within the studied FWs (Table 17, Paper III) was generally similar, as reported before for source separated FWs in Europe, North America, and Asia (Malamis et al., 2015, Zhang et al., 2011, Zhang et al., 2007, Zhang et al., 2012). With FW1, FW2 and OFMSW, the Pb, Hg, and Cd contents were low (<1 mg/kgTS), and Cu (10 mg/kgTS) and Zn (30 mg/kgTS) contents were

comparable to those reported with European FWs (Malamis et al., 2015, Zhang et al., 2012). The content of both Ni (<1 mg/kgTS) and Cu (<2 mg/kgTS) in the studied FWs were in the lower range when compared with the results obtained from the literature (1–10 mgCu/kgTS, 1–30 mgCr/kgTS, Facchin et al., 2013, Malamis et al., 2015, Zhang et al., 2011, Zhang et al., 2007, Zhang et al., 2012).

The hygienic quality of the studied FWs was analyzed with hygiene indicators (Figure 9, Paper II, Valorgas, 2010b). FWs showed similar concentrations of coliforms (0–1 logs), Enterococcus (4 logs) and clostridia (3 logs), while the OFMSW sample had a higher concentration of coliforms (3 logs) and Enterococcus (6 logs). In fresh FW, coliforms have usually been reported in higher concentrations (4–5 logs) in biogas plants treating FW (Sahlström et al., 2008). The lower concentrations in the present study were most likely due to the storage time in a freezer before analysis. However, the concentrations of Enterococcus were similar to those in fresh FW (around 4 logs, Sahlström et al., 2008), which is due to the high resistance of the spore-forming micro-organisms (both Enterococcus and clostridia) (Sahlström, 2003) to, e.g., freezing.

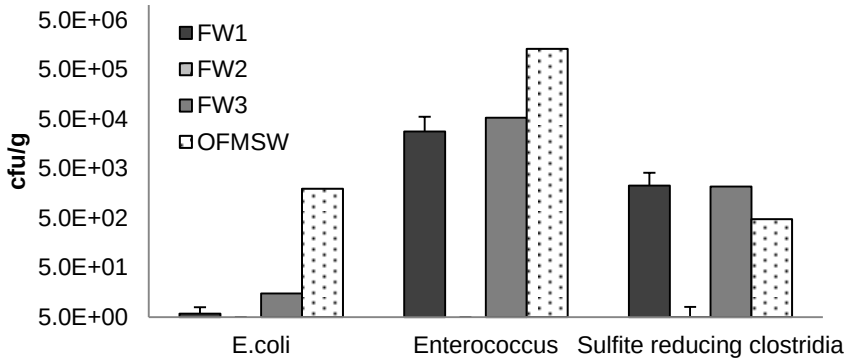

Figure 9. The hygienic quality of studied food wastes. All samples were stored frozen, n=6 for FW1 and FW2 (Paper II), n=1 for FW3 and OFMSW (Valorgas, 2010b).

5.1.1 Effect of autoclave pretreatment on FW characteristics

The applied autoclave pretreatment conditions, 160°C, 6.2 bars, were based on previous studies and applications for the pretreatment of MSW with similar conditions and autoclaving equipment (Papadimitriou et al., 2008, Papadimitriou, 2010, Papageorgiou et al., 2009). The autoclaving of the FW affected the characteristics, especially the organic content and BMP value of the FW (Table 17, Paper I, II). Because the autoclave treatment applies both heat and pressure, the material starts to hydrolyze, which can be seen in increased SCOD (soluble chemical oxygen demand, per FM) and decreased concentrations of easily hydrolysable carbohydrates and hemicellulose (per VS, Table 17, Table 18). However, the TS, VS, and TKN concentrations (per FM) were observed decrease, because the autoclaving introduces steam to the FW and dilutes the material (Zhou et al., 2013). Conversely, both cellulose and lignin contents, P_{tot}, Fe, and some heavy metals increased in FW after autoclaving.

The increased content of P, Fe, Cu, Zn, Cr, and Pb (per TS) seemed to indicate contamination from the autoclaving apparatus, as these elements can also be found in steel, either as alloy or impurities. The autoclaved FW showed the absence of all indicator organisms, which was due to effective sterilization during the pretreatment (Figure 9, Paper II).

Table 18. The effect of autoclaving pretreatment on the FW organic content.

Studied material	TS (%)	VS (%)	Treatment conditions	Effect of treatment on organic matter	Reference
FW	16.6	15.5	Autoclave, 120°C, 1 bar, 30 min	SCOD increase 24% (per FM)	Ma et al., 2011
FW	20.0	18.0	Thermal hydrolysis, 175°C, 60 min	VSS decrease 40% SCOD increase 0–50% depending on molecular weight fraction Soluble sugars increase 50%	Liu et al., 2012
FW	-	41[a]	High pressure autoclave, 185°C, 12 bars , 10 min	VSS decrease 10%	Lissens et al., 2004
FW	10.9	8.7	Thermal hydrolysis, 170–175°C, 5 bars, 60 min	Suspended solid decrease 30%	Zhou et al., 2013
Commingled HW	-	-	Autoclave 160°C, 6.2 bars	Hemicellulose decrease 10%	Papadimitriou, 2010
FW	20.8	19.2	Autoclave, 160°C, 6.2 bars, 45 min	SCOD increase 18% Soluble carbohydrates decrease 51% Hemicellulose decrease 36 %	Present study

Household waste (HW), volatile suspended solids (VSS)
[a]gVSS/L
-, not available

After autoclaving, the increased SCOD and lowered TVFA were due to the dilution and solubilization of the FW during treatment. Solubilization occurred as the heat and pressure of the treatment disintegrated the cell membranes, which is dependent on both the treatment time and conditions (reviewed in Ariunbaatar et al., 2014a, Carlsson et al., 2012). The reduction in hemicellulose content was most likely due to the branched structure of the hemicellulose, which enabled easier hydrolysis during autoclaving (Hendriks and Zeeman, 2009, Papadimitriou, 2010). The autoclave treatment also decreased the soluble carbohydrate content, which indicated the formation of Maillard-like compounds (Liu et al., 2012, Monlau et al., 2013) through reactions between sugars and amino acids (Bougrier et al., 2008, Liu et al., 2012, Monlau et al., 2013). The reactions have also been reported to change the color of the treated material to dark brown (Ariunbaatar et al., 2014b, Bougrier et al., 2008), and this phenomenon was also observed with the studied autoclaved FW (FW2, Figure 6). Maillard compounds are difficult to degrade during AD which decreased the CH_4 yield in BMPs by 10% (Table 17), as has been observed before with thermal treatment temperatures ranging from 140 to 175°C (Table 7).

5.2 Continuous anaerobic digestion of food waste

The continuous AD of FW (FW1) and autoclaved FW (FW2) was studied in mesophilic laboratory STRs (Paper I). The reactor experiment lasted for 473 days, during which the OLR was increased from its initial 2 to 6 kgVS/m^3d (HRTs from 117 and 94 to 39 and 31, Figure 10). The FWs – FW1 and FW2, with TS contents of 25% and 20% – were used as feed as is, without water/leachate circulation during digestion. The initial NH_4-N concentration in the studied STRs was 2.4 g/kgFM, which was dependent on the ammonium concentration of the inoculum. During the reactor runs, the NH_4-N concentration in the FW1 reactor increased to 4 kg/kgFM during OLR 3 kgVS/m^3d, which was mainly due to the accumulation of solids (and subsequently the TKN) into the reactor. However, in reactors treating autoclaved FW2, the NH_4-N remained steady (around 2 g/kgFM) decreasing to 1.2 g/kgFM at the end of the experiment.

The long-term mesophilic AD of FWs with high OLRs has been observed to lead to VFA accumulation and subsequently to the inhibition of micro-organisms due to high NH_4-N content, which has been linked to TE deficiencies (Banks et al., 2012, Yirong et al., 2015, Zhang and Jahng, 2012, Zhang et al., 2015a). When supplemented with TEs, successful FW digestion has been reported at OLRs from 5 to 7 kgVS/m^3d (Table 6). In the present study, the increase of the NH_4-N concentration in the FW1 reactor led to increased TVFA concentrations; thus, the FW2 reactor also experienced a TVFA increase at the same time (Figure 10). After the OLR was increased to 3 kgVS/m^3d, both reactors were supplemented with a TE solution containing Se and Co, as Se in particular has been reported to be important in reducing VFA formation (Banks et al., 2012) when OLR is high (Zhang et al., 2015b). After a few weeks, full TE supplementation was started (Al, B, Co, Cu, Fe, Mn, Mo, Ni, Se, W, and Zn), which led to a decrease in VFA concentrations in both reactors, as has been reported in previous studies as well (Table 6).

Overall, with TE additions, both FW1 and FW2 reactors were able to operate at high OLRs. During OLRs 2 and 3 kgVS/m^3d, FW1 showed better performance (e.g. CH_4 yield 0.44–0.48 m^3/kgVS) compared with FW2 (CH_4 yield 0.37–0.43 m^3/kgVS) despite the high NH_4-N concentration in the reactor. The maximum CH_4 yield for FW1 was obtained at OLR 3 (0.48 m^3/kgVS) and with FW2 at OLR 4 (0.44 m^3/kgVS), which were in the same range as reported with FWs at different OLRs (Table 6). The studied FW reactors showed stable performance at OLR 4 kgVS/m^3d, after which the OLR was increased to 6 m^3/kgVS, where some VFA peaks started to show in the FW1 reactor alongside the decrease in pH and NH_4-N concentrations. However, FW2 reactors remained more stable in terms of VFA accumulation (Figure 10).

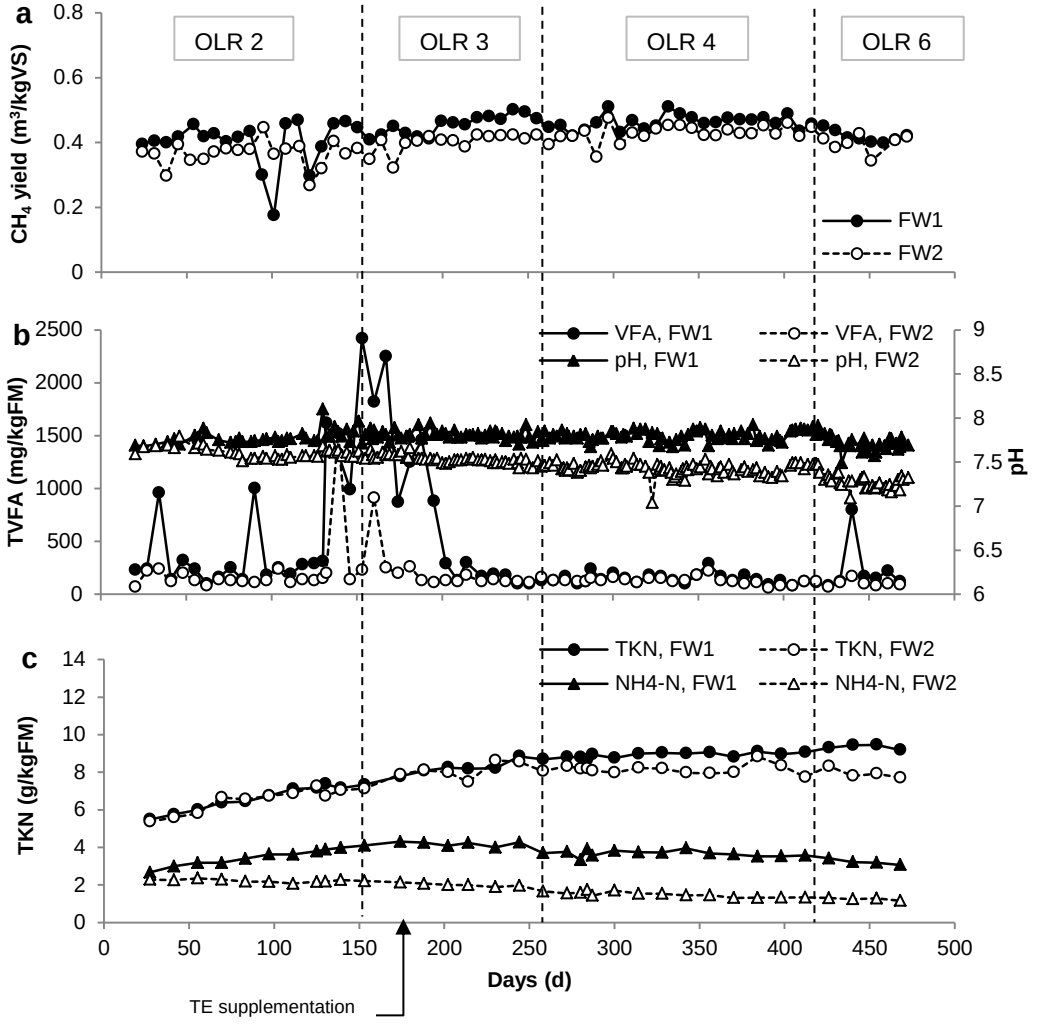

Figure 10. a) The methane yields, b) concentrations of total VFAs and pH value, and c) concentrations of TKN and NH_4-N within STR reactors treating FW1 and FW2 (autoclaved). TE supplementation was started on day 179. (Paper I).

The effect of autoclaving of FW to the performance of the continuous AD was observed with the nitrogen content, CH_4 yield, and gas composition (Paper I, II). Decreased CH_4 and NH_4-N were both related to the formation of Maillard compounds during autoclaving, which decreased CH_4 yield in BMP tests and indicated the inability of micro-organisms to fully degrade the autoclaved FW (Paper I, Table 17). The formation of the Maillard compounds also bound the amino acids to the recalcitrant molecules, which decreased the ammonification during STR (Paper I) and BMP tests (Paper I, II). Additionally, the biogas composition analyses showed, that the reactor fed with the autoclaved FW2 had low H_2S content (<75 ppm) throughout the experiment, while the H_2S increased in the FW1 reactor after OLR 4 kgVS/m^3d (up to 480 ppm, Figure 11). The decreased H_2S formation was mainly due to the autoclaving pretreatment, which decreased the availability of S in the proteins. Other explanations can be, e.g., the precipitation of S to iron sulfides along with the high Fe content (23

g/kgTS, Table 17) and lower pH (around 7.3), which would inhibit the H_2S-forming micro-organisms (O'Flaherty et al., 1998). The microbial consortia within the FW2 reactor were also shown to be different from the FW1 reactor (Blasco et al., 2014), which supports the findings with the decreased CH_4 formation, ammonification and H_2S formation after autoclaving FW.

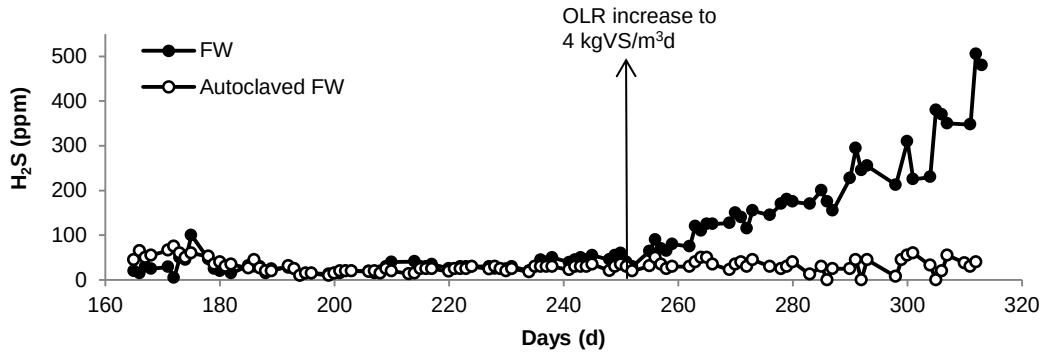

Figure 11. The H_2S concentration within STRs treating FW and autoclaved FW during days 166–314. (Paper I).

5.3 Usability of FW digestate in agriculture

5.3.1 Digestate characteristics

Organic content and stability

The digestate characteristics are dependent on the feedstock and AD performance. Overall, the substrate degradation during AD decreased the solids and organic content (e.g., TS content from 25% to 7% in FW1, Table 17, Table 19, Paper II). The C/N ratios were relatively low (from 1.5 to 4.4) in digestates due to the mineralization of carbon during AD. The fiber content (per VS) in the studied FWs decreased overall; thus, the increase in lignin content was due to the low biodegradability of the ligno-cellulosic complexes (Hendriks and Zeeman, 2009) and the reduction of TS during AD. The biodegradation of the fibers can be assessed with the ratio between cellulose, hemicellulose, and lignin (cel+hemi/lign), which was constant (1.2) in FW2 before and after digestion (Table 17, Table 19). This indicates the degradation of hemicellulose and cellulose already during autoclaving and not during AD. The higher content of hardly degradable cellulose, lignin, and proteins in the FW2 digestate likely reduced the BMP during batch experiments compared to the FW digestate (Paper I, II).

Table 19. The characteristics of the studied digestates.

Parameter	Unit	FW1[a]	FW2[a]	FW3[b]	OFMSW[b]	VWAS[b]	Paper
General characteristics							
pH	-	8.0 ± 0.0	7.7 ± 0.1	8.3	8.3	7.6	II, III
TS	g/kgFM	67.4 ± 0.1	78.5 ± 5.1	19.9	32.2	34.2	II, III
VS	g/kgFM	45.6 ± 3.0	60.5 ± 6.5	12.3	18.9	23.9	II, III
VS/TS	%	67.7 ± 4.3	77.0 ± 3.7	61.7	58.7	69.9	II, III
SCOD	g/kgFM	13.1 ± 1.5	15.3 ± 1.3	11.2	7.3	8.4	II, III
COD	g/kgFM	77.1	100.3	21.8	30.6	26.7	III
TVFA	g/kgFM	0.3 ± 0.01	0.2 ± 0.03	4.1	0.3	3.4	II, III
Organic characteristics							
Crude protein	g/kgVS	211.4 ± 35.0	342.3 ± 43.5	213.9	158.5	244.5	II
Crude fat	g/kgVS	38.3 ± 0.2	35.5 ± 5.6	n.d.	n.d.	n.d.	II
Soluble carbohydrate	g/kgVS	3.5 ± 0.2	4.0 ± 0.3	3.4	3.2	3.8	II
Cellulose	g/kgVS	44.6 ± 8.4	95.7 ± 22.0	32.5	30.4	25.1	II
Hemicellulose	g/kgVS	54.9 ± 4.9	83.5 ± 9.8	75.8	47.6	67.2	II
Lignin	g/kgVS	27.5 ± 0.1	148.4 ± 9.7	30.9	21.2	32.1	II
(cel+hemi)/lign	-	3.6	1.2	3.5	3.7	2.9	II
Total nutrients							
TKN	g/kgFM	7.8 ± 0.6	7.3 ± 0.5	4.7	4.5	2.1	II, III
NH_4-N	g/kgFM	4.1 ± 0.3	1.9 ± 0.4	3.9	3.2	1.7	II, III
NH_4-N/TKN	%	52.2 ± 0.7	25.7 ± 7.2	82.1	71.1	78.6	II, III
C_{tot}	g/kgFM	26.5 ± 0.6	29.3 ± 4.7	6.8	10.3	13.5	II, III
C/N	-	3.4	4.0	1.5	2.3	6.1	II, III
P_{tot}	g/kgFM	1.3 ± 0.2	1.3 ± 0.1	n.d.	n.d.	n.d.	II
K_{tot}	g/kgFM	3.0 ± 0.6	2.4 ± 0.3	n.d.	n.d.	n.d.	II
Soluble nutrients (1:5 water extraction)							
N_{tot}	g/kgFM	6.0	3.0	4.4	4.0	2.2	III
NH_4-N	g/kgFM	4.4	1.9	3.3	2.8	1.6	III
NO_3-N	g/kgFM	0.013	0.011	0.011	0.007	0.003	III
PO_4-P	g/kgFM	0.27	0.14	0.06	0.13	0.35	III
P_{tot}	g/kgFM	0.33	0.19	0.11	0.15	0.35	III
K_{tot}	g/kgFM	3.2	2.5	1.9	1.9	0.6	III
Heavy metals							
Ni	mg/kgTS	17.8	16.6	42.4	6.7	22.3	III
Cu	mg/kgTS	25.6	22.4	21.7	58.7	626.5	III
Zn	mg/kgTS	116.0	94.6	175.0	401.0	1006.0	III
Cr	mg/kgTS	9.8	11.9	7.5	13.0	32.9	III
Pb	mg/kgTS	2.1	5.6	5.6	11.7	98.0	III
Cd	mg/kgTS	0.2	0.1	0.3	1.5	1.1	III
Hg	mg/kgTS	0.1	0.2	0.1	0.3	1.8	III
As	mg/kgTS	0.7	0.4	1.0	3.3	2.6	III

[a]n=2–3 (Paper II), [b]n=1 (Valorgas, 2010b)
n.d., not determined

Considering the digestate characteristics from an agronomic viewpoint, the TS content and stability (pH, organic matter content and composition) are important measures, as they have a positive effect on the soil carbon balance and nutrient availability (Abubaker et al., 2012, Galvez et al., 2012, Odlare et al., 2008), but in too-large doses, they can inhibit soil micro-organisms (Alburquerque et al., 2012b, Gutser et al., 2005) and cause phytotoxicity (Abdullahi et al., 2008, Trzcinski and Stuckey, 2011). The TS and VS content of the studied digestates were within the range of other FW-based digestates from

the literature (TS 2–8%, Table 8, Table 19). The lower TS range (2–3%) was related to the internal water additions/recirculation in the biogas plants from which the digestates FW3, OFMSW, and VWAS originated (Paper III). Overall, the results support the fact that the digestate's TS concentration is dependent on the reactor configuration (e.g., wet/dry process) and process parameters (OLR, HRT) (Teglia et al., 2011), i.e., the TS and VS reduction during AD, despite the uniform characteristics of the feedstocks.

All studied digestates were neutral or slightly alkaline (pH 6.7–8.4), which is typical for FW-based digestates (Table 8, Table 19, Paper III). 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). The effect of digestate pH on soil is dependent on soil characteristics (Alvarenga et al., 2015). Hence, in a 4-year fertilization study, the soil initial pH of 5.4–5.7 was not affected after application of FW digestates (Odlare et al., 2008). The stability of the digestates is related to the composition of the organic content, e.g., organic acids, which affect the pH value. All studied FW digestates were characterized with higher SCOD concentrations (11–19 g/kgFM) compared with OFMSW and VWAS digestates (7–8.5 g/kgFM). However, in terms of TVFA concentration, only FW2 and OFMSW were considered stable, as the TVFA was under the limit of 1500 mg/l (Table 19, Paper III), which is proposed for digestate fertilizers within the end-of-waste criteria (Saveyn and Eder, 2014). However, VFAs are reported to be quickly degraded in soils after digestate application by soil micro-organisms (Kirchmann and Lundvall, 1993). The non-VFA-SCOD found in digestates was most likely related to, for example, undegraded carbohydrates and other acids such as humic acids (Scaglia et al., 2015, Zheng et al., 2014). The stability of the digestate can also be assessed with RMP (BSI, 2010), which was low in the digestates FW1 and FW2: 60–130 m^3/kgVS, depending on the OLR (Paper I, II). The RMP values obtained with FWs were similar and slightly lower than RMP values obtained with OFMSW (66–198 m^3/kgVS, Trzcinski and Stuckey, 2011), where the different reactor configurations (wet/dry) and operation also affect the RMP values through VS reduction.

Nutrient content and availability

The characterization of the studied digestates (Papers II, III) showed the high fertilizer value of the FW- and OFMSW-based digestates compared with VWAS digestate, as the N availability 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). Similarly high NH_4-N (around 4 g/kgFM) concentrations and NH_4-N/TKN (>50%) ratios have been obtained in FW digestates (Table 8). The high fertilizer value in the studied digestates was also supported by the high ratio between C and organic N (C/N_{org} around 8 in FW and OFMSW digestates), which indicates high N release in soils compared to VWAS digestate (C/N_{org} ratio of 29) (Gutser et al., 2005). Overall, the FW and OFMSW were characterized as being rich in N and relatively poor in P. The VWAS digestate had a relatively low concentration of both nutrients, which

leads to reduced fertilizer value and the need for additional mineral fertilizer supplements 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 a source of nutrients (Teglia et al., 2011).

The availability of P for plant growth is dependent on its solubility, which was analyzed with Hedley fractionation (Paper III). The total P in the digestates was not fully plant-available, and in FW and VWAS digestates, 50–70% of the P was considered plant-available (water and $NaHCO_3$ extractable, Figure 12). OFMSW digestate showed a lower P solubility of 30%, indicating a difference in the digestate composition compared with FW digestates. However, this was not detected in any other characterization analysis. Measuring P solubility is essential when evaluating the mineral fertilizer substitution capacity of the digestates to avoid the overestimation of P availability. Some life cycle analyses have overestimated the P substitution by assuming that 100% of mineral fertilizer P can be substituted with digestates (Bernstad and la Cour Jansen, 2011, Boldrin et al., 2011), while some studies applied a more accurate P substitution rate of 50% (Evangelisti et al., 2014).

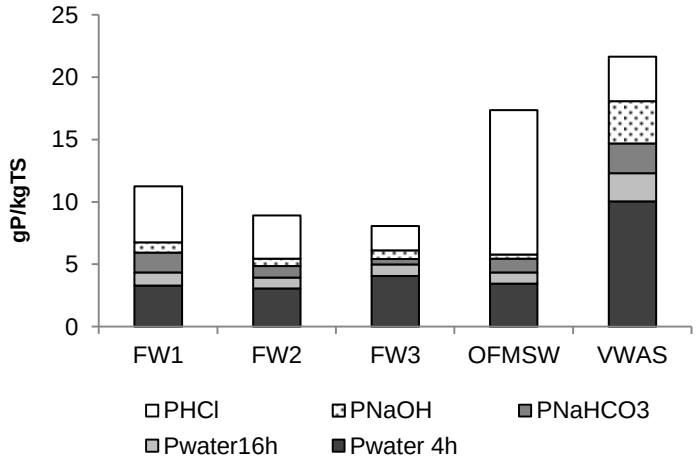

Figure 12. The Hedley fractionation of studied digestates. Water and $NaHCO_3$ extractable P can be considered as plant available. (Paper III).

Biosecurity

The biosecurity issues related to digestate use arise from the characteristics related to, e.g., pathogens (Paper II, Valorgas, 2010b) and heavy metals contents (Paper III). According to the European Animal By-Products Regulation (1069/2009/EC, European Parliament and the Council, 2009) digestates must meet hygienic standards before use as fertilizers in agriculture, and the threshold values are 1000 cfu/g for *E. coli* or Enterococcaceae and no Salmonella detected in a 25 g sample. In the studied digestates, no Salmonella was detected when indicator organisms were analyzed from fresh FW1 and FW2 (Paper II) and after freezing from FW3, OFMSW, and VWAS digestates (Valorgas, 2010b). *E. coli* was detected in only one sample (FW3, 105 cfu/g), and the digestates were considered suitable for

agricultural use. Digestates FW1 and FW2 were not hygienized (FW2 autoclaved prior to AD), and these digestates showed high enterococcus concentrations (8 logs in the FW1 and FW2 digestate) but a lack of coliforms, which was connected with the freezing of the feedstock prior to AD (Figure 13, Paper II). The observed increase in enterococci and clostridia in the FW2 digestate indicates the potential of autoclaved material for microbial growth. Digestates FW3, OFMSW, and VWAS showed lower concentrations of enterococcus and clostridia, but it was uncertain whether or not these digestates were hygienized prior to AD. Coliforms and Salmonella are both vulnerable to the hygienization process (Bagge et al., 2005, reviewed in Sahlström, 2003), which is why their absence could refer to the use of hygienization with these digestates (FW3, OFMSW, VWAS). However, both enterococcus and sulfate-reducing clostridia are spore-forming bacteria and are known to survive the hygienization treatment (Bagge et al., 2005), which explains the detected indicators in all studied digestates. Additionally, the storage of the digestates may increase the concentration of the indicator organisms due to contamination and microbial growth (Bagge et al., 2005).

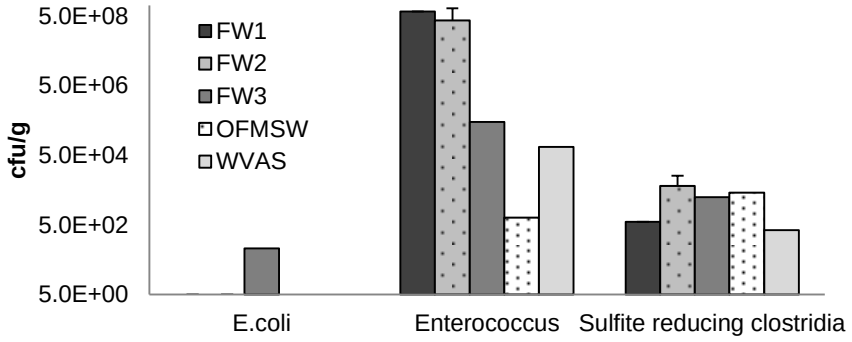

Figure 13. The concentration of *E. coli*, enterococcus and sulfite reducing clostridia in the studied digestate samples. FW2= autoclaved FW digestate. For FW1 and FW2 digestates n=7 (fresh samples, Paper II), for FW3, OFMSW, and VWAS n=1 (frozen samples, Valorgas, 2010b).

The heavy metal content (per TS) tends to increase and concentrate from feedstocks to digestates due to the reduction of solids content during AD. Overall, the heavy metals in the digestates was similar to those reported with different organic waste digestates originating from FW (Govasmark et al., 2011), a mixture of biowastes and industrial wastes (Kupper et al., 2014), and sewage sludge (Zirkler et al., 2014, Paper III). FW and OFMSW digestates had fairly similar contents of heavy metals (Table 19), which reflected the content in the feedstocks (Table 17). According to the legislative heavy metal content limits for digestates in Finland (Ministry of Agriculture and Forestry, 2011) and the UK (BSI, 2010), the studied FW- and OFMSW-based digestates were suitable for agricultural use. However, the VWAS digestate exceeded the legislative limits, most likely due to the feedstock characteristics.

5.3.2 Digestate fertilizer use

The transformation of digestate organic N into its mineral forms in soil was studied via mineralization experiments with different digestate N application rates from 171 to 318 mgTKN/kg soil (Table 20, Paper III). Additionally, plant growth and N uptake in pot experiments were studied with Italian ryegrass (cv. Fabio) in order to compare the N fertilizer value of the digestates (Figure 14, Paper III). Overall, both tests showed potential for FWs to be used as such as fertilizer in agriculture. After 48 days, the mineralization of organic N was of the same magnitude (around 30 mgN/kgFM) as in all other digestates except the FW3 digestate (mineralization of N_{org} 2 mgN/kgFM), which was due to the already mineralized N within digestate FW, where the application of the organic N was 25–60% lower than with other digestates. Digestates FW1, FW2, OFMSW, and VWAS had lower initial NH_4-N concentrations, and 15–30% of their organic N mineralized during the test.

In the ryegrass growth experiments, digestate applications produced ryegrass yields of 38–60 gDM/pot, depending on the applied N amount; these yields were 5–30% higher than the control with similar inorganic N concentration (Figure 14). Similar results have been obtained with other FW-based digestates in pot and field trials (see Table 9, Abubaker et al., 2012, Furukawa and Hasegawa, 2006, Haraldsen et al., 2011, Rigby and Smith, 2014). FW1 and FW2 digestates had 20–30% higher yields than the control, and high NH_4-N utilization efficiencies (NUE_{NH4-N} >90%) were observed because soluble N 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 with the control, and NUEs were between 74 and 82%, indicating that the soluble N was not fully available for plant growth.

Table 20. The applied and mineralized nitrogen during the 48-day mineralization test with the studied digestates (Paper III).

Sample	FW1	FW2	FW3	OFMSW	VWAS
Applied mg/kgFM					
TKN	205	171	235	244	318
NH_4-N	97	50	158	142	137
NO_3-N	0	0	1	0	0
N_{org}	108	121	77	102	181
Mineralization from applied N					
mg/kgFM	36	34	2	29	26
% of N_{org}	33	28	2	28	14

The low initial NH_4-N in the FW2 digestate was due to the pretreatment of the feedstock, where the nitrogen-containing molecules were transformed into recalcitrant and hardly degradable Maillard compounds; 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 micro-organisms were still to some extent able to transform the rather recalcitrant nitrogen, and the ryegrass was able to use the remaining NH_4-N for plant growth. However, the relatively low NUE_{TKN} found (33%) with both FW2 and VWAS digestates indicated that the TKN consisted of

recalcitrant N, which was not plant-available and fully mineralizable (Gunnarsson et al., 2010). 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 a 50% decrease in N_{org} mineralization compared to the other studied digestates. This difference was connected with the composition of the WAS feedstock, which led to a low TKN and NH_4-N concentration in the VWAS digestate and lower fertilizer performance compared with FW and OFMSW digestates.

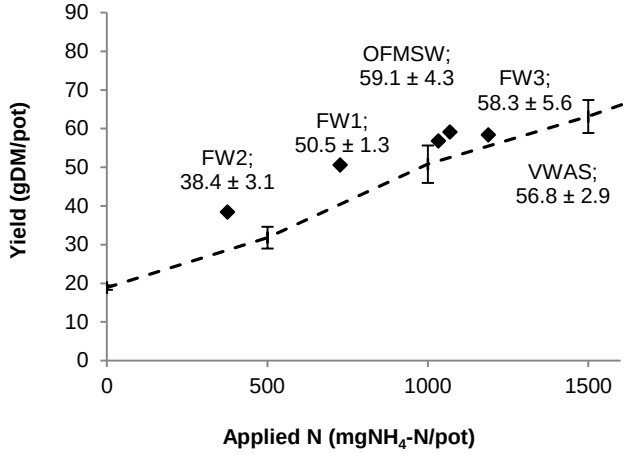

Figure 14. Ryegrass yield in digestate samples compared with the control mineral fertilizer treatment. The dotted line represents the control and error bars the standard deviations within control samples. (Paper III).

5.3.3 Digestate post-treatment

The energy, mass, and nutrient balances of a theoretical AD plant treating FW were evaluated, focusing especially on the treatment of the digestate liquid fraction. The aim was to evaluate the digestate liquid post-treatment in order to assess the feasibility of producing concentrated nutrient products (Paper IV). Additionally, four treatment options for the digestate liquid were compared, consisting of combinations of ammonia stripping, evaporation and membrane filtration (reverse osmosis), which have been applied in the full-scale treatment of digestate and manure-based liquids (e.g., Boehler et al., 2015, Flotats et al., 2011, Fuchs and Drosg, 2013). The concentration of the digestate liquid into concentrated fertilizer products consumed around 7% of the produced energy from the FW (Table 21). In total AD, solid-liquid separation and digestate liquid treatment accounted for 26% of the primary energy, of which 19% was used in AD and digestate separation. The result was in the same range as previously reported for ADs treating 20–60 kt/a of OFMSW and a mixture of municipal and agricultural wastes (17% and 20% of the total energy production in Berglund and Börjesson, 2006, Pöschl et al., 2010, respectively).

The energy consumption of the digestate liquid treatment processes consisted mainly of the heat demand, which in the all the studied digestate liquid treatments accounted for 80–90% of the total energy demand, as the process temperature for both stripping and evaporation was high (80°C) in

order to achieve efficient nutrient recovery (Mehta et al., 2015). However, the heat demand of stripping and evaporation can be rationalized by integration with AD, which enables the recovery of the plant's excess heat (Bonmatí and Flotats, 2003a, Hjorth et al., 2010, Mehta et al., 2015), especially in situations where heat energy is not utilized, e.g., in district heating systems. Additional reduction in the energy consumption of the studied digestate liquid treatment technologies could be achieved through using lower process temperatures. For example, ammonia stripping (N recovery >80%) has been reported at temperatures from 35 to 50°C (Antonini et al., 2011, Laureni et al., 2013, Liu et al., 2015), while the use of lower temperatures (35–40°C) with evaporation is possible with the increase of vacuum (pressure 5–7 bars, Bonmatí and Flotats, 2003b, Chiumenti et al., 2013). Additionally, the full-scale applications also reduce heat consumption using heat exchangers to recycle the process heat.

Table 21. The energy balance of a theoretical full scale AD plant treating FW with four digestate liquid treatment options. (Paper IV).

Process	Electricity (MWh_{el}/a)	Heat (MWh_{th}/a)	Total (MWh/a)	Total (% of primary energy)
Energy production				
Primary energy production in AD	-	-	62100	-
Energy in CHP	23598	29187	52785	-
Energy consumption				
AD	5293	6142	11435	18.4
Solid-liquid separation	306	-	306	0.5
Stripping (S1)	406	3727	4133	6.7
Stripping + RO (S2)	590	3727	4317	7.0
Evaporation + RO (S3)	551	3658	4209	6.8
Stripping + evaporation + RO (S4)	922	3727	4649	7.5

-, not applicable

The studied digestate liquid treatment systems produced fertilizer products containing N (ammonium sulfate from stripping) or N, P, and K (concentrate from evaporation, stripping residue from stripping, retentate from RO) in different proportions, which affect their use as fertilizers in agriculture and affect the amount of fertilizers spread on agricultural lands. The evaporation treatment combined with RO (S3) produced the most concentrated nutrient product by concentrating the original FW mass of 60 kt/a into 16 kt/a (Figure 15). The concentrate also showed high nutrient content (18 gN/kgFM, 12 gNH_4-N/kgFM, 0.3 gP/kgFM, 9 gK/kgFM), which, in terms of N and K, was in line with commercial liquid fertilizers intended for, e.g., vegetable fertilization (24 gN/kgFM, 55 gP/kgFM, 40 gK/kgFM, Yara, 2015). Also, the ammonium sulfate from stripping (S1, S2, S4) was a comparable nutrient product with mineral N fertilizers, and hence, the concentrate and ammonium sulfate could potentially replace liquid mineral fertilizers, especially in cases where P fertilization is not needed. With stripping treatment, the challenge is the management of the remaining stripping residue, which still contains P and K with a large liquid volume. One option could be further treatment of the stripping residue, e.g., with RO (as in S2), where the more concentrated retentate could be used in agriculture as NPK fertilizer (Ledda et al., 2013).

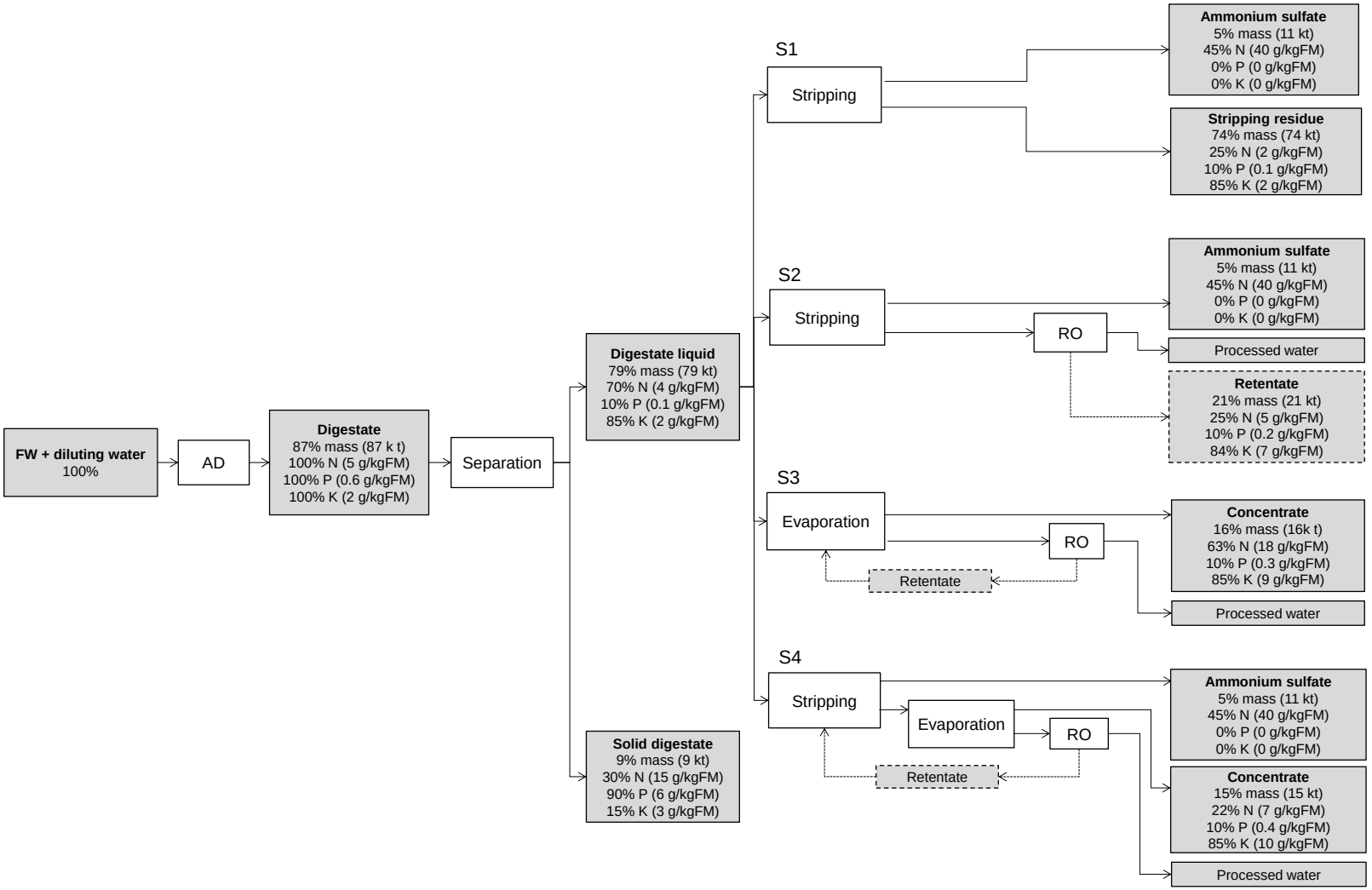

Figure 15. The mass and nutrient balance of the theoretical AD plant treating FW with four digestate liquid treatment options. The chemical additions are not included in mass/nutrient balance (% FW feedstock) but are included in nutrient concentrations (g/kgFM). Gray boxes represent feedstock/product and white boxes represent studied unit operations. (Paper IV).

Conclusions

This thesis showed the high potential of FW as feedstock for AD with its capability to produce high CH_4 yields in optimized conditions. The produced digestate showed suitability to be utilized as nutrient, especially nitrogen, source in crop fertilization independently and after post-treatment. The results were supported by the previous studies with long-term continuous AD and digestate fertilization with FW digestates. Overall, the agronomic usability of the FW digestate is highly dependent on the feedstock characteristic and thus, the sustainable recycling of FW nutrients through the food chain requires understanding about the complexity between FW generation and characteristics, their effect on AD and further on soil and plant systems.

In this thesis it was shown that the use of FW as AD feedstock without dilution is possible, with the TS contents of 20 and 25%. With the undiluted FW, a high OLR of 6 kgVS/m^3d was achieved with relatively high CH_4 yields (400–430 m^3/kgVS) in continuous AD, while the optimum OLR was 3 kgVS/m^3d yielding around 480 m^3/kgVS of CH_4. The trace element supplementation enabled a stable long-term operation and gradual increase in OLRs with no VFA accumulation. The possibility to increase OLRs affects the retention time of the process enabling faster treatment and reduced reactor capacity in AD plants. However, there are still future work possibilities with the balancing of the trace element supplementation and in the finding of suitable co-feedstocks for AD, to provide the sufficient trace elements for the digestion.

The autoclave pretreatment studied in this thesis, affected the FW characteristics and subsequently, the AD performance, where the formation of hardly biodegradable Maillard compounds, from the sugars and proteins, led to 10% lower CH_4 yields during digestion and 50% decreased NH_4-N concentration within the digestate. Due to the pretreatment, the decreased availability of proteins for ammonification could reduce the risk of ammonia inhibition during AD and NH_4-N volatilization from the digestate, thus reducing the fertilizer value of the digestate. Additionally, the lowered H_2S formation enables easier biogas cleaning and security. Along with the hygienization capacity of the autoclave pretreatment, these effects could contribute to the reduction of energy and running cost of the autoclave treatment, balancing the high energy consumption of the pretreatment in 160 °C and 6.2 bars, thus, this matter should be further studied. Further studies are also needed to find the most suitable autoclave pretreatment conditions for the FW, in terms of the total energy balance of the pretreatment and AD, optimized CH_4 production and digestate and biogas quality.

This thesis showed the suitability of FW digestate for fertilizer use in agriculture. The studied FW feedstocks were characterized to have rather similar nutrient and organic matter content, which was shown to produce digestates with increased agronomic value compared with VWAS digestate, in terms of nutrient content and usability as well as biosecurity, including hygienic quality and heavy metal content. In FW digestates, the majority (50–70%) of the N and P were in the soluble and plant available form, and the digestates produced around 5 to 30% higher ryegrass yield compared with a

mineral fertilizer in pot experiments. Overall, the nitrogen mineralization in soil was on the same level in the studied FW digestates, which indicates the availability of the digestate organic nitrogen for microbial degradation in soil. Further studies about the FW biosecurity, related e.g. to organic contaminants and plant pathogens, are still needed to ensure the safe use of FW digestates in agriculture. Additionally, there is a growing need for nutrients in industrial applications, to which FW digestates could provide possible nutrient feedstock, which requires further studies.

In this study, the mass, nutrient and energy balance analysis showed that the integration of the AD of FW and the digestate liquid post-treatment technologies enables the production of concentrated nutrient products rich in N and K. With the combination of different digestate liquid processing technologies, such as evaporation, stripping and reverse osmosis, nutrient products with optimal composition can be produced to correspond the fertilizer demand. Overall, this study indicated the high energy potential of the FW during AD, which can be integrated with heat demanding digestate liquid post-treatment processes (e.g. stripping and/or evaporation) to utilize the heat energy from the CHP unit. This is appropriate especially in situations, where the CHP heat cannot be utilized in e.g. district heating systems. However, as this study evaluated concentration of the liquid digestate based on the theoretical mass and nutrient balances, the characteristics, quality, biosecurity aspects and the fertilizer effect of the nutrient products are still to be studied in practice. Energy and mass balance studies act as a tool for AD plant operators to recognize the process stages which could be improved, and it is thus important to understand the total energy, mass and nutrient balance of an AD plant, to manage the FW treatment and nutrient recycling in energy efficient and sustainable way.

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ORIGINAL PAPERS

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ANAEROBIC DIGESTION OF AUTOCLAVED AND UNTREATED FOOD WASTE

by

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Anaerobic digestion of autoclaved and untreated food waste

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ABSTRACT

Anaerobic digestion of autoclaved (160 °C, 6.2 bar) and untreated source segregated food waste (FW) was compared over 473 days in semi-continuously fed mesophilic reactors with trace elements supplementation, at organic loading rates (OLRs) of 2, 3, 4 and 6 kg volatile solids (VS)/m^3 d. Methane yields at all OLR were 5–10% higher for untreated FW (maximum 0.483 ± 0.013 m^3 CH_4/kg VS at 3 kg VS/m^3 d) than autoclaved FW (maximum 0.439 ± 0.020 m^3 CH_4/kg VS at 4 kg VS/m^3 d). The residual methane potential of both digestates at all OLRs was less than 0.110 m^3 CH_4/kg VS, indicating efficient methanation in all cases. Use of acclimated inoculum allowed very rapid increases in OLR. Reactors fed on autoclaved FW showed lower ammonium and hydrogen sulphide concentrations, probably due to reduced protein hydrolysis as a result of formation of Maillard compounds. In the current study this reduced biodegradability appears to outweigh any benefit due to thermal hydrolysis of ligno-cellulosic components.

1. Introduction

Anaerobic digestion is an efficient technique for the treatment of source segregated biodegradable municipal wastes, e.g. biowastes and food waste (FW), as it recovers energy in the form of biogas for use in combined heat and power (CHP) plants, in vehicles and for grid injection; and also allows recycling of nutrients through application of digestion residues in crop production. Both the Renewable Energy directive (2009/28/EC, EU, 2009) and the Landfill directive (99/31/EC, EU, 1999) have been strong drivers in promoting the use of anaerobic digestion for this application in recent years.

Although co-digestion of FW with sewage sludge and animal manures has been common practice, treatment of FW alone has often proved difficult (Banks et al., 2008; Neiva Correia et al., 2008; Zhang et al., 2012). These difficulties have been attributed to ammonia inhibition resulting from high protein content (Gallert et al., 1998), and are often indicated by accumulation of volatile fatty acids (VFA) (Banks et al., 2012). To achieve stable anaerobic digestion with FW alone, organic loading rates (OLR) are usually maintained at low values: 2.25 kg VS/m^3 d at a hydraulic retention time (HRT) of 80 days in Banks et al. (2011) and from 1 to 4 kg VS/m^3 d (HRT 14–30 days) as reported in Cecci et al. (2003). VFA accumulation at higher OLR has recently been linked to trace element (TE) deficiencies (Banks et al., 2012). When supplemented with TE successful FW digestion has been reported at OLRs of 5 kg VS/m^3 d (Banks et al., 2012) and 6.64 kg VS/m^3 d (Zhang and Jahng, 2012).

Thermal and hydrothermal pre-treatments have been widely studied as a means of hydrolyzing recalcitrant components in a wide range of wastes to make them easier to degrade (Papadimitriou, 2010; Ren et al., 2006; Takashima and Tanaka, 2008); these techniques have also been used as pre-treatments before anaerobic digestion of mixed biowastes (Lissens et al., 2004; Sawayama et al., 1997). One such hydrothermal treatment is autoclaving, where water is used as a reagent at increased temperature and pressure, to hydrolyse and solubilise sugars, starch, proteins and hemicellulose (Papadimitriou, 2010; Ren et al., 2006). Materials pre-treated by autoclaving under various conditions have shown increased methane production in batch tests: digested swine slurry autoclaved at 120 °C showed an increase in CH_4 yield of 115% (Menardo et al., 2011) and autoclaving of mixed kitchen garbage (175 °C, 40 bar, 1 h) increased CH_4 yield by 30% (Sawayama et al., 1997). Improved methane production has also been observed in continuously-stirred tank reactors (CSTRs) treating waste activated sludge (WAS), with 12% and 25% increases after autoclaving at 135 °C and 190 °C, respectively (Bougrier et al., 2007).

In contrast, more aggressive thermal and hydrothermal pre-treatments at higher temperatures (around 180 °C) have been reported to decrease biodegradability and biogas production during anaerobic digestion of WAS and sewage sludge (Bougrier et al., 2008; Pinnekamp, 1989). This is believed to be related to the formation of complex and inhibitory Maillard compounds, produced by reactions between amino acids and carbohydrates

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(Bougrier et al., 2008; Takashima and Tanaka, 2008). Maillard compounds start to form at temperatures above 100 °C depending on the retention time (Müller, 2001; Nursten, 2005), while the formation of more complex compounds, such as acrylamides and other vinylogous compounds, increases at higher temperatures (180 °C, Stadler et al., 2004).

The aim of this study was to evaluate the anaerobic digestion of untreated and autoclaved (160 °C, 6.2 bar) FW at a range of different OLRs (2, 3, 4 and 6 kg VS/m^3 day) in semi-continuously fed intermittently-stirred mesophilic reactors. The biochemical methane potential (BMP) of the feedstocks and the residual methane potential (RMP) of the digestates were also assessed in batch assays.

2. Materials and methods

2.1. Origin and characterization of FW and inocula

The source segregated domestic FW used in the study was collected from the South Shropshire Biowaste digestion plant in Ludlow, UK. Biodegradable bags used for waste collection were removed and the FW material was mixed and divided into two equal portions. One portion was pre-treated at 160 °C and 6.2 bars in a novel double-auger autoclave (AeroThermal Group Ltd., UK) that provides improved mixing and steam penetration; the other portion was left untreated. Both portions were then passed through a macerating grinder (S52/010 Waste Disposer, IMC Limited, UK), packed into 35-l plastic boxes (7 untreated and 8 autoclaved), frozen and shipped at −20 °C to MTT Agrifood Research, Finland.

At MTT the frozen material was chopped into smaller portions corresponding to amounts required for weekly feeding of the digesters, and these smaller portions were again stored at −20 °C. Each week portions of the autoclaved and untreated FW were thawed and stored at 4 °C and used as daily feed. The pH, total solids (TS), volatile solids (VS), ammonium nitrogen (NH_4–N), total Kjeldahl nitrogen (TKN), soluble chemical oxygen demand (SCOD) and VFA content was determined for each new box of feed.

The reactors were inoculated with digestate from a mesophilic CSTR digesting mechanically dewatered sewage sludge (Biovakka Suomi Ltd., Turku, Finland) (Table 1). In the BMP assays inoculum was taken from an anaerobic digester treating municipal and industrial biowastes (Envor Biotech Ltd., Forssa, Finland).

2.2. Semi-continuous trials

Four 11–l stainless steel stirred tank reactors (STRs) (Metener Ltd., Finland) were operated at 37 °C. Stirring (32 rpm) was semi-continuous with 5 s on and 60 s off. The reactors were fed manually five times a week through an inlet tube which extended below the digestate surface, and which was also used for digestate sampling. Digestate overflowed from the reactors by gravity through a u-tube trap to prevent gas escape. Between days 1 and 195 hourly gas volume and methane content were measured using an automatic system in which the produced biogas was collected into a small (~220 ml) gas storage vessel on top of the reactor. From day 195 onwards, due to break down of the automated system, gas volume was measured by water displacement in a volume-calibrated cylindrical gas collector, after which the gas was collected in aluminium gas bags.

Reactors were fed with untreated FW (R1) and autoclaved FW (R3). After 18 days acclimation period with reduced feeding the experiments started at an OLR of 2 kg VS/m^3 day, corresponding to HRT of 117 and 94 days for R1 and R3 respectively. On day 151, after 1.1 (R1) and 1.4 (R3) HRTs, the OLR was raised to 3 kg VS/m^3 day and after 1.3 (R1) and 1.7 (R3) HRTs to 4 kg VS/m^3 day on day 256 (HRT 78 d and 58 d for untreated, 63 d and 47 d for autoclaved FW, respectively).

On day 327 parallel reactors fed on untreated (R2) and autoclaved FW (R4) were started at an OLR of 3 kg VS/m^3 day, using 5.7 l of digestate from R1 and R3 respectively as inoculum. After 2.8 and 3.4 HRTs in reactors R1 and R3 and 1.2 and 1.4 HRTs in reactors R2 and R4, the OLR in all four reactors was further increased to 6 kg VS/m^3 day on day 418, with a corresponding decrease in HRT to 39 d and 31 d in the untreated and autoclaved FW reactors. Most of the data presented below are taken from reactors R1 and R3 due to the longer running period. During days 179–193 reactors R1 and R3 were once a week supplemented with 11 ml of a trace element (TE) solution containing Se (0.2 mg/l) and Co (1.0 mg/l). From day 199 onwards all reactors were given a weekly supplement of two TE solutions, one containing cation elements (mg/l): Al 0.1, B 0.1, Co 1.0, Cu 0.1, Fe 5.0, Mn 1.0, Ni 1.0, Zn 0.2; and the other oxyanions (mg/l): Mo 0.2, Se 0.2 and W 0.2 (Banks et al., 2012). 1 ml of each of these TE solutions was added for each kg of digestate removed from the reactors over the one-week period.

Grab samples of digestate (about 250 g) were taken every two weeks for analysis of TS, VS, SCOD, NH_4–N, TKN, and samples for VFA analysis (about 50 g) were taken once a week. Digestate pH was measured weekly. Larger volumes of digestate were collected on days 130 (2 l), 214 (1 l), 287 (1 l) and 321 (1 l). After removal of these larger samples, daily feeding of the reactors was adjusted to compensate for the reduced volume until the normal operating level was restored.

Table 1
Characteristics of untreated food waste (FW), autoclaved FW and inoculum.

	Control FW	Autoclaved FW	Inoculum
pH	4.96 ± 0.16	5.01 ± 0.12	N/A
TS (g/kg)	247.5 ± 4.7	210.9 ± 18.6	77.3
VS (g/kg)	229.9 ± 4.5	194.6 ± 17.6	43.1
VS/TS (%)	92.9	92.3	55.8
SCOD (g/l)	98.2 ± 6.5	117.5 ± 10.3	11.9
TVFA (g/l)	3.1 ± 0.6	2.2 ± 0.2	2.4
TKN (g/kg)	7.4 ± 0.3	6.8 ± 0.3	4.9
NH_4–N (g/kg)	0.32 ± 0.12	0.41 ± 0.10	2.4
Fe (g/kg$_{TS}$)	0.13 ± 0.01	22.73 ± 12.54	N/A
SMP (m^3 CH_4/kg$_{vs}$)	0.501 ± 0.020	0.445 ± 0.001	N/A
SMP (m^3 CH_4/kg$_{TS}$)	0.462 ± 0.019	0.408 ± 0.001	N/A
SMP (m^3 CH_4/kg$_{FM}$)	0.112 ± 0.005	0.084 ± 0.0001	N/A
Density (kg/l)	1.064 ± 0.0042	1.063 ± 0.0002	N/A

N = 24 for pH, N = 8 for TS, VS, SCOD, TVFA, TKN, NH_4–N, N = 2 for specific methane potentials (SMPs) and Fe, N = 3 for density.
N/A, not available.

2.3. Biochemical and residual methane potential assays

BMP and RMP assays were performed at 37 °C using automated testing equipment (Bioprocess Control Ltd., Sweden). The assays were mixed mechanically (84 rpm) for one minute per hour. Carbon dioxide was absorbed by NaOH before the automated gas volume measurement, which was based on liquid displacement. Assays were conducted in duplicate or triplicate, each with a total liquid volume of 400 ml (BMP) or 200 ml (RMP assays). The inoculum to substrate ratio in BMP assays was 1:1 on a VS basis. $NaHCO_3$ (3 g/l) was used as a buffer and if the pH was lower than 7.5 it was adjusted to around 8 with 3 M NaOH. In RMP assays digestates from the STR reactors were incubated without inoculum. The results are given as average values of the triplicate or duplicate assays.

2.4. Analyses and calculations

TS and VS were determined according to SFS 3008 (Finnish Standard Association, 1990) and NH_4–N according to McCullough

(1967). TKN was analysed by a standard method (AOAC, 1990) using a Foss Kjeltec 2400 Analyzer Unit (Foss Tecator AB, Höganäs, Sweden), with Cu as a catalyst. For soluble COD analysis FW samples were diluted 1:10 with distilled water, and agitated for 1 h. Diluted FW and raw 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, 2002). pH was determined using a VWR pH100 pH-analyzer (VWR International). Iron concentration was analysed according to Luh Huang and Schulte (1985) using inductively coupled plasma emission spectrometry (ICP-OES) (Thermo Jarrel Ash Iris Advantage, Franklin, USA).

Samples for VFA analysis were centrifuged (1831× g, 10 min) and filtered with Chromafil GF/PET-20/25 filters. Concentrations of acetic, propionic, iso-butyric, n-butyric, iso-valeric, valeric and caproic acids were determined using a HP 6890 gas chromatograph with an HP 7683 autosampler (Hewlett-Packard, Little Falls, USA) and GC ChemStation Rev. B.03.02 software. The GC was fitted with a 10 m × 0.53 mm × 1 μm HP-FFAP capillary column (Agilent Technologies, USA) and a flame ionisation detector with helium as a carrier gas (9 ml/min). Oven temperatures were 60–78 °C (25 °C/min), isothermal 1 min, 150 °C (7.5 °C/min) and 25 °C/min to 180 °C with 3 min final time. The injector and detector temperatures were 220 °C and 280 °C, respectively.

From day 1 to 195 methane composition was determined automatically during emptying of the gas storage vessel by infrared analysis (ExTox Gasmess-Systeme GmbH, Germany). From day 195 to 314, gas composition was analysed using a portable Combimass GA-m gas analyzer (Binder Engineering GmbH, Germany), and during days 315–446 the infrared measuring equipment was used.

The reactor was fed for 5 days a week, but the OLR in kg VS/m^3 day is expressed as the average daily weight of substrate fed to the reactor over a one-week period. HRT was calculated based on feedstock densities. All biogas and methane yields were converted to STP conditions (0 °C, 100 kPa) according to the ideal gas law. Methane yields in the RMP assays were calculated in two ways; by dividing the cumulative methane production by the (1) VS of the added digestate and (2) by the ratio of VS of the added digestate and the VS of the feed of the semi-continuous reactors at the time of digestate sampling. The latter enables direct comparison of the methane yield in the RMP with that in the reactors. Free ammonia (NH_3–N) concentrations were calculated according to Anthonisen et al. (1976):

$$NH_3{-}N = (NH_4{-}N \times 10^{pH})/((K_b/K_w) + 10^{pH}), \quad (1)$$

where K_b is the ammonia ionisation constant and K_w the ionisation constant of water at 37 °C.

3. Results and discussion

3.1. Material characterization

The autoclaved FW appeared much darker than the untreated FW and had a pleasant caramel odor. TS and VS in the autoclaved FW were both about 15% lower than in the untreated FW due to dilution by steam condensation during the autoclave treatment (Table 1). TKN on fresh matter basis was lower in the autoclaved FW (6.8 ± 0.3 g N/kg) than in untreated FW (7.4 ± 0.3 g N/kg). The autoclaved FW had about 22% higher NH_4–N and 16% higher SCOD, indicating that autoclaving had solubilised some organic nitrogen and carbon components. Total VFA concentrations were lower in the autoclaved material (2.2 ± 0.2 g/l) than in the untreated FW (3.1 ± 0.6 g/l) suggesting either that some VFA had volatilised during or after autoclaving, or that some acidification of the untreated material had occurred.

Changes in the chemical composition of materials during autoclave treatment are dependent on the temperature as well as the materials used. In this study autoclaving conditions of 6.2 bars and 160 °C were used. Increased concentrations of NH_4–N and solubilisation of carbohydrates have previously been reported after autoclave treatment of dewatered sewage sludge (175 °C, 20 bar), with an increase from 2.6 to 3.2 g NH_4–N/l (Inoue et al., 1996); temperatures above 90 °C have also been reported to increase ammonia concentrations from 0.35 gN/l to 0.7 gN/l in WAS (Bougrier et al., 2008).

3.2. BMP assay

The 35-day BMP value for untreated FW was 0.501 ± 0.020 m^3 CH_4/kg VS, while that for autoclaved FW was 0.445 ± 0.001 m^3 CH_4/kg VS (Fig. 1, Table 1). The lower methane yield of the autoclaved FW could be explained by Maillard reactions. Support for the occurrence of these is given by the darkening in colour of the autoclaved FW and the caramelised odor, while the increase in SCOD provides evidence of increased solubilisation of carbon compounds. Similar phenomena have also been observed with autoclaved WAS (Bougrier et al., 2008) and municipal solid waste (Takashima and Tanaka, 2008). In other studies higher methane yields have been reported after similar thermal treatments (Lissens et al., 2004), but this can be attributed to the improved availability of the ligno-cellulosic materials; and when these form a large proportion of the waste the resulting increase may far exceed any decrease due to Maillard compounds. In contrast where ligno-cellulosic content is low, as in this type of food waste (Zhang et al., 2012) reductions in methane yield may result.

3.3. Semi-continuous operation

3.3.1. Effect of loading rate on methane yields

Process parameters from the whole experimental period (days 1–473) are shown in Fig. 2 and detailed results from the last four weeks of stable operation at each OLR are presented in Table 2. Operation was considered stable when variations were <0.2 units in pH, <90 mg/l in VFA and <1.8% in CH_4.

Throughout the experimental period specific methane yields were 5–10% higher for untreated FW than for autoclaved FW. The methane yields at OLR 2 kg VS/m^3 day were on average 0.443 ± 0.038 and 0.373 ± 0.037 m^3 CH_4/kg VS for untreated (R1) and autoclaved FW (R3), respectively. The highest yield for untreated FW was observed at OLR 3 kg VS/m^3 day (0.483 ± 0.013 m^3 CH_4/kg VS) while autoclaved FW produced the

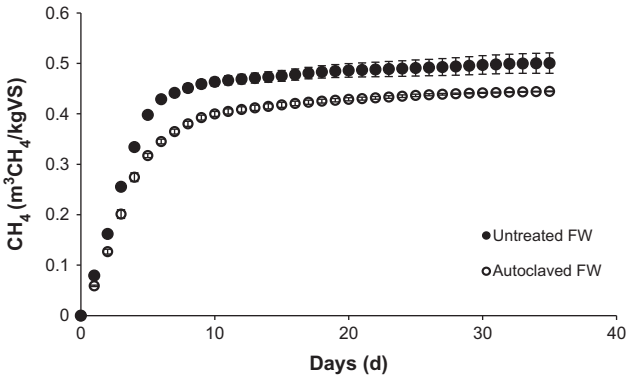

Fig. 1. Biochemical methane potential (BMP) and standard deviation of untreated and autoclaved food waste (FW) in 35-day assays.

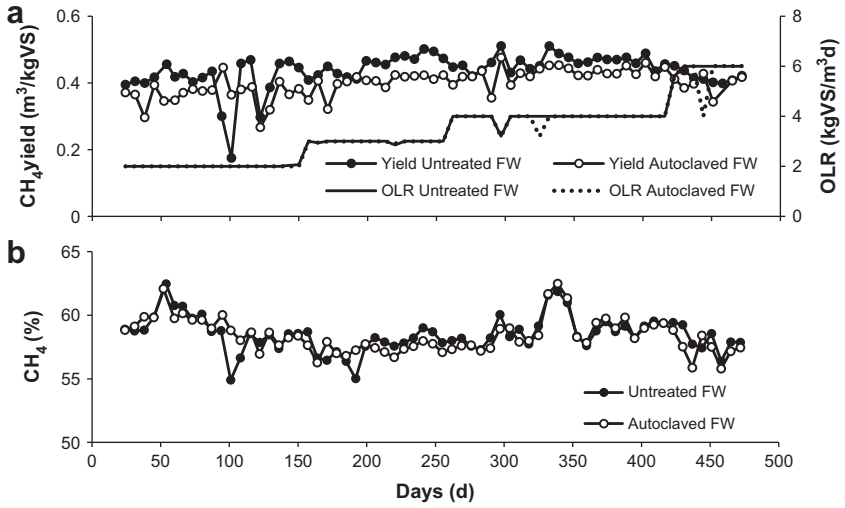

Fig. 2. Methane yields and contents in reactors treating untreated food waste (FW) and autoclaved FW during the semi-continuous operation with OLRs (organic loading rate) of 2 to 6 kg VS/m^3 d.

Table 2
Reactor characteristics during the last 4 weeks of each organic loading rate (OLR, kg VS/m^3 d) periods.

OLR	Reactor	HRT (d)	Specific CH_4 yield (m^3/kg VS)	TS (g/kg)	VS (g/kg)	VS removal (%)	pH	TVFA (mg/l)	TKN (g/kg)	NH_4–N (g/kg)	SCOD (g/l)
2	R1	117	0.443 ± 0.038	69.2 ± 1.7	44.5 ± 0.9	80.6	7.8 ± 0.13	267.5 ± 53.2	7.2 ± 0.1	3.8 ± 0.14	16.0 ± 2.9
	R3	94	0.373 ± 0.037	76.6 ± 2.3	55.6 ± 1.9	71.4	7.6 ± 0.04	132.5 ± 17.1	7.0 ± 0.3	2.1 ± 0.06	14.8 ± 0.1
3	R1	78	0.483 ± 0.013	71.1 ± 2.6	51.4 ± 2.5	77.7	7.8 ± 0.03	188.0 ± 71.9	8.4 ± 0.4	4.2 ± 0.15	15.6 ± 3.1
	R2		0.478 ± 0.009	69.8 ± 2.2	56.1 ± 9.1	75.6	7.8 ± 0.08	108.0 ± 17.9	8.9 ± 0.1	4.1 ± 0.14	23.0 ± 4.3
	R3	63	0.423 ± 0.002	84.0 ± 5.3	66.3 ± 4.4	65.9	7.5 ± 0.02	136.0 ± 26.1	8.2 ± 0.6	2.0 ± 0.05	17.6 ± 2.3
	R4		0.433 ± 0.009	76.4 ± 1.0	63.0 ± 1.1	67.6	7.5 ± 0.03	92.0 ± 23.9	7.9 ± 0.3	1.7 ± 0.03	19.7 ± 0.5
4	R1	58	0.465 ± 0.023	85.2 ± 5.6	64.2 ± 3.7	72.1	7.8 ± 0.07	112.0 ± 25.9	9.0 ± 0.1	3.5 ± 0.03	36.2 ± 0.6
	R3	47	0.439 ± 0.020	86.1 ± 2.6	69.9 ± 2.7	64.1	7.4 ± 0.06	90.0 ± 24.5	8.3 ± 0.5	1.3 ± 0.01	20.3 ± 0.5
6	R1	39	0.405 ± 0.006	102.1 ± 7.3	72.8 ± 4.1	68.3	7.7 ± 0.06	165.0 ± 42.0	9.4 ± 0.2	3.2 ± 0.08	28.3 ± 11.6
	R2		0.435 ± 0.008	90.3 ± 2.8	68.7 ± 2.8	70.1	7.7 ± 0.05	140.0 ± 54.8	9.4 ± 0.1	3.3 ± 0.05	25.9 ± 10.5
	R3	31	0.393 ± 0.044	85.7 ± 1.7	69.1 ± 1.4	64.5	7.2 ± 0.05	108.0 ± 35.6	7.8 ± 0.1	1.2 ± 0.07	18.2 ± 2.0
	R4		0.383 ± 0.013	88.3 ± 5.4	72.0 ± 3.1	63.0	7.3 ± 0.06	110.0 ± 21.6	8.2 ± 0.3	1.2 ± 0.13	18.7 ± 3.0

N/A, not available.
N = 2–5, for pH N = 15.

highest yield at OLR 4 kg VS/m^3 day (0.439 ± 0.020 m^3 CH_4/kg VS). When the OLR was further increased to 6 kg VS/m^3 day methane yields decreased by 12% and 11% in untreated FW and autoclaved FW, respectively. The specific methane yield for autoclaved FW was lower at OLR 2 kg VS/m^3 day than at higher OLRs, which could possibly indicate some acclimatisation. This was not seen in the untreated FW where the lowest specific methane yield occurred at OLR 6 kg VS/m^3 day, which could indicate retarded hydrolysis as no increased SCOD nor VFA was detected. At OLR 6 kg VS/m^3 day the difference in methane yields between the parallel (R2 and R4) and original (R1 and R3) reactors was <7% (Table 2).

In reactors R1 and R3 relatively long operating times were applied, to allow the process to stabilise between incremental increases in OLR. Using this approach, stable digestion of both autoclaved and untreated FW was achieved at the relatively high OLR of 6 kg VS/m^3 day. It was also shown, however, that when an inoculum acclimated to the feedstocks was used in R2 and R4, the OLR could be rapidly increased without operational disturbances such as VFA accumulation. The maximum loading rates applied were similar to the 6.64 kg VS/m^3 day achieved by Zhang and Jahng (2012) and higher than the 5 kg VS/m^3 day of Banks et al. (2012). Both of these long-term digestion studies used trace elements supplementation, as did the present study.

As far as is known, this is the first study to report anaerobic digestion of autoclaved food waste in a semi-continuously fed system. Methane yields of 0.483 ± 0.013 and 0.423 ± 0.002 m^3 CH_4/kg VS for the untreated and autoclaved FW at OLR 3 kg VS/m^3 day are in good agreement with previous studies, where a full-scale digester fed on the same type of source-segregated household food waste at an average OLR of 2.5 kg VS/m^3 day yielded 0.402 m^3 CH_4/kg VS (Banks et al., 2011). Earlier pilot-scale studies gave an average of 0.390 m^3 CH_4/kg VS, but using a different source of source-segregated domestic food waste at higher OLR (3.5 to 4 kg VS/m^3 day), and without TE supplementation (Banks et al., 2008). Laboratory-scale FW digestion with TE supplementation was reported to yield 0.352-0.439 m^3 CH_4/kg VS at an OLR of 6.64 kg VS/m^3 day by Zhang and Jahng (2012); while in the study by Banks et al. (2012) the methane yield for TE supplemented FW was 0.435 m^3 CH_4/kg VS.

The maximum methane yields for untreated and autoclaved FW in the semi-continuous trials were 0.483 ± 0.013 and 0.439 ± 0.020 m^3 CH_4/kg VS respectively. These were slightly lower than the BMP values in each case. The results therefore strongly indicate that even after long periods of operation no significant acclimatisation that could improve the biodegradability of compounds produced in the autoclaving process had taken place.

With mixed biowastes, the benefits of increased biogas production due to improved degradation of ligno-cellulosic materials may outweigh any losses in biodegradability as a result of formation of recalcitrant compounds during thermal treatment. FW, however,

has a relatively low ligno-cellulosic fibre content compared to other municipal biowaste components (e.g. garden or yard waste, paper and card), and in the present study the net effect of treatment was a reduction in specific methane yield. This balance may however change with different autoclaving conditions, and in particular a lowering of temperature may produce more favourable results.

3.3.2. Digestion parameters

Results for pH, VFA, TS, VS, SCOD, NH_4–N, TKN are presented in Table 2 and Fig. 3. pH in the untreated FW reactor remained around 7.8 throughout the experimental period, while with autoclaved FW the pH decreased from pH 7.6 at OLR 2 kg VS/m^3 day to 7.3 at OLR 6 kg VS/m^3 day.

At an OLR of 2 kg VS/m^3 day, total VFA concentration in both reactors remained under 250 mg/l. When the OLR was increased to 3 kg VS/m^3 day, VFA in the untreated FW reactor increased to 2400 mg/l by day 153, and consisted mainly of acetic (about 85%) and propionic acids (about 10%). In the autoclaved FW reactor VFA concentration showed smaller increases with peaks of 1500 mg/l on day 139 (consisting 98% of acetic acid) and 910 mg/l on day 160 (27% acetic acid and 65% propionic acid). The relatively large samples (2 l) taken from the reactors on day 130 could have contributed to these increases in VFA concentration, but similar removals of digestate at later stages in the experimental run did not have this effect. VFA concentrations reduced to under 200 mg/l in both reactors by day 214, shortly after the introduction of trace element additions of selenium and cobalt on day 179 and full TE supplementation on day 199. This behaviour is consistent with previous reports of responses to TE supplementation where the VFA increase was linked with the loss of electron transfer interspecies during digestion (Banks et al., 2012).

TS, VS and TKN contents in both reactors gradually increased during the experimental period, with TS increasing from under 70 to over 80 g/kg. Despite the lower feedstock solids concentration, the solids content in the autoclaved FW reactor was slightly higher than in the untreated FW up to the end of OLR 4 kg VS/m^3 day. After OLR was increased to 6 kg VS/m^3 day there was an increase in solids concentrations in the untreated FW reactor, which was not apparent with the autoclaved FW. The initial TKN concentration in both reactors was 4.9 g N/kg and showed a similar increase to ~8 g N/kg by around day 200. TKN in the untreated FW reactor continued to increase until around day 300 at which point it stabilised at ~9 g N/kg, whereas for the autoclaved FW it remained at ~8 g N/kg. The differences in TKN reflected the differences in feedstock concentrations. The increases in solids content

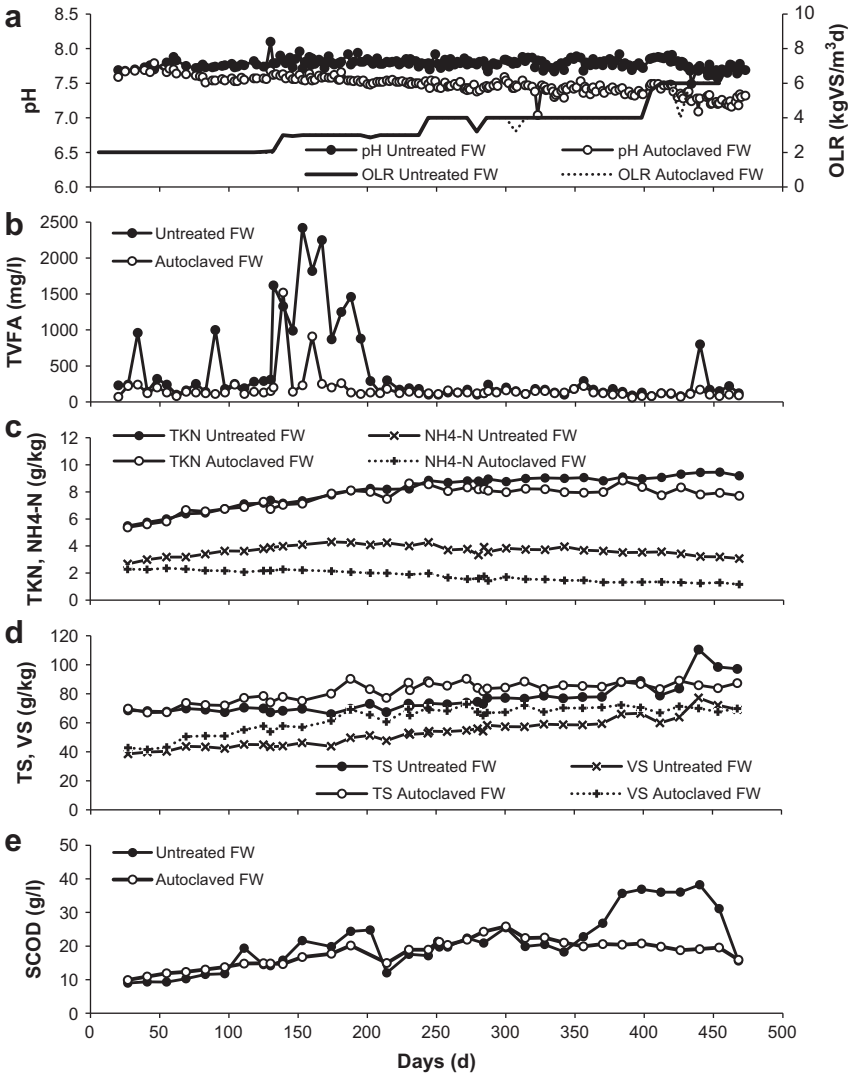

Fig. 3. Chemical characteristics (pH, TVFA, TKN, NH_4–N, TS, VS, SCOD) of untreated food waste (FW) and autoclaved FW reactor contents during the semi-continuous operation with OLRs (organic loading rate) of 2 to 6 kg VS/m^3 d.

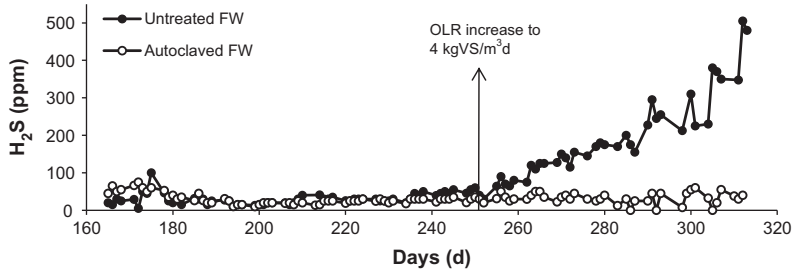

Fig. 4. H_2S contents in reactors treating untreated food waste (FW) and autoclaved FW during days 166–314.

were most likely associated with the increase in loading, although it is possible that some accumulation was due to stratification despite the intermittent mixing, as surplus digestate was discharged from an overflow at the top of the reactor. Mass balance calculations affirmed, in the beginning of OLR 4 kg VS/m^3 day, that accumulation of TKN was taking place.

The SCOD concentration in both reactors increased from around 10 g/l to over 20 g/l during the first 300 days of operation, then stabilised in the autoclaved FW reactor. In the untreated FW reactor the SCOD increased sharply to ~36 g/l for over 50 days then decreased equally sharply in the end of the run: these variations did not correspond to changes in OLR and were not accomplished with changes in methane yield nor digestate VFA. Total VFA concentrations accounted for only 0.5–2% of the SCOD. A probable explanation for the general increase in SCOD in both reactors is an increase in the quantity of soluble microbial products present in the digestate; this phenomenon has previously been observed with solid substrates and at long retention times (Kuo et al., 1996; Rinćon et al., 2012).

3.3.3. Ammonium and ammonia

NH_4–N concentration in the untreated FW reactor increased during the first ~170 days from 2.4 (inoculum) to 4 g/kg and then showed a very gradual decrease to around 3 g/kg by the end of the experimental run. This decrease could be associated with the increase in microbial biomass (Lindorfer et al., 2011) or in soluble microbial products caused by the increasing OLR. In the autoclaved FW reactor, however, NH_4–N decreased from 2.4 to about 1.2 g/kg by the end of the experimental period. The low NH_4–N concentrations in the autoclaved FW reactor were probably mainly due to the effect of autoclaving and the formation of Maillard compounds from the reaction of proteins with carbohydrates (Bougrier et al., 2007, 2008). Free ammonia concentrations in the reactors were calculated, but NH_3 remained below 0.30 g/kg in untreated FW and below 0.10 g/kg in the autoclaved FW reactor.

The pH value in the untreated FW reactor rose to around 7.8 by day 55 and remained relatively stable until the OLR was raised to 6 kg VS/m^3 day, at which point it fell very slightly. In the autoclaved reactor after a slight initial rise pH decreased during the experimental run to a final value of around 7.3. These pH values reflect the relative NH_4–N concentrations in each case, as NH_4–N provides buffering capacity (Procházka et al., 2012). High NH_4–N concentration can also inhibit the digestion process, but this is greatly dependent on the feedstock materials and acclimation times (Chen et al., 2008; Procházka et al., 2012). In the present study, after TE supplementation was introduced, there was no evidence of the VFA accumulation that is often associated with ammonia toxicity, and the free ammonia concentrations were similar to those previously observed in FW digestion (Zhang et al., 2012).

3.3.4. Gas composition

The biogas methane content in both autoclaved and untreated FW digesters was similar and ranged between 55 and 63% during the experiment, with an average of around 58% (Table 2, Fig. 2). It did not appear to be affected by changes in applied OLR. In contrast, in a study by Zhang and Jahng (2012) on FW digestion the methane content was found to decrease from 53% to 48% as the OLR was gradually increased from 2.19 to 6.64 kg VS/m^3 day.

Hydrogen sulphide concentration was monitored between days 166 and 313 while the reactors were operated at OLR 3 and 4 kg VS/m^3 day (Fig. 4). H_2S concentrations at OLR 3 kg VS/m^3 day were <100 ppm in the untreated FW reactor and <75 ppm in the autoclaved FW reactor. Shortly before the OLR was increased to 4 kg VS/m^3 day the H_2S concentration in the untreated FW reactor began to increase, and reached 480 ppm by day 314 at which point monitoring ceased; while in the autoclaved FW reactor H_2S content remained <60 ppm. H_2S was also monitored at the OLR of 6 kg VS/m^3 day (days 448–473) and concentrations were 751 ± 182 ppm in the untreated FW reactors (R1 and 2) compared to 63 ± 4 ppm in the autoclaved FW reactors (R3, R4).

In the autoclaved FW reactors H_2S concentrations remained low, probably due to the effect of autoclaving on proteins in the food waste, which may have reduced the availability of sulphur.

Table 3
Residual methane potentials (RMPs), total methane yield and VS removals of food waste digestates after organic loading rates (OLRs, kg VS/m^3 day) 2, 4 and 6 in the stirred tank reactors (STRs).

OLR	Reactor	RMP (m^3/kg VS)	$RMP_{original}$ (m^3/kg VS_{feed})[a]	Total CH_4 yield in STR + RMP (m^3/kg VS_{feed})[a]	VS removal in STR + RMP (%)
2	R1	0.069 ± 0.005	0.013 ± 0.0009	0.456	85.1
	R3	0.063 ± 0.002	0.017 ± 0.0006	0.390	75.3
4	R1	0.065 ± 0.001	0.017 ± 0.0004	0.482	80.9
	R3	0.057 ± 0.002	0.020 ± 0.0006	0.459	67.4
6	R1	0.105 ± 0.002	0.032 ± 0.0005	0.437	76.9
	R3	0.095 ± 0.012	0.034 ± 0.0045	0.427	69.3

N = 2–3.
[a] Results calculated according to VS fed to STRs.

The low H_2S concentration could also be due in part to precipitation through the formation of iron sulphides. The iron content in the autoclaved FW was 170 times higher than in the untreated FW (Table 1), possibly due to metal contamination from the autoclaving apparatus. O'Flaherty et al. (1998) showed that sulphate-reducing bacteria (SRBs) have an optimum pH slightly higher than that of methanogenic archaea, and hence the higher pH in the untreated FW reactors may have favoured the growth of SRBs causing increased H_2S concentrations. Decreasing HRT will also give SRB an additional competitive advantage.

3.4. Residual methane potential assays

50-day RMP values were determined at the end of each period of reactor operation at OLRs 2, 4 and 6 kg VS/m^3 day (Table 3). The RMPs increased with the increasing OLRs and decreasing HRTs from 0.069 ± 0.005 m^3 CH_4/kg VS to 0.105 ± 0.002 m^3 CH_4/kg VS with the untreated FW and from 0.063 ± 0.002 m^3 CH_4/kg VS to 0.095 ± 0.012 m^3 CH_4/kg VS with the autoclaved FW (OLR 2 to 6 kg VS/m^3 day). However, RMPs after operation with OLR 4 kg VS/m^3 day were 6 and 10% lower in untreated and autoclaved FW compared to OLR 2 kg VS/m^3 day reflecting the highest CH_4 yields obtained with OLRs 3 and 4 kg VS/m^3 day in STRs. Also few days longer storage time might have affected the RMPs after OLR 4 kg VS/m^3 day allowing materials to slightly degrade before the RMP start.

Overall, when results were calculated per VS of FWs fed to the STRs, $RMP_{original}$ increased total methane yield of the semi-continuous reactors by 2.9–4.7% with the untreated FW and by 4.3–5.2% with the autoclaved FW (Table 3). The calculated total methane yield with the untreated FW was, after OLRs 2, 4 and 6 kg VS/m^3 day, 3.6–12.6% lower than the BMP value (0.501 m^3 CH_4/kg VS) being closest after OLR 4 kg VS/m^3 day and thus reflecting the specific yields in STRs. Autoclaved FW showed similar STR reflecting behavior but after OLR 4 kg VS/m^3 day the calculated total methane yield was 3.1% higher than the BMP value (0.445 m^3 CH_4/kg VS). The VS removals were not cohesive with the calculated total methane yields, which could partly be explained with deviations between samples. The results suggest that in both materials there was still a small part of biodegradable material after semi-continuous reactors and the amount increased with the increasing OLRs and decreasing HRTs.

4. Conclusions

Stable digestion of untreated and autoclaved FW was possible in TE-supplemented mesophilic reactors at OLRs up to 6 kg VS/m^3 d, with yields of 0.435 and 0.393 m^3 CH_4/kg VS, respectively. Using an acclimated inoculum allowed rapid increases in OLR without process disturbance. Untreated FW showed a higher specific methane yield than autoclaved FW at all OLRs and in batch assays. This difference may be due to the formation of Maillard compounds, with the resulting reduction in biodegradability apparently outweighing any benefits from thermal hydrolysis of ligno-cellulosic components under the autoclaving conditions used. Biogas H_2S concentrations were much lower in reactors treating autoclaved FW.

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II

CHARACTERISTICS AND AGRONOMIC USABILITY OF DIGESTATES FROM LABORATORY DIGESTERS TREATING FOOD WASTE AND AUTOCLAVED FOOD WASTE

by

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Characteristics and agronomic usability of digestates from laboratory digesters treating food waste and autoclaved food waste

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ABSTRACT

Digestate characteristics such as organic and nutrient content, hygienic quality and stability are valuable measures when evaluating the use of food waste (FW) digestate as organic fertiliser. This study compared the characteristics of FW and autoclaved (160 °C, 6.2 bar) FW and their digestates from laboratory-scale reactors. Decreased ammonification and low ammonium nitrogen content were observed in the digestate from an autoclaved FW reactor due to autoclave treatment of FW, which affected the nitrogen-containing molecules by formation of Maillard compounds. The methane potential of autoclaved FW and its digestate was decreased by 40% due to reduced microbial activity as microbes were not able to adapt to the conditions within a reactor fed with autoclaved FW. Both studied materials were suitable for agricultural use in terms of their nutrient content, hygienic quality and stability, and thus the decrease in ammonium nitrogen in digestate from an autoclaved FW reactor supported the use of digestate as soil amendment rather than fertiliser.

1. Introduction

It is estimated that globally one third of the food produced for consumption becomes food waste (FW) during production, processing, distribution and consumption (Gustavsson et al., 2011). In Europe the total FW quantity produced each year is 90 million tonnes (180 kg per capita), of which an estimated 38 million tons (76 kg per capita) is generated in households (European Commission, 2010), while in the USA 36 million tons (120 kg per capita) of residential and commercial FW was produced in 2011 (US EPA, 2013). FW composition derived from households varies seasonally and geographically. In a study from Finland, Italy, Portugal, and the UK, the average household food waste consisted mainly of fruit and vegetable waste (>50%) and to a lesser extent of beverages (coffee filters and tea bags, 9%), meat and fish (6%), bread and bakery (5%) and mixed meals (12%), and had relatively high protein (16–55% of VS, volatile solids) and fat (15–30% of VS) contents (Valorgas, 2011).

In Europe anaerobic digestion (AD) together with composting are increasingly used as treatment methods for organic wastes such as FW due to the EU Waste Framework Directive (2008/98/EC, European Parliament and the Council, 2008), which obligates member states to carry out source segregation and safe treatment of biowastes. With AD, energy- and nutrient-rich organic compounds can be digested to simultaneously produce fertilisers/soil amendments, renewable energy and/or fuel for transport. When used as fertiliser the nutrients in FW digestate can be returned to agriculture to close the nutrient cycle, thereby reducing the need for inorganic fertilisers, and their use as soil amendments improves the physical, chemical and biological properties of the soil. In the EU digestate use in agriculture is regulated by national legislations deriving from EU regulations concerning animal by-products and their digestion residues (European Council, 2011; European Parliament and the Council, 2009). In addition to the hygienic quality, the fertilising effect of the mineral and organic forms and plant availability of nutrients are essential when considering the usefulness of the digestate as soil fertiliser/amendment. Determination of the stability – e.g. residual methane potential of digestates, emissions during digestate storage and use – can be minimised and the energy production of AD optimised.

In biogas production, pretreatment of food waste affects the characteristics of the FW digestate. The aim of pretreatment is to enhance biodegradability and methane yields and to improve the hygienic quality of the material. Thermal autoclave treatment

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(130–180 °C) has been observed to lower the methane conversion of high protein-containing substrates (Cuetos et al., 2010; Pinnekamp, 1989) such as FW by 5–10% during semi-continuous mesophilic AD (Tampio et al., 2014). At higher autoclaving temperatures organic material hydrolyses and solubilises; however, toxic (Cuetos et al., 2010) or hardly biodegradable compounds such as Maillard compounds can also be formed through reactions between sugars and amino acids (Bougrier et al., 2008; Monlau et al., 2013), which further affects the AD process and the digestate quality, e.g. decreasing the ammonium nitrogen content of the digestate (Tampio et al., 2014). However, more detailed research about the effects of these compounds and nitrogen transformation during AD of pretreated FW is needed to evaluate the end-use value of the digestate.

The aim of this study was to compare the characteristics, quality and agronomic usefulness of FW and autoclaved FW (AFW) digestates. For that purpose digestates from laboratory semi-continuously stirred tank reactors were characterised for hygienic quality, nutrient content as well as residual methane and ammonification potentials. Furthermore, as reference the ammonification and residual methane potentials were compared with digestate from a full-scale AD plant.

2. Materials and methods

2.1. Origin of food waste and digestates

The FW used in this study was source-segregated domestic FW collected from the South Shropshire Biowaste digestion plant in Ludlow, UK. FW was divided into two portions and subsequently one portion was pre-treated with a novel double-auger autoclave (AeroThermal Group Ltd, UK) at 160 °C and 6.2 bars (referred to as AFW) while the other portion was left untreated (referred to as FW). FW portions were then passed through a macerating grinder (S52/010 Waste Disposer, IMC Limited, UK), frozen and shipped to Natural Resources Institute Finland where the FW samples were melted and stored at 4 °C before use as described in more detail in Tampio et al. (2014).

Three different digestates were used in this study. Two digestates were collected from laboratory stirred tank reactors fed with FW (digestate referred to as FW digestate) and AFW (digestate referred to as AFW digestate). The reactors were fed through a feeding inlet tube extended below the digestate surface, and digestate overflowed by gravity through a u-tube trap to prevent gas escape. For this study the digestates were sampled both from the overflow digestate and through the inlet tube from the reactor. The reactors were operated up to 473 days. Organic loading rates were gradually increased from 2 to 6 kg VS/m^{3}d, decreasing the hydraulic retention times from 117 and 94 to 39 and 31 days in reactors treating FW and AFW, respectively (Table 1). Starting from runs with organic loading rate of 3 kg VS/m^{3}d the reactors were supplemented with trace elements according to Banks et al. (2012) with element concentrations of Al (0.1 mg/l), B (0.1 mg/l), Co (1.0 mg/l), Cu (0.1 mg/l), Fe (5.0 mg/l), Mn (1.0 mg/l), Ni (1.0 mg/l), Zn (0.2 mg/l), Mo (0.2 mg/l), Se (0.2 mg/l) and W (0.2 mg/l). Reactor configuration and feeding practices are described in more detail in Tampio et al. (2014).

Digestates were stored at 4 °C for a maximum of one week (characterisation and hygiene analysis) or up to 4 weeks (batch assays) before use. Digestates used in characterisation studies were from organic loading rates of 2, 3 and 6 kg VS/m^{3}d (total organic carbon was analysed during organic loading rate 4 kg VS/m^{3}d) while digestate samples for the hygiene analyses were collected during organic loading rates of 4 kg VS/m^{3}d (4 samples) and 6 kg VS/m^{3}d (3 samples) (Table 1). The food waste samples were collected simultaneously with the digestates (6–7 samples) and thawed and stored in a freezer (4 °C) for 1–5 days prior to analyses.

Table 1
Source of digestates used for characterisation, hygiene analyses and batch assays. Organic loading rate (OLR) and hydraulic retention time (HRT) of the reactors and supplementation of trace elements (TEs) are shown for time of sampling as well as the sampling procedures. FW = food waste, AFW = autoclaved food waste.

Digestate	OLR	HRT	TE	Sampling feeding inlet	Sampling overflow
FW	2	117	–	Characterisation	–
AFW		94	–	Characterisation	–
FW	3	78	+	Characterisation	–
AFW		58	+	Characterisation	–
FW	4	63	+	Hygiene	Batch assays
AFW		47	+	Hygiene	–
FW	6	39	+	Hygiene	–
AFW		31	+	Characterisation, hygiene	Batch assays
Reference	N/A	N/A	–	–	Batch assays

–, no trace elements addition or sampling.
+, trace element addition.
N/A, not available.

The third digestate (referred to as reference digestate) used in this study originated from a full-scale mesophilic anaerobic digester treating municipal and industrial biowastes (Envor Biotech Ltd, Forssa, Finland).

2.2. Batch assays

The batch assays for biochemical methane potentials and for residual methane and ammonification potentials were performed in duplicate or triplicate 0.5 l bottles with a total liquid volume of 400 ml using automated testing equipment (Bioprocess Control Ltd, Sweden) at 37 °C. The contents were mechanically mixed (84 rpm) for 1 min per hour, and CO_2 from the produced biogas was fixed by NaOH prior to automated, liquid displacement-based gas volume measurement.

Batch assays were performed with the digestates alone (residual methane potential) and using the digestates as inocula and FW and AFW as substrates (biochemical methane potential, Table 1). In all assays with FW and reference digestates the volume of inoculum was 300 g and the substrate to inoculum ratios on a VS basis 1:1. With AFW digestate assays 340 g of inoculum was used with a VS/VS ratio of 1:2. In all assays distilled water was added to obtain 400 ml liquid volume. pH (if lower than 7.3) was adjusted to around 8 with 3 M NaOH and in the case of the reference digestate inoculum $NaHCO_3$ (3 g/l) was added as a buffer. Finally, the contents of all bottles were flushed with N_2 to obtain anaerobic conditions.

2.3. Analyses and calculations

From fresh samples, total and volatile solids (TS and VS) were determined according to SFS 3008 (Finnish Standard Association, 1990) and ammonium nitrogen (NH_4–N) according to McCullough (1967). Total Kjeldahl nitrogen (TKN) was analysed by a standard method (AOAC, 1990) using a Foss Kjeltec 2400 Analyser Unit (Foss Tecator AB, Höganäs, Sweden), with Cu as a catalyst. For soluble chemical oxygen demand analysis FW samples were diluted 1:10 with distilled water, and agitated for 1 h. Diluted FW and digestate samples were centrifuged (2493 × *g*, 15 min) after which the supernatant was further centrifuged (16,168 × *g*, 10 min) and stored in a freezer, then thawed before analysis according to SFS 5504 (Finnish Standard Association, 2002). pH was determined using a VWR pH100 pH-analyser (VWR International). Soluble-N was analysed as TKN after 1:15 dilution with distilled water and soluble-P and soluble-K were measured from 1:5 dilution with ICP-OES (inductively coupled plasma optical emission spectrometry).

From dried (60 °C) samples, crude protein by Duma's method was analysed with standard methods (AOAC, 1990) using a Leco FP 428 nitrogen analyser (Leco Corp., St Joseph, USA) and by multiplying the N% by a factor of 6.25. Crude fat was analysed with a Soxcap-Soxtec-Analyser (AOAC, 1990; Foss Tecator Application Note AN 390). For soluble carbohydrate analyses, samples were inverted with 1 N HCl (50 °C, 12 h) and analysed according to Somogyi (1945). NDF (neutral detergent fibre) was analysed with a filtering apparatus according to Van Soest et al. (1991) and both ADF (acid detergent fibre) and lignin (permanganate-lignin) were determined according to Robertson and Van Soest (1981). Hemicellulose content was calculated from the difference between NDF and ADF while cellulose content was calculated from the difference between ADF and lignin. Total-C was analysed by Duma's method according to manufacturer's instructions with a Leco CN-2000 Elemental Analyser (Leco Corp., St. Joseph, MI, USA). For the analysis of total-P and total-K, samples were digested with HNO_3 (Luh Huang and Schulte, 1985) and analysed with ICP-OES according to manufacturer's instructions.

Hygienic quality was analysed using *Escherichia coli*, other coliforms, total coliforms, enterococci, sulphite-reducing clostridia and Salmonella as indicator organisms. Analyses of different coliforms were performed according to Baylis and Patrick (1999) using Harlequin *E. coli*/coliform (LabM) culture medium with 24–48 h incubation time at 37 °C. Enterococci were determined with KF streptococcus agar (incubated 48 h at 44.5 °C) according to SFS-EN ISO 7899 (Finnish Standard Association, 2000) and sulphite-reducing clostridia with sulphite-iron agar (incubated anaerobically 48 h at 37 °C) according to SFS-EN 26461 (Finnish Standard Association, 1993). For the qualitative analyses of Salmonella, samples were pre-enriched in buffered peptone water (37 °C, 16–20 h) and incubated in Rappaport–Vassiliadis broth (42 °C, 24 h). Aliquots from the broth were cultured on Salmonella-selective Rambach and xylose-lysine-decarboxylase agars and incubated at 42 °C for 24 h. If growth was observed, colonies were confirmed with triple sugar iron agar, urea-agar and lysine carboxylase broth (37 °C, 24 h) (ISO, 2002).

All methane yields were converted into the standard temperature and pressure conditions (0 °C, 100 kPa) according to the ideal gas law using ambient temperature and air pressure. In the ammonification batch assays, the starting NH_4–N, total Kjeldahl nitrogen, TS and VS contents in the bottles were calculated according to the mass balances from the original concentrations of FWs and digestates and the amounts used in the assays.

3. Results and discussion

3.1. Food waste characteristics

The studied FW had TS of ca 230 g/kgFM, and VS/TS ratio of 93% while AFW had about 10–15% lower TS and VS, likely due to dilution by condensed water during the autoclave treatment (Table 2). The FW contained proteins up to 220 g/kgTS while fats and soluble carbohydrates were ca 140 and 120 g/kgTS. Cellulose and hemicellulose contents were around 50 g/kgTS and low lignin content, 6 g/kgTS, was observed. The autoclaving affected the organic composition (per TS) by decreasing the soluble carbohydrates by 50% and hemicelluloses by 40% while increasing the lignin content from 6.6 to 81.6 g/kgTS, whereas the effects on other components were minor. AFW also had increased SCOD and lowered VFA, likely due to solubilisation, volatilisation and acidification of material during autoclave treatment.

The protein (220 g/kgTS) and fat (140 g/kgTS) content in the FW corresponded well with previous studies with FWs from Europe where protein and fat contents in FWs have varied between 100 and 260 g/kgTS (Table 3). Cellulose and hemicellulose contents were similar in the source-sorted FW in Ludlow, UK, while the present lignin content of 6 kg/kgTS was 60% lower (Table 3, Zhang et al., 2012). The low lignin content of FW as well as the high standard deviations in lignin observed with both FW samples were probably due to the complex nature of lignin, different analysing methods (Hatfield and Fukushima, 2005) and the heterogeneity of the FW material (Papadimitriou, 2010). The autoclave treatment decreased the soluble carbohydrate content, indicating the formation of Maillard compounds (Liu et al., 2012; Monlau et al., 2013) through reactions between sugars and amino acids (Bougrier et al., 2008; Monlau et al., 2013; Pinnekamp, 1989). The reduction in hemicellulose content was most likely due to the branched structure of the hemicellulose, which enables easier hydrolysis during pre-treatment (Papadimitriou, 2010; Pérez et al., 2002).

3.2. Digestate characteristics

The FW digestate had TS and VS of 67.4 and 45.6 g/kg, while the values were slightly higher in the AFW digestate (78.5 and 50.5 g/kg, respectively, Table 2). AD decreased TS, VS, fats and soluble carbohydrates content and increased cellulose and hemicellulose contents (g/kgTS) similarly with both substrates. However, the lignin content increased nearly tenfold in the FW while in the autoclaved digestate lignin content was doubled. Protein content increased by 15% more with the autoclaved material during AD.

With AFW digestate the protein content and the hemicellulose, cellulose and lignin contents (g/kgTS) were 25–80% higher than for FW digestate while the NH_4–N/TKN ratio was ~30% lower (Table 2). The reduced NH_4–N and NH_4–N/TKN ratio and higher protein contents in the AFW digestate resulted from formation of Maillard compounds during autoclave treatment, which affected the digestate by decreasing protein degradation and leading to reduced fertiliser value.

The content of fibres (cellulose, hemicelluloses and lignin; g/kgTS) increased 30–800% during AD partly due to low biodegradability of the ligno–cellulosic complexes (Pérez et al., 2002), but also indicating some solid material accumulation during the digestion process. The ratio between cellulose (CEL), hemicellulose (HEMI) and lignin (LIGN), CEL + HEMI/LIGN (Eleazer et al., 1997), was used to evaluate the biodegradation of these compounds during autoclaving and AD. For FW, AFW, FW and AFW digestates, the CEL + HEMI/LIGN ratio was 16.3, 1.2, 3.0 and 1.2, respectively. The stable CEL + HEMI/LIGN ratio (1.2) of AFW after AD indicates that the hemicellulose and cellulose had already degraded during autoclaving and could not degrade further during AD. The higher content of hardly degradable cellulose, lignin and proteins in the AFW digestate compared to the FW digestate likely reduced methane production during batch experiments, which supports the results from Tampio et al. (2014) where the methane yield in stirred tank reactors was 5–10% lower with AFW compared to FW.

3.3. Methane and ammonification potentials

First, the residual methane potentials of the FW, AFW and reference digestates were assayed to evaluate the potential recoverable methane and possible emission risk during digestate handling. The FW digestate produced methane more slowly than the AFW and reference digestates; however, it and the reference digestate had higher residual methane potential (around 0.135 m^3CH_4/kgVS) than the AFW digestate (~0.080 m^3CH_4/kgVS). With the FW digestate the cumulative methane potential curve was of a “sigmoid type”, indicating some inhibition (Vavilin et al., 2008). During the assay, the NH_4–N concentration in the AFW digestate

Table 2
Characteristics of food waste (FW) and autoclaved food waste (AFW) as well as FW and AFW digestates. Averages and standard deviations are shown, feed N = 3–4, FW digestate N = 2, AFW digestate N = 3, if not otherwise stated.

Parameter	Unit	Feed		Digestate	
		FW	AFW	FW	AFW
General characteristics					
pH	–	5.2 ± 0.22	5.2 ± 0.21	8.0 ± 0.02	7.7 ± 0.05
TS	g/kgFM	248.6 ± 2.86	215.5 ± 8.66	67.4 ± 0.07	78.5 ± 5.12
VS	g/kgFM	231.1 ± 1.93	198.8 ± 7.50	45.6 ± 2.96	60.5 ± 6.53
VS/TS	%	92.6 ± 0.29	92.5 ± 0.29	67.7 ± 4.33	77.0 ± 3.72
TKN	g/kgFM	7.62 ± 0.33	6.9 ± 0.27	7.8 ± 0.59	7.3 ± 0.52
NH4–N	g/kgFM	0.4 ± 0.14	0.4 ± 0.03	4.07 ± 0.25	1.9 ± 0.41
NH4–N/TKN	%	4.7 ± 1.71	5.3 ± 0.53	52.2 ± 0.66	25.7 ± 7.18
SCOD	g/kgFM	101.7 ± 12.55	112.8 ± 16.19	13.1 ± 1.51	15.3 ± 1.28
VFA	g/kgFM	3.5 ± 0.41	2.2 ± 0.21	0.3 ± 0.01	0.2 ± 0.03
Organic characteristics					
Crude protein	g/kgTS	218.9 ± 17.51	208.6 ± 26.96	311.2 ± 31.82	443.4 ± 36.08
Crude fat	g/kgTS	141.7 ± 9.48	142.5 ± 6.22	56.7 ± 3.39	46.1 ± 6.75
Soluble carbohydrate	g/kgTS	122.7 ± 17.94	59.7 ± 5.38	5.2 ± 0.00	5.2 ± 0.64
Cellulose	g/kgTS	51.5 ± 6.94	62.5 ± 9.62	66.4 ± 16.69	123.5 ± 23.20
Hemicellulose	g/kgTS	56.2 ± 6.97	35.9 ± 8.14	81.6 ± 12.37	108.2 ± 8.07
Lignin	g/kgTS	6.6 ± 8.29	81.6 ± 10.72	40.8 ± 2.47	192.9 ± 12.15
(CEL + HEMI)/LIGN	–	16.32	1.21	3.63	1.20
Total nutrients					
Total-C[a]	g/kgTS	469.1	486.6	386.1	415.4
TKN	g/kgTS	30.7 ± 1.68	32.1 ± 1.62	115.6 ± 8.38	93.2 ± 3.23
C/N		15.3	15.2	3.3	4.5
Total-P	g/kgTS	3.8 ± 0.06	6.5 ± 1.31	19.9 ± 3.63	16.2 ± 2.63
Total-K	g/kgTS	11.4 ± 1.57	10.31 ± 0.41	44.1 ± 8.64	30.7 ± 1.73
Soluble nutrients					
Soluble-N	g/kgTS	9.6 ± 0.52	16.3 ± 0.44	74.9 ± 6.75	42.2 ± 4.33
Soluble-P	g/kgTS	1.7 ± 0.75	1.7 ± 0.28	2.6 ± 1.09	1.4 ± 0.55
Soluble-K[a]	g/kgTS	9	9	22.6	26.3

[a] N = 1.

increased by 0.95 g/kgFM while with the other two digestates NH_4–N increase was ca 0.3 g/kgFM (Fig. 1, Table 4).

Secondly, the three digestates were assayed as inocula to digest both FW and AFW to assess the effect of long-term cultivation (>300 days in stirred tank reactors) on micro-organisms' capability to degrade FW and AFW. Both FW and AFW digestate inocula produced 0.451 m^3CH_4/kgVS from FW while from AFW the biochemical methane potential was 10% less with FW digestate and as much as 30% less with AFW digestate as inoculum. With the reference digestate higher methane potentials were observed with both FW and AFW. Both FW and reference digestate inocula degraded ca 50% of VS with both FWs while with the AFW digestate the VS removals were around 37%. However, with the low VS removal AFW digestate produced as much methane from FW using FW digestate as inoculum (0.451 m^3CH_4/kgVS; Table 4). During the assays with digestate inocula and FWs NH_4–N concentration increased (inoculum excluded) more with the FW digestate (0.68 and 0.34 g/kgFM) than with the AFW digestate (0.41 and 0.17 g/kgFM) assayed with both FW and AFW, respectively, while the highest NH_4–N increases were obtained with reference inoculum (0.73 and 0.51 g/kgFM with FW and AFW; Table 4).

The lower biochemical methane potentials, VS removals and decreased NH_4–N formation with AFW along with low NH_4–N starting concentration (~1 g/kg) with AFW digestate were connected to the formation of hardly degradable Maillard compounds during the autoclave treatment of FW, leading to reduced biodegradability of the material (Bougrier et al., 2008; Monlau et al., 2013), which was previously reported to decrease NH_4–N concentration in anaerobic digesters (Tampio et al., 2014). Combination of AFW and AFW digestate most likely inhibited the growth of certain microbes due to decreased protein degradation, leading to ca 40% reduced biochemical methane potential. Also the higher initial VS content in the AFW digestate assay bottles (22.6 gVS/bottle versus 13.4 and 9.8 gVS/bottle with FW and reference digestates) may have caused inhibition due to VFA accumulation (Lesteur et al., 2010), decreasing the residual methane potential of

Table 3
Characteristics of food wastes in various European countries. Organic fraction of municipal solid waste (OFMSW), restaurant waste (RW), household waste (HW), food waste (FW), autoclaved food waste (AFW), source-sorted (ss), mechanically recovered (mr).

Waste	Country	Protein (g/kgTS)	Fat (g/kgTS)	Cellulose (g/kgTS)	Hemicellulose (g/kgTS)	Lignin (g/kgTS)	Reference
ss-OFMSW	Denmark	105–171	102–177	N/A	N/A	N/A	Hansen et al., 2007
RW	Spain	275	288	N/A	N/A	N/A	Garcia et al., 2005
HW	Spain	163	113	N/A	N/A	N/A	Garcia et al., 2005
FW	Finland	169	175	N/A	N/A	N/A	Valorgas, 2011
FW	Italy	233	215	N/A	N/A	N/A	Valorgas, 2011
FW	UK	161–172	194–257	N/A	N/A	N/A	Valorgas, 2011
ss-FW	UK	257	165	55	42	18	Zhang et al., 2012
mr-OFMSW	UK	204	108	397	82	289	Zhang et al., 2012
FW	UK	219	142	52	56	7	Present study
AFW	UK	209	143	60	34	82	Present study

N/A, not available.

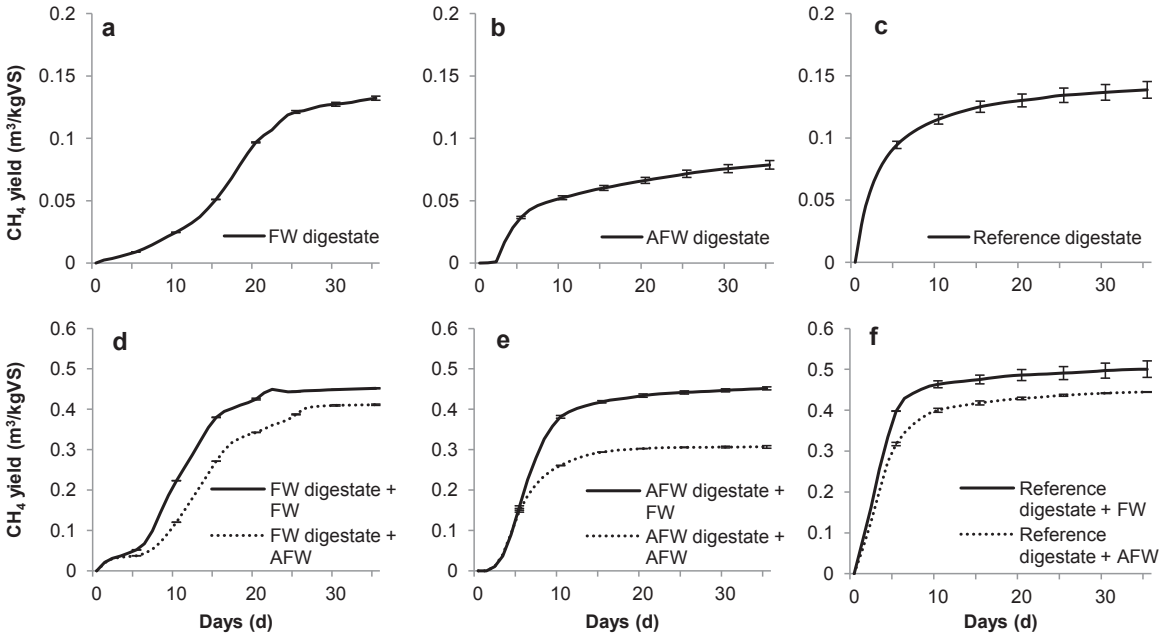

Fig. 1. Residual methane potentials (RMP) of food waste (FW, a), autoclaved FW (AFW, b) and reference (c) digestates. Biochemical methane potentials (BMPs) of FW and AFW digested with FW digestate (d), AFW digestate (e) and reference digestate (f) (inoculum RMP subtracted). Error bars represent standard deviations and are plotted in five-day intervals, N = 2–3.

the AFW digestate. However, the initial VS concentrations did not correlate with the biochemical methane potential results with FWs.

The FW digestate showed good gas production with both FWs studied as did the reference digestate, indicating the capability of microbes to degrade the feed material. However, the AFW digestate showed lower methane production with both FWs, which indicates that the adaptation of the microbial population towards the AFW was not successful. Prior studies have shown that autoclaving of FW changes the microbial populations, especially bacteria, during AD (Blasco et al., 2014) due to the transformation of proteins, leading to further decreases in methane yields during AD. With these batch experiments it was confirmed that the autoclaving of FW affected the ammonification capacity of the digestate, which led to reduced methane formation.

3.4. Hygienic quality

The hygienic quality of the FW (7 samples) and digestate (6 samples) were tested with hygiene indicators *E. coli*, other coliforms, total coliforms, enterococci and sulphite-reducing clostridia and Salmonella (Fig. 2). No Salmonella was detected in any of the feed or digestate samples (data not shown). In one of the six FW samples a few colonies of *E. coli* were discovered, while both enterococci (average $2.79 \times 10^4 \pm 2.74 \times 10^4$ cfu/g) and clostridia ($2.24 \times 10^3 \pm 1.86 \times 10^3$ cfu/g) were also discovered. In AFW all hygiene indicators were under the detection limit (5 cfu/g). In both digestates, high enterococci concentrations (6.77×10^8 cfu/g $\pm 7.40 \times 10^8$ and $3.71 \times 10^8 \pm 4.64 \times 10^8$ in the FW and AFW digestate) were detected, while the clostridia concentration

Table 4
Initial TS, VS and NH_4–N (g/kgFM) during batch assays with different inocula (FW, AFW and reference digestates) and food waste (FW) and autoclaved food waste (AFW) as substrates. Residual methane potentials (RMPs) and biochemical methane potentials (BMPs) are shown with standard deviations (N = 2–3).

Inoculum	FW digestate			AFW digestate			Reference digestate		
Added substrate	–	FW	AFW	–	FW	AFW	–	FW	AFW
Characteristics									
TS initial (g/kg)	49.8	85.7	86.1	69.9	99.6	99.5	38.6	65.1	65.2
TS final (g/kg)	43.9	53.0	54.7	61.9	67.9	68.8	32.2	41.8	45.0
VS initial (g/kg)	33.4	66.8	66.8	56.6	84.1	84.1	24.4	48.9	48.9
VS final (g/kg)	27.5	33.6	35.4	48.3	52.5	53.4	19.1	24.03	26.3
VS removal (%)	17.6	49.8	47.1	14.6	37.5	36.6	21.9	50.8	46.2
TKN initial (g/kg)[a]	6.08	7.17	7.45	5.48	7.11	7.00	N/A	N/A	N/A
NH_4–N initial (g/kg)[a]	3.02	3.09	3.11	1.03	1.05	1.07	1.31	1.35	1.35
NH_4–N final (g/kg)	3.31	4.07	3.75	1.98	2.41	2.19	1.64	2.41	2.19
NH4–N increase (g/kg)	0.3	0.98	0.64	0.95	1.36	1.12	0.33	1.06	0.84
NH4–N increase, inoculum excluded (g/kg)	N/A	0.68	0.34	N/A	0.41	0.17	N/A	0.73	0.51
RMP or BMP measured (m^3CH_4/kgVS)	0.132 ± 0.002	0.452 ± 0.001	0.411 ± 0.002	0.079 ± 0.003	0.451 ± 0.004	0.307 ± 0.003	0.139 ± 0.007	0.501 ± 0.020	0.445 ± 0.001

–, no FW added.
N/A, not available/applicable.
[a] Calculated value.

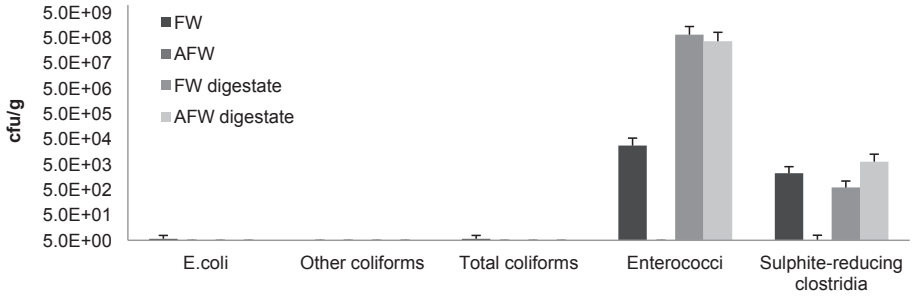

Fig. 2. Hygienic quality of food waste (FW), autoclaved food waste (AFW) and FW and AFW digestates. Averages and positive standard deviations are shown, N = 6–7.

remained lower (6.14×10^2 cfu/g ± 4.98×10^2 and $6.48 \times 10^3 \pm 6.29 \times 10^3$ cfu/g in the FW and AFW digestates, respectively).

The absence of coliforms in the studied FW was likely due to the freezer storage time (before preparation as feed and analysis). In fresh FW these indicators have usually been detected in concentrations of 10^4–10^5 cfu/g in biogas plants treating FW as such (Sahlström et al., 2008) and co-digesting FW with manures and animal by-products (Bagge et al., 2005). The present concentration of enterococci was similar to that reported for fresh FW (around 10^4 cfu/g) by Sahlström et al. (2008) due to the resistance of enterococci towards freezing (Geiges, 1996). Similarly high concentrations of sulphite-reducing clostridia were detected as these spore-forming organisms are also resistant to freezing (Geiges, 1996).

The results show that the studied autoclave treatment effectively reduced all the hygiene indicator concentrations in AFW due to high temperature and pressure, which are widely used for sterilisation. However, the observed increase in concentrations of enterococci and clostridia (up to 8 logs) in the AFW digestate clearly indicates the potential of hygienised material for microbial growth. The increase was apparently due to growth of indicator organisms in the stirred tank reactors, originating from the sludge with which the reactors were inoculated or possibly from contamination of the AFW samples. Absence of coliforms in the studied digestates indicates that either there were no coliforms in the original inoculum or the microbes were not able to survive due to competition of microbial communities while the conditions were favourable for clostridia and enterococci.

Altogether, according to the EU's Animal By-Product regulations (European Council, 2011; European Parliament and the Council, 2009) digested FW and digested autoclaved FW were both hygienically suitable for land application as the concentration of *E. coli* was under the threshold value 1000 cfu/g and no Salmonella was detected.

3.5. Agronomic usefulness of digestates

The total and soluble nutrient composition of the FWs and digestates was studied to evaluate the agronomic usefulness of the digestates (Table 2). The total nutrient levels of nitrogen (31 gN/kgTS), potassium (11 gK/kgTS) and carbon 470–487 (gC/kgTS) were similar between the studied FWs, and thus the AFW had a higher total-P content (~7 g/kgTS in AFW, 4 g/kgTS in FW). The soluble P and K contents in both FWs were around 1.7 and 9 g/kgTS while the soluble-N concentration increased from 10 to 16 g/kgTS after autoclaving. When digestates were compared the AFW digestate had 20% lower total Kjeldahl nitrogen and 44% lower soluble-N levels compared to the FW digestate. The C/N ratios were relatively low with both studied digestates (3.3–4.5), which was due to the mineralisation of carbon during AD.

The total nutrient concentration in FWs correlated well with different European (UK, Finland, Italy) food wastes, where total-N concentrations varied between 24 and 34 g/kgTS, total-P between 2.7 and 6.4 and total-K between 8.6 and 14.3 g/kgTS (Valorgas, 2011). Only total-P was observed in slightly higher concentrations in the AFW where some additional phosphorus could have dissolved from the autoclaving apparatus due to P impurities in steel. Soluble N increase after autoclaving was probably due to solubilisation of nitrogen into other compounds than NH_4–N, e.g. to soluble Maillard compounds.

In the digestates the NPK-ratios (per TS) were 100:17:38 in the FW digestate and 100:17:33 in the AFW digestate, which were similar to the results obtained with source-sorted FW in the UK (NPK 100:11:41; Zhang et al., 2012). Compared to available commercial fertilisers (~20 %N) the N content in the FW digestate was low but the proportion of K and P was higher, and thus it was considered to be a suitable fertiliser for leguminous plants (Israel, 1987) and plants at reproductive state (Clemens and Morton, 1999). However, when considering the low NH_4–N/TKN ratio of the AFW digestate (26%) compared to the FW digestate (52%), the AFW digestate was evaluated to be more suitable for use as soil amendment than fertiliser (Nkoa, 2013). The 10–15% lower N-tot and K-tot concentrations (per FM) would also increase the volume of AFW digestate needed for fertilising in similar quantities. The TS contents of the studied digestates were 67 g/kgFM (FW digestate) and 79 g/kgFM (AFW digestate), which are similar to those of manure used as fertiliser in agriculture (Amon et al., 2006), enabling the spreading of digestates with similar machinery as manure.

Calculated with the values obtained from this study the FW produced in Europe (38 million tonnes; European Commission, 2010) accounts for approximately 296 000 tonnes of N, 46 200 tonnes of P and 108 000 tonnes of K. These calculated values represent 2.8, 4.5 and 5.0% of the manufactured fertilisers consumed in the EU (10.4 Mt of N, 1.0 Mt of P, 2.2 Mt of K; Eurostat, 2013). With European FW, approximately 1.74 million hectares of field could be fertilised, using an assumed N fertilisation rate of 170 kg/ha.

4. Conclusions

Anaerobic digestion of high protein-containing FW produces digestates with relatively high NH_4–N (4 g/kgFM), which supports its use as a fertiliser in agriculture. Also the hygienic quality, nutrient concentrations (NH_4–N, P, K), TS content and low residual methane emission potential facilitate fertilisation use.

Anaerobic digestion of autoclaved FW results in digestate with higher undegraded protein and lower ammonium content than without autoclaving, leading to reduced microbial activity and decreased methane yield in batch assays. This increases the volumes needed to achieve the desired fertilising effect by

approximately 10–15% compared to FW; this, coupled with its low ammonium content, supports the use of autoclaved FW digestate in soil amendment practices.

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III

AGRONOMIC CHARACTERISTICS OF FIVE DIFFERENT URBAN WASTE DIGESTATES

by

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Research article

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 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.

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 and 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 and 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 and 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

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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 and Smith, 2014; Svensson et al., 2004) and on soil quality (Abubaker et al., 2012; Odlare et al., 2008, 2011; Rigby and 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 and 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, Fig. 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,

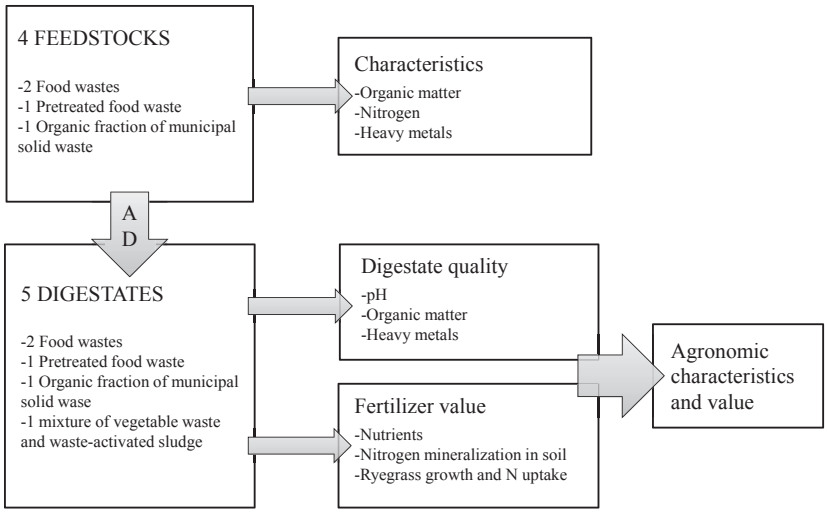

Fig. 1. The analyzed agronomic characteristics of the studied digestates and feedstocks.

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^{3}day.
[c] kgCOD/m^{3}day.

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 0–2000 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 h light in 16 °C and 10 h 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 h. 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 and 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 h, 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^3d) in reactors fed with FW1 and FW2 than with FW3 (26 days, 3.3 kgVS/m^3d) and OFMSW (24 days, 2.4 kgVS/m^3d) 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 and 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 and 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., 2012, 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 and 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 and 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).

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.

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

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

N/A, not available.
[a] BSI, 2010.
[b] Decree of the Ministry of Agriculture and Forestry No 24/11 on Fertiliser Products.
[c] Saveyn and 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 170 kgTKN/ha.

(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., 2010) 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, Fig. 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),

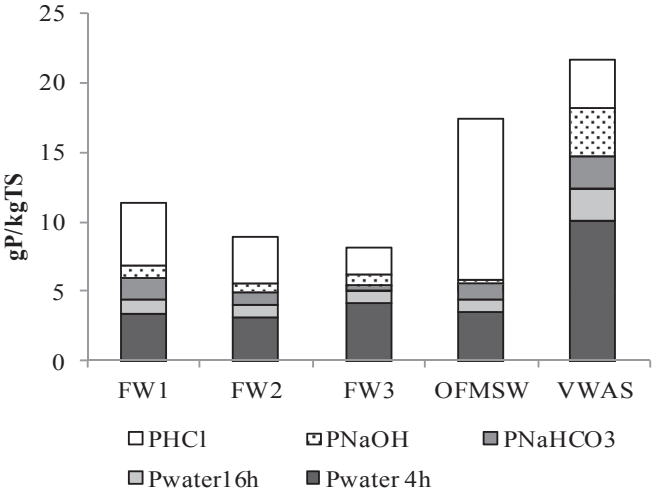

Fig. 2. Solubility of phosphorus determined with Hedley fractionation.

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 and 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, Fig. 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, Fig. 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 and 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.

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

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, Fig. 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 $NUE_{NH4\text{-}N}$, 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 and 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

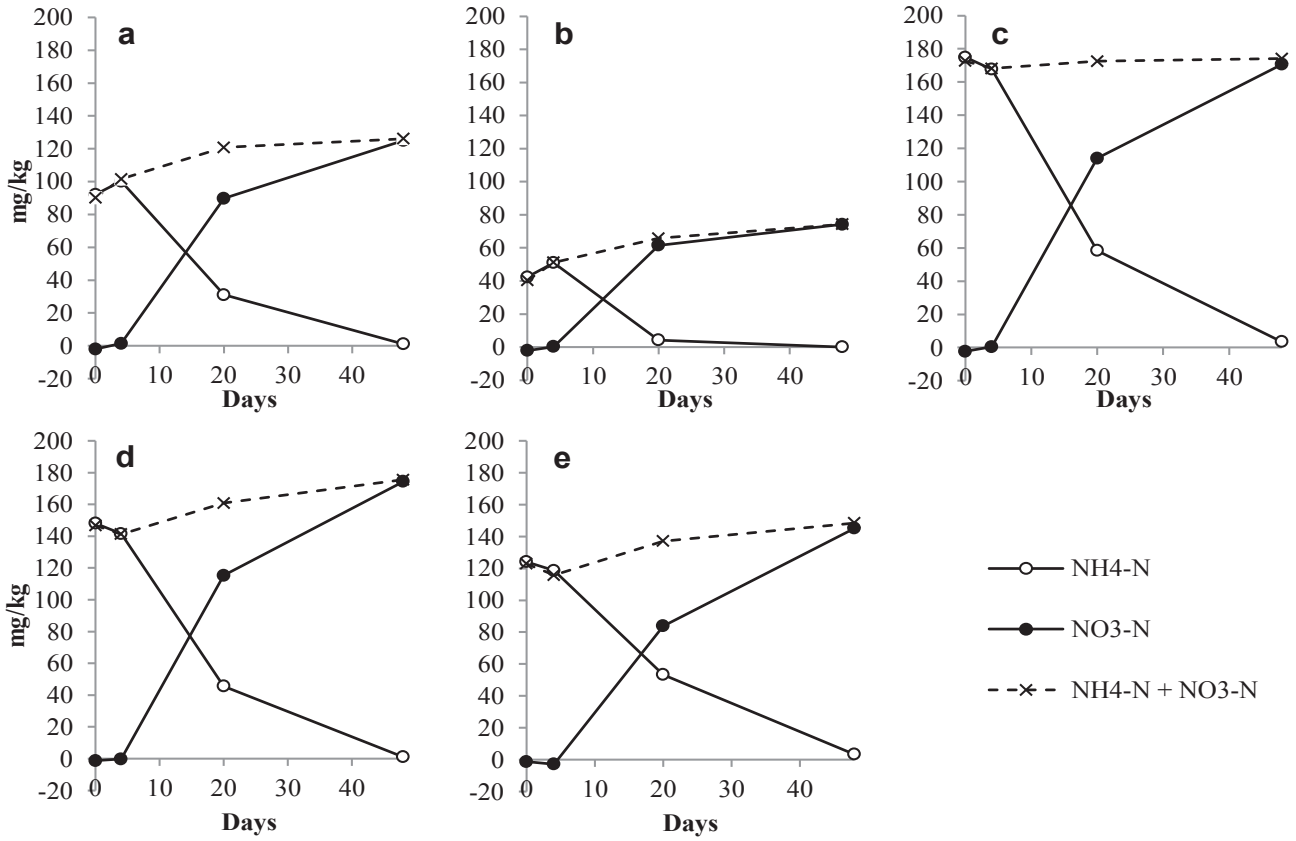

Fig. 3. Nitrogen mineralization during 48-day incubation tests. Digestates FW1 (a), FW2 (b), FW3 (c), OFMSW (d), VWAS (e).

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.

Treatment	Applied (mg/pot)			Yield	N uptake	NUE_{NH4-N}	NUE_{TKN}
	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

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

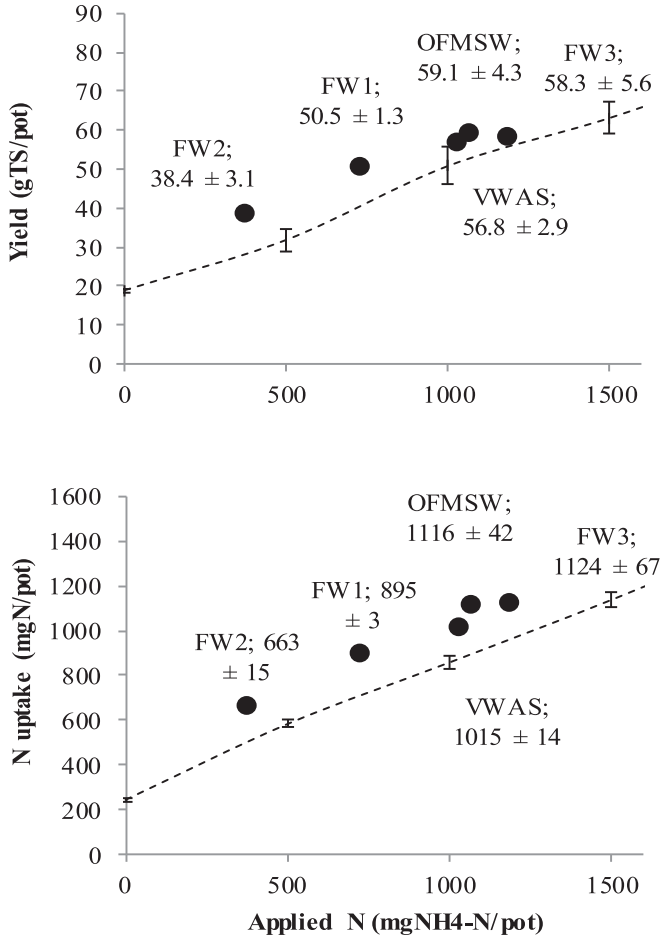

Fig. 4. Ryegrass yield and nitrogen uptake of digestates compared to control treatments. The dotted line represents the control treatments and error bars the standard deviation within control samples.

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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IV

LIQUID FERTILIZER PRODUCTS FROM ANAEROBIC DIGESTION OF FOOD WASTE: MASS, NUTRIENT AND ENERGY BALANCE OF FOUR DIGESTATE LIQUID TREATMENT SYSTEMS

by

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Liquid fertilizer products from anaerobic digestion of food waste: mass, nutrient and energy balance of four digestate liquid treatment systems

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ABSTRACT

This study compared four different digestate liquid treatment systems of a theoretical anaerobic digestion plant in order to facilitate the utilization of municipal food waste nutrients in agriculture. The mass, nutrient and energy balances of a theoretical plant digesting 60 kt/y of food waste were used to evaluate the feasibility of the treatments to concentrate nutrients into liquid fertilizer products. The studied technologies for digestate liquid treatment were ammonia stripping, ammonia stripping combined with reverse osmosis (RO), evaporation combined with RO, and stripping combined with both evaporation and RO. As a result, processing of digestate into concentrated fertilizer products consumed less than 10% of the produced energy from food wastes and was also sufficient for the heat-demanding digestate liquid treatments, evaporation and stripping. The digestate liquid treatment systems were considered as nitrogen and potassium concentration methods which were able to concentrate up to 67% of the feedstock nitrogen into transportable fertilizer products with low mass. Of the studied digestate systems evaporation combined with RO was evaluated as the most efficient nutrient recovery technology for the production of transportable fertilizer products due to the high concentration of nutrients and nutrient availability as well as low product mass and energy consumption. Overall, the selection of the treatment technology is dependent on the location of the anaerobic digestion plant relative to the agricultural land and the type of fertilizer products needed.

1. Introduction

Anaerobic digestion (AD) of food waste (FW) is increasingly used to produce renewable energy, in the form of heat and power or vehicle fuel, and nutrient-rich digestate for agriculture, to decrease the use of energy intensive mineral fertilizers (Laureni et al., 2013). However, the digestate has usually unbalanced nutrient ratios for plant growth (Camilleri-Rumbau et al., 2014). Large mass due to high water content increases the transportation need of the digestate, as the AD plants treating municipal FW are usually located far from agricultural lands (Babson et al., 2013). Digestate treatment by solid–liquid separation is an increasingly used treatment for the production of phosphorus containing solid digestate and liquid digestate containing water-soluble nitrogen and potassium. The solid–liquid separation of the digestate divides most of the mass into the liquid fraction decreasing its nutrient concentrations (Hjorth et al., 2010). Low nutrient concentrations and large mass complicate the use of the liquid digestate in agriculture and increase the transportation need (Chiumenti et al., 2013). To efficiently utilize the FW nutrients, the treatment of liquid digestate is needed to decrease its mass and increase nutrient concentrations.

The digestate liquid can be treated to remove water and simultaneously concentrate nutrients. This lowers the environmental impact (i.e. global warming potential and acidification) and reduces transportation costs to areas with nutrient deficits compared with digestate use as such (Rehl and Müller, 2011). In addition to the decreased transportation costs, the additional economic benefits of the digestate liquid treatment are related to the

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profit gained from the selling of the fertilizers (Fuchs and Drosg, 2013; Rehl and Müller, 2011). With the combination of solid–liquid separation and digestate liquid treatment, fertilizer products with optimal composition can be produced (Hjorth et al., 2010). Produced fertilizers can be designed to match the crop nutrient requirements and to achieve better control of the nutrient contents of the applied fertilizer to reduce the nutrient run-off and leaching. These products could be also used to supplement the raw digestate fertilization by replacing mineral fertilizers.

Technologies for digestate liquid treatment such as ammonia stripping, evaporation, struvite precipitation, membrane separation, as well as various combinations of these, have been previously studied considering nutrient recovery and production of nutrient-rich products with, e.g., digestate liquids, manure and urine (Antonini et al., 2011; Bonmatí et al., 2003; Bonmatí and Flotats, 2003a, b; Chiumenti et al., 2013; Ek et al., 2006; Ledda et al., 2013). However, to ensure the usability and sustainability of different digestate liquid treatment techniques and to facilitate the agricultural utilization of the nutrients over longer transportation distances, the total digestate treatment chain and all produced mass flows should be taken into consideration as well as all the process inputs, e.g., chemicals and energy (Mehta et al., 2015). As life cycle assessment and energy efficiency studies have mainly concentrated on the use of raw digestate or separated solid digestate (e.g. Bacenetti et al., 2013; Berglund and Börjesson, 2006; Evangelisti et al., 2014; Pöschl et al., 2010; Smyth et al., 2009) only a few studies exist where the digestate liquid and its treatment has been taken into consideration (Rehl and Müller, 2011). In addition, these life cycle studies focus solely on environmental and ecological effects and do not evaluate the fertilizer products from the viewpoint of biogas plant efficiency or agriculture and plant nutrition. From these perspectives information about the mass, nutrient and energy balances of an AD plant with digestate liquid treatment is important, in addition to environmental aspects.

The aim of this study was to compare the potential of four digestate liquid treatment systems of a theoretical AD plant digesting municipal FW to produce fertilizer products with low water and concentrated nutrient contents. The studied treatment systems were different combinations of ammonia stripping, evaporation and membrane filtration, which have been applied in the full scale treatment of digestate or manure based liquids (see e.g. Boehler et al., 2015; Flotats et al., 2011; Fuchs and Drosg, 2013). For all four systems the mass, nutrient and energy balances were calculated and the nutrient recovery, mass reduction and energy efficiencies were compared based on typical literature values from laboratory, pilot and full scale studies. The performance of the treatment systems was also assessed in relation to the energy consumption of fertilizer product transportation to see the effect of digestate liquid treatment on the transportability of the products.

2. Materials and methods

2.1. Overview of the theoretical AD plant

This study investigated a theoretical mesophilic AD plant which was assumed to digest source-segregated municipal FW (60 kt/y, kilotonnes per year). Fig. 1 presents the applied AD plant system boundaries which include pretreatment, a digester, digestate treatment and biogas upgrading. The FW was pretreated and hygienized (1 h at 70 °C) and subsequently diluted to a total solids (TS) content of 15% with processed water or water from the local water supply. The digestate treatment was assumed to include the separation of the digestate into liquid and solid digestates using a centrifuge. The liquid digestate was assumed to be treated with one of the four digestate liquid treatment systems consisting of ammonia stripping, evaporation and membrane (reverse osmosis, RO) technologies (Fig. 2). The formed biogas was assumed to be upgraded in a combined heat and power unit (CHP) into heat and electricity to be used in the AD plant and the excess electricity was to be fed to the power grid.

2.2. Pretreatment, hygienization, AD and gas upgrading

2.2.1. Mass and nutrient balances

The feed for AD was based on the characteristics of source-segregated FW: TS 25%, volatile solids (VS) 23%, Ntot 7.5 kg/tFM (fresh matter), NH_4-N 0.4 kg/tFM, Ptot 0.9 kg/tFM, Ktot 2.8 kg/tFM (Tampio et al., 2014, 2015). The FW (60 kt/y) was assumed to be pretreated as in Banks et al. (2011) by shredding/maceration and then hygienized (1 h at 70 °C according to European Council, 2011; European Parliament and the Council, 2009). Pretreatment and hygienization were not considered to affect the FW mass and nutrient content as material was not removed during the pretreatment step. The dilution water was assumed to be added to the FW during the maceration step (40 kt/y water to achieve TS of 15%, mixture referred to as feedstock). The mass of the produced digestate was calculated by subtracting the mass of the biogas from the feedstock (60 kt of FW + 40 kt of dilution water). The calculation of the biogas mass was based on biogas composition (60% CH_4, 40% CO_2) and component densities (CH_4 0.72 kg/m^3, CO_2 1.96 kg/m^3, see Supplementary material for calculations). In the digestate, the total nutrient concentrations (Ntot, Ptot, Ktot, kg/tFM) were assumed to be the same as in the feedstock, while the ammonium nitrogen in FW was assumed to increase from 0.4 kg/tFM to 4 kg/tFM after digestion (Tampio et al., 2014, 2015).

2.2.2. Energy balance

The energy balance included both heat and electricity consumption and production in the AD plant. The amount of thermal energy (th) needed for heating the FW (60 kt) to 75 °C to maintain the required temperature during hygienization was calculated assuming the specific heat capacity of the feedstock to be the same as that of water (4.18 kJ/kg°C, see Supplementary material for calculations). The heat energy from the hygienization was assumed to be sufficient for the mesophilic (40 °C) digester (Berglund and Börjesson, 2006; Prapaspongsa et al., 2010) and thus, no additional heating was allocated for the heating of FW prior to the digester. However, the heating of the dilution water (40 kt/y) was calculated with the specific heat capacity of water using temperature difference from 15 to 40 °C. Heat losses from the hygienization and digester units were assumed to be in total 15% of the heat demand (digester heat loss 15% in Smyth et al., 2009, 20% in Rapport et al., 2011) being dependent on the reactor design as well as the difference between the reactor and outdoor temperature. The electricity (el) consumption of 37.5 kWh/tFM feedstock for the hygienization and pretreatment unit (reviewed in Pöschl et al., 2010; see Supplementary material), and 18 kWh/tFM for the digester was used (reviewed in Berglund and Börjesson, 2006; reviewed in Pöschl et al., 2010; see Supplementary material).

The energy content (MWh/y) of the produced biogas was calculated by multiplying the biochemical methane potential of the FW (BMP, 450 m^3CH_4/tVS, Tampio et al., 2014) with the amount of feedstock VS fed to the reactor. The conversion factor of 1 m^3(CH_4) = 10 kWh was used. For the conversion of the biogas into heat and electricity in the CHP unit, the energy conversion efficiencies of 38% for electricity and 48% for heat were used (Bacenetti et al., 2013; Poeschl et al., 2012). Additionally, for the CHP-unit electricity the consumption of 5% of the energy produced in CHP was applied (Banks et al., 2011; Havukainen et al., 2014; Naegele et al., 2012; Pöschl et al.,

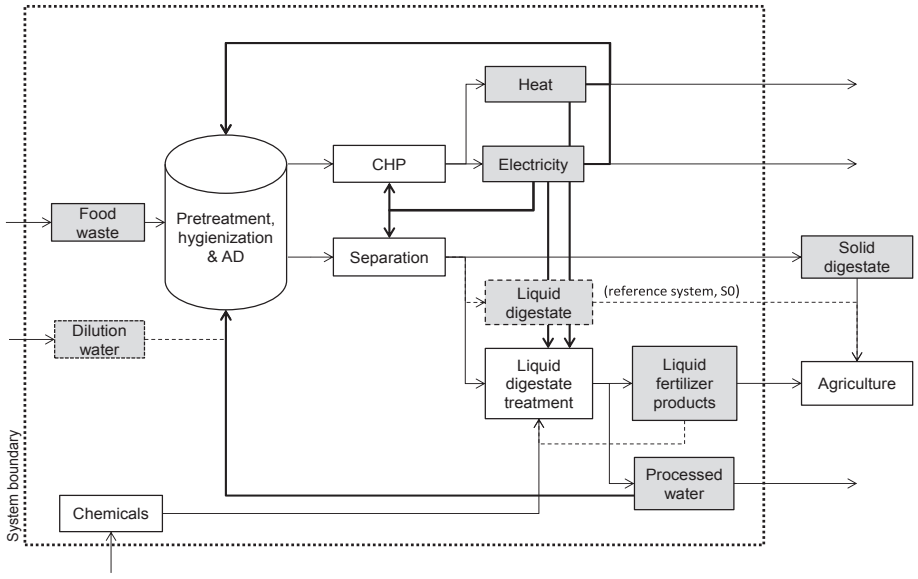

Fig. 1. System boundaries of the studied anaerobic digestion plant and digestate liquid treatment with material and energy flows. Grey boxes represent feedstock/product and white boxes represent studied unit operations.

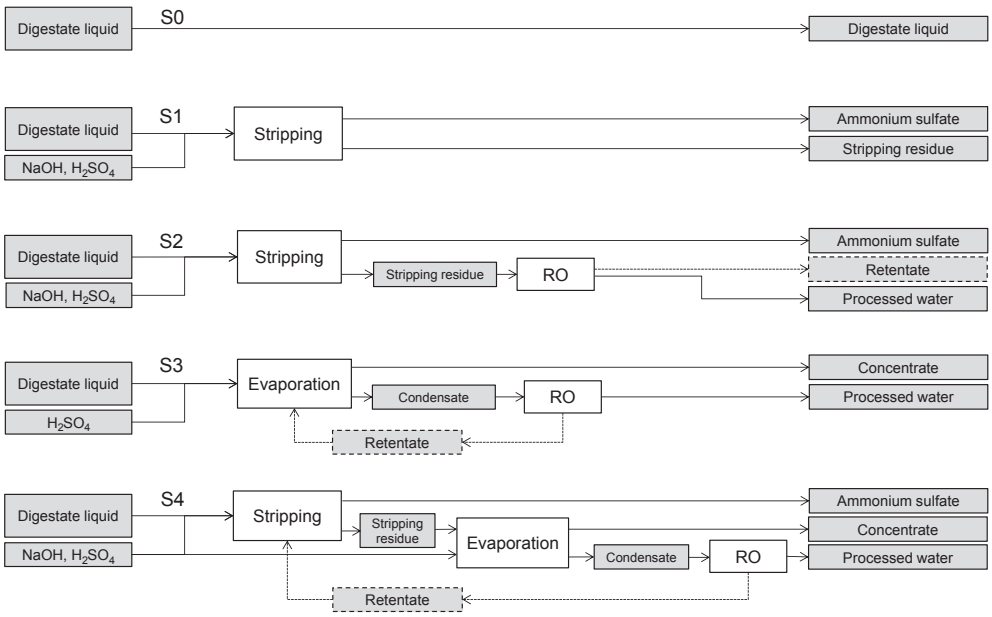

Fig. 2. Digestate liquid treatment systems (S0–S4) and products evaluated in this study. White boxes represent treatments and grey boxed represent treatment inputs and outputs.

2010; see Supplementary material). However, the use of different desulfurization methods for the biogas prior to CHP can increase the energy consumption, which was not taken into consideration in this study. E.g. in Karellas et al. (2010) the combined desulfurization with a spray scrubber and CHP was reported consuming 15% of the electricity produced in CHP in an AD plant treating pig manure, wheat straw and glycerol (45 kt/y).

2.3. Digestate treatment

The digestate was assumed to be separated with a decanter centrifuge producing liquid and solid fractions of which the liquid fraction was further treated to produce fertilizer products. The solid digestate was assumed to be used as such in agriculture, as it is the practice e.g. in the UK, Scandinavia and Switzerland, where the legislation does not require further treatment with e.g. composting (Saveyn and Eder, 2014). Centrifuge separation efficiencies for mass, TS, VS and nutrients (Ntot, NH_4-N, Ptot, Ktot) were adopted from the literature and the electricity consumption was assumed to be 3.5 kWh/tFM digestate (Flotats et al., 2011, reviewed in Hjorth et al., 2010, Ledda et al., 2013, Møller et al., 2000, 2002, Table 1, see Supplementary material). Polymer/flocculent additions used in separation were not included in the mass balance as the annual total amount of additions was considered negligible (for example 1.625 $g/gTS_{digestate}$ of both polymer and mineral conditioner, Alvarenga et al., 2015).

It was assumed that all outputs from the different digestate liquid treatment systems were suitable for agricultural use and/or processed water suitable for discharging (Fig. 2). Processed water was used as dilution water before the digester and the surplus

Table 1
The separation efficiency of decanter centrifuge, nutrient recovery efficiencies of digestate liquid treatment technologies and energy consumption of each treatment. Values chosen based on literature (see also Supplementary material).

Material	Separation/recovery efficiency, %							Energy consumption,
	Mass	TS	VS	Ntot	NH_4-N	Ptot	Ktot	kWh/t to be treated
Solid-liquid separation of digestate								
Liquid digestate	90[a]	20[a]	20[b]	70[a]	81[a]	10[a]	85[a]	3.5[a]
Digestate liquid treatment								
Stripping								
Ammonium sulfate	–	–	–	–	95[c]	–	–	2[d] + heat[e]
Evaporation								
Concentrate	20[f]	–	–	90[f]	–	100[f]	100[f]	5[f] + heat[e]
Reverse osmosis								
Retentate	28[g]	100[g]	100[g]	–	95[g]	95[g]	99[g]	2.5[g]

–, not applicable.
[a] Flotats et al., 2011, reviewed in Hjorth et al., 2010, Ledda et al., 2013, Møller et al., 2000, 2002.
[b] Separation efficiency for VS was assumed to be same as for TS.
[c] Basakcilardan-Kabakci et al., 2007, Bonmatí and Flotats, 2003a, Flotats et al., 2011, Guštin and Marinšek-Logar, 2011, Laureni et al., 2013, Liu et al., 2015.
[d] kWh/kgN recovered in ammonium sulfate, reviewed in van Eekert et al., 2012.
[e] Calculated with the specific heat capacity of water.
[f] Bonmatí and Flotats, 2003b, Chiumenti et al., 2013, Ek et al., 2006, Flotats et al., 2011, Maurer et al., 2003.
[g] Carretier et al., 2015, Chiumenti et al., 2010, Ek et al., 2006, Flotats et al., 2011, Ledda et al., 2013, Mondor et al., 2008.

water was assumed to be discharged. Additional treatments of processed water with, e.g., active carbon filters (Zarebska et al., 2015) were not taken into consideration. With each treatment system, the consumption of chemicals (NaOH, H_2SO_4, m^3/y) was included in the calculation of the output mass and characteristics.

2.3.1. Reference system, S0

In the reference system (referred as S0), the digestate liquid did not undergo any treatment after digestate separation (Fig. 2). Thus, due to the lack of liquid treatment, the water for feedstock dilution came from the local water supply, i.e., outside the system boundaries. The digestate liquid was not used for dilution due to the high nitrogen content which could accumulate during digestion and inhibit the process.

2.3.2. Stripping, S1

Ammonia stripping combined with H_2SO_4 scrubbing (system referred to as S1) was studied to produce ammonium sulfate and stripping residue (Fig. 2). During stripping, NH_4-N is transformed to NH_3 along with the temperature and a pH increase and further recovered with H_2SO_4 in the form of ammonium sulfate ($(NH_4)_2SO_4$) during scrubbing. In the mass and nutrient balance calculations the nitrogen (NH_4-N) recovery efficiency was assumed to be 95% based on laboratory, pilot and full scale studies (Basakcilardan-Kabakci et al., 2007; Bonmatí and Flotats, 2003a; Flotats et al., 2011; Guštin and Marinšek-Logar, 2011; Laureni et al., 2013; Liu et al., 2015, Table 1; see also Supplementary material). $(NH_4)_2SO_4$ was assumed to be a chemically pure product with no TS, VS, Ptot or Ktot, while the Ntot was assumed to consist solely of the NH_4-N. The NH_4-N mass balance was based on the assumption that the NH_4-N concentration in the produced ammonium sulfate was 40 kg/tFM (Laureni et al., 2013). The energy consumption during stripping consisted of the heat energy for the temperature increase from the digester to the stripper (from 40 to 80 °C) which was calculated using the specific heat capacity of water. Electricity consumption for stripping of 2 kWh/kgN recovered was used (reviewed in van Eekert et al., 2012, Table 1; see Supplementary material). The stripping was assumed to be executed in atmospheric pressure and thus, no energy consumption for the production of vacuum was allocated. NaOH (50%) consumption for the pH increase before stripping was assumed to be the same as the pH increase of urine (pH from 9 to 10, 20 L/m^3, Antonini et al., 2011). NaOH consumption could be reduced with CO_2 stripping before ammonia stripping (Boehler et al., 2015), which was, however, not taken into consideration in this study. The H_2SO_4 (93%) consumption during ammonia stripping was calculated using the molar ratios of H_2SO_4 and $(NH_4)_2SO_4$ and the nitrogen concentration of 40 kg/tFM in the ammonium sulfate, from which the consumption of 0.08 m^3/t liquid digestate was used.

2.3.3. Stripping and reverse osmosis, S2

Ammonia stripping was combined with reverse osmosis treatment (system referred as S2) to produce ammonium sulfate, retentate and processed water (Fig. 2). After stripping, the stripping residue was directed to the RO treatment producing processed water flow for discharge. For stripping treatment, the mass and nutrient separation/recovery efficiencies, chemical and energy consumptions calculations were based on the same values as in system S1 (Table 1). The mass and nutrient balances for the RO treatment were calculated based on the typical values from the literature (Table 1) and the electricity use of 2.5 kWh/t stripping residue was applied (Carretier et al., 2015; Chiumenti et al., 2010; Ek et al., 2006; Flotats et al., 2011; Ledda et al., 2013; Mondor et al., 2008; see Supplementary material). The regeneration and/or change of RO membranes were not taken into consideration.

2.3.4. Evaporation and reverse osmosis, S3

Evaporation combined with RO (referred as S3) was studied to concentrate nutrients in the liquid digestate and a major part of the liquid into condensate which was further treated with RO to produce retentate. The retentate was in turn recycled back to separation, and processed water was directed to discharge (Fig. 2). The pH of the digestate liquid was controlled with H_2SO_4 to prevent the volatilization of NH_4^+ during evaporation where the liquid was heated to 80 °C. The mass and nutrient balance calculations for the evaporation were based on typical literature values from laboratory, pilot and full scale studies (Bonmatí and Flotats, 2003b; Chiumenti et al., 2013; Ek et al., 2006; Flotats et al., 2011; Maurer et al., 2003, Table 1; see also Supplementary material). The TS and VS separation efficiencies in the concentrate were assumed to be 100% and the NH_4-N recovery rate the same as in Ntot (80%). The H_2SO_4 (93%) consumption of 0.005 m^3/t digestate liquid for the pH decrease during evaporation was based on the pH decrease of urine and manure with strong H_2SO_4 (pH from 9 to 6 in Ek et al., 2006, pH from 7.2 to 5.5 in Sørensen and Eriksen, 2009). The consumption of other chemicals such as antifoaming additives was not included.

The energy consumption of evaporation consisted of the heat energy needed to increase the digestate liquid temperature from 40 to 80 °C plus the electricity consumption of 5 kWh/t liquid digestate based on typical literature values (Bonmatí and Flotats, 2003b; Chiumenti et al., 2013; Ek et al., 2006; Flotats et al., 2011; Maurer et al., 2003, Table 1; see also Supplementary material). No vacuum conditions for the evaporation were applied as the process temperature was high (80 °C). Mass and nutrient separation/recovery efficiencies, chemical and energy consumptions considering RO were the same as in S2.

2.3.5. Stripping, evaporation and RO, S4

Combined stripping, evaporation and RO (S4) were studied to produce both ammonium sulfate and concentrate. After stripping, the stripping residue was evaporated to produce concentrate and condensate. The condensate was treated in RO after which the retentate was further recycled back to digestate separation and the processed water was discharged (Fig. 2). The same mass and nutrient separation/recovery efficiencies, chemical and energy consumptions for stripping were used as in system S1, for evaporation as in system S3 and for RO as in system S2 (Table 1). However, it was assumed that heat from the stripping (80 °C) was sufficient for the evaporation and thus, no heat energy was allocated for evaporation (Ervasti et al., 2011).

In systems S1-S4, no heat losses were calculated for stripping and evaporation, nor the energy consumption of the membrane changes for RO, due to the lack of reference data in the literature. As the operational temperatures were the same in stripping and evaporation (80 °C), the effect of heat losses in the total energy balances between systems S1-S3 was assumed to be relatively small. In the full scale stripping and evaporation plants the heat losses due to the convection and radiation through treatment apparatus, and through the evaporation of material, are reduced with the insulation and use of heat exchangers.

2.4. Transportation

The energy consumption during the transportation of the fertilizer products from the digestate liquid treatments was calculated using the energy consumption of a semitrailer truck in Finland, 0.17 kWh/t-km (full 25t load, EURO 5 truck type, VTT, 2012). The transportation of liquid products from each digestate liquid treatment systems was studied from 0 to 250 km.

3. Results

3.1. Mass and nutrient balance of AD and digestate treatment

The mass and nutrient flows of digestate and digestate liquid treatments formed the mass and nutrient balance of the systems showing the concentration of nutrients into fertilizer products. The calculations were based on the mass flow of the feedstock (FW + diluting water) and added chemicals as well as the characteristics of the digestate. The mass of the digestate accounted for 87% of the initial feedstock fed to the AD plant (60 kt of FW + 40 kt of dilution water), while 13% of the feedstock was transformed into biogas during AD. The digestate separation produced liquid (79 kt/y) more than eight times the amount of solid digestate (9 kt/y) (Table 2, Table 3).

The treatment of the digestate liquid with stripping (S1) concentrated 45% of the initial feedstock nitrogen into ammonium sulfate (40 kg/tFM) with the mass of 11 kt/y (Table 2, Table 3). The mass of the ammonium sulfate accounted for only 5% of the feedstock mass flow when the chemical additions were not considered (Table 3). The remaining stripping residue flow was 85 kt/y with low nitrogen concentration (1.5 kgN/tFM) compared with the untreated digestate liquid (4.0 kgN/kgFM) but with comparable in the concentrations of both phosphorus (0.1 kgP/tFM in the stripping residue and digestate liquid) and potassium (1.9 kgK/tFM in the stripping residue, 1.8 kgK/tFM in the digestate liquid). In total, the amount of outputs from the stripping system was 85 kt/y (ammonium sulfate + stripping residue, Table 2) which was 70% of the feedstock nitrogen, 10% of phosphorus, 85% of potassium and 79% the feedstock mass without chemical additions (Table 3).

In combined stripping + RO treatment (S2), stripping produced the same amount of ammonium sulfate as in a system with stripping only (S1, 11 kt/y), while the RO treatment of stripping residue produced retentate and processed water flows of 21 kt/y and 53 kt/y, respectively (Table 2). The retentate still contained nutrients (5.4 kgN/tFM, 0.2 kgP/tFM, 6.9 kgK/tFM), and was assumed to be used as fertilizer in agriculture (as in Ledda et al., 2013), and not recycled within the digestate liquid treatment as in systems S3 and S4. Thus, in this system the mass of the fertilizer products (ammonium sulfate, retentate) was 32 kt/y (Table 2), and concentrated 70% of the feedstock nitrogen, 10% of phosphorus and 84% of potassium into 26% of the feedstock mass without chemical additions (Table 3).

Evaporation treatment combined with RO (S3) produced only 16 kt/y of nutrient-rich concentrate (17.9 kgN/tFM, 0.3 kgP/tFM, 9.0 kgK/tFM). Subsequently, RO treatment produced 18 kt/y of retentate and 45 kt/y of processed water from the condensate (Table 2). Without chemical additions the fertilizer product from system S3 (concentrate) accounted for 63% of the feedstock nitrogen, 10% of phosphorus, 85% of potassium and 16% of the feedstock mass (Table 3).

When stripping and evaporation were combined with RO (S4) two fertilizer product flows, ammonium sulfate and concentrate, were produced as well as retentate and processed water (11 kt/y, 15 kt/y, 16 kt/y, 42 kt/y, respectively, Table 2). In total, with fertilizer products, ammonium sulfate and concentrate, 67% of the nitrogen, 10% of phosphorus and 85% of potassium from the feedstock was recovered and concentrated into 20% of the mass flow of the feedstock, when the chemical additions were not considered (Table 3). The nitrogen recovery of 67% in S4 was 3% lower compared with system S1 with stripping, which was due to the produced retentate and processed water flows in S4, which contained traces nitrogen but were not considered as fertilizer products. The nutrient concentrations in the concentrate from S4 were similar to the concentrate from S3 (0.3–0.4 kgP/tFM, 9–9.6 kgK/tFM), except for the nitrogen content, which was for the most part recovered with stripping (6.8 kgN/tFM in S4, 17.9 kgN/tFM in S3, Table 2).

The efficiency of the four liquid digestate treatment systems in concentrating nitrogen from the feedstock into fertilizer products was assessed with the ratio between the recovered nitrogen (% of the feedstock) and the recovered mass (% of the feedstock, from Table 3). The resulting efficiencies were 0.9 for systems S0 and S1 and 2.7, 4.0 and 3.4 for systems S2, S3 and S4, respectively, where the increasing ratio demonstrates increasing the concentration of nitrogen into products with low mass. The most efficient system for the digestate liquid treatment was S3, combining both evaporation and RO to produce a fertilizer product with low mass and high nitrogen concentration.

3.2. Energy balance

The energy production in AD was based on the methane production of the FW, around 62 GWh per year, which was utilized in CHP for the production of electricity (24 GWh_{el}/y), and heat (29 GWh_{th}/y, Table 4). In total, AD consumed 9% of the produced total energy as electricity for pretreatment and hygienization of the feedstock (2250 MWh_{el}/y), gas conversion in CHP (1180 MWh_{el}/y) and digestion (1860

Table 2
Mass and nutrient flows presented as tonnes per year (t/y), and concentrations (kg/tFM, in parentheses) in feedstock, digestate, separated solid and liquid digestate fractions and outputs of the digestate liquid treatment systems (S0-S4). Fresh matter (FM), reverse osmosis (RO).

Material	Mass	TS	VS	Ntot	NH_4-N	Ptot	Ktot
Reactor feedstock							
Food waste	60000	15000 (250.0)	13800 (230.0)	450 (7.5)	24 (0.4)	54 (0.9)	168 (2.8)
Dilution water (recycled water in S3-S4)	40000	–	–	–	–	–	–
Biogas, digestate and solid–liquid separation							
Biogas	12586	12586	12586	–	–	–	–
Digestate	87414	2414	1214	450	264	54	168
Liquid digestate, S0 no treatment	78673	483 (6.1)	243 (3.1)	315 (4.0)	214 (2.7)	5 (0.1)	143 (1.8)
Solid digestate, S0-S4	8741	1932 (221.0)	972 (111.1)	135 (15.4)	50 (5.7)	49 (5.6)	25 (2.9)
Digestate liquid treatment							
Stripping, S1							
Ammonium sulfate	11373[a]	0 (0)	0 (0)	203 (40)	203 (40)	0 (0)	0 (0)
Stripping residue	73594[b]	483 (6.4)	243 (3.2)	112 (1.5)	11 (0.1)	5 (0.1)	143 (1.9)
Stripping + RO, S2							
Ammonium sulfate	11373[a]	0 (0)	0 (0)	203 (40)	203 (40)	0 (0)	0 (0)
Stripping residue (to RO)	75168[b]	483 (6.4)	243 (3.2)	112 (1.5)	11 (0.1)	5 (0.1)	143 (1.9)
Retentate	20606	483 (23.4)	243 (11.8)	111 (5.4)	10 (0.5)	5 (0.2)	141 (6.9)
Processed water (recycled)	52988	0 (0)	0 (0)	1 (0.01)	1 (0.01)	0 (0.01)	1 (0.03)
Evaporation + RO, S3							
Concentrate	15820[c]	483 (30.5)	243 (15.4)	284 (17.9)	192 (12.2)	5 (0.3)	143 (9.0)
Condensate (to RO)	62938	0 (0)	0 (0)	32 (0.5)	21 (0.3)	0 (0)	0 (0)
Retentate (recycled)	17623	0 (0)	0 (0)	30 (1.7)	20 (1.2)	0 (0)	0 (0)
Processed water (recycled)	45316	0 (0)	0 (0)	1 (0.02)	1 (0.02)	0 (0)	0 (0)
Stripping + evaporation + RO, S4							
Ammonium sulfate	11373[a]	0 (0)	0 (0)	203 (40)	203 (40)	0 (0)	0 (0)
Stripping residue (to evaporation)	73594[b]	483 (6.4)	243 (3.2)	112 (1.5)	11 (0.1)	5 (0.1)	143 (1.9)
Concentrate	14798[d]	483 (32.6)	243 (16.4)	101 (6.8)	10 (0.7)	5 (0.4)	143 (9.6)
Condensate (to RO)	58875	0 (0)	0 (0)	11 (0.2)	1 (0.02)	0 (0)	0 (0)
Retentate (recycled)	16485	0 (0)	0 (0)	11 (0.7)	1 (0.1)	0 (0)	0 (0)
Processed water (recycled)	42390	0 (0)	0 (0)	0 (0.001)	0 (0.001)	0 (0)	0 (0)

–, not applicable.
[a] H_2SO_4 addition of 6293 m^3/y.
[b] NaOH addition of 1573 m^3/y.
[c] H_2SO_4 addition of 85 m^3/y.
[d] $H_2SO_4$80 m^3/y.

MWh_{el}/y). The heat consumption of the AD in total was 10% of the produced total energy including hygienization (4200 MWh_{th}/y), digester (1200 MWh_{th}/y) and heat losses (800 MWh_{th}/y) (Table 4).

All the four studied digestate treatment systems (S1-S4) and the reference system (S0) included solid–liquid separation with a centrifuge, which used 300 MWh of electricity per year (0.5% of the produced energy, Table 4) as the material electricity consumption was calculated per produced digestate. The digestate stripping (S1, S2) had an electricity demand of 400 MWh_{el}/y, while the addition of the RO treatment (S2) increased the electricity demand by 190 MWh_{el}/y (total 590 MWh_{el}/y in S2). Evaporation (S3) consumed a similar amount as stripping and the electricity consumption of RO was 160 MWh_{el}/y (total 550 MWh_{el}/y in S3). In the system combining stripping + evaporation + RO (S4), the electricity consumption in total was 920 MWh_{el}/y, which accounted 1.5% of the energy produced in AD. The heat demand in all four studied treatment systems (S1-S4) was similar, around 3700 MWh/y (6% of the produced energy in AD), as the heat consumption for both stripping and evaporation was similar, RO did not use any heat, and in the system combining stripping + evaporation + RO (S4) the heat demand was allocated only for stripping (Table 4).

The total energy consumption of the digestate liquid treatment systems increased from systems S1 to S4 as the units in the treatment of digestate liquid increased being 4100, 4300, 4200 and 4600 MWh/y in systems S1, S2, S3 and S4, respectively (Table 4). The combined energy consumption of the digestate treatment

Table 3
The partition of feedstock mass and nutrients in the digestate, separated solid and liquid digestates and fertilizer products from the digestate liquid treatment systems (S0-S4). The partition is presented as % of the feedstock. The mass is calculated from the feedstock fed to the digester (60 kt food waste + 40 kt dilution water). The addition of chemicals is not taken into consideration in the mass partition. Reverse osmosis (RO).

% of feedstock	Mass	Ntot	NH_4-N	Ptot	Ktot
Digestate and solid–liquid separation					
Digestate	87	100	1100	100	100
Liquid digestate, S0	79	70	891	10	85
Solid digestate, S0-S4	9	30	209	90	15
Stripping, S1					
Ammonium sulfate	5	45	846	0	0
Stripping residue	74	25	45	10	85
Fertilizer products in total	79	70	891	10	85
Stripping + RO, S2					
Ammonium sulfate	5	45	846	0	0
Retentate	21	25	42	10	84
Fertilizer products in total	26	70	888	10	84
Evaporation + RO, S3					
Concentrate	16	63	802	10	85
Stripping + evaporation + RO, S4					
Ammonium sulfate	5	45	846	0	0
Concentrate	15	22	40	10	85
Fertilizer products in total	20	67	886	10	85

systems and AD was 15.9–16.4 GWh, which accounted for 26% of the total energy produced.

The energy consumption of the digestate liquid treatment per recovered nitrogen in the concentrated fertilizer products (ammonium sulfate, concentrate) were the lowest (150 kWh/kgN) with the following systems: evaporation + RO (S3); stripping + evaporation + RO (S4) (Table 4). In systems with stripping (S1 and S2) the energy consumption was higher (200–210 kWh/kgN) due to the lower amount of nitrogen in the produced ammonium sulfate compared with the products from systems S3 and S4. However, when only electricity consumption was taken into consideration, the evaporation + RO (S3) and stripping (S1) had lower electricity consumptions per recovered nitrogen (19–20 kWh_{el}/kgN in S1 and S3, around 30 kWh_{el}/kgN in S2 and S4, Table 4) as the electricity demand per year was low and nitrogen recovery efficiency higher compared with the systems stripping + RO (S2) and stripping + evaporation + RO (S4).

3.3. Transportation of fertilizer products

The energy consumption associated with the transportation of the fertilizer products in the digestate liquid treatment systems (S0-S4) was evaluated in relation to the increasing transportation distance (from 10 to 250 km, Fig. 3). Of the studied products, the concentrate from the evaporation treatment (S3) used less than 700 MWh/y during 250 km transportation, and was considered as the most energy efficient to transport longer distances. In contrast, the transportation the digestate liquid as such (S0) and stripping residue from S1 consumed the largest amount of energy (around 700 MWh/y transportation distance 50 km, 3300–3600 MWh/y distance 250 km) (Fig. 3, Table 2).

4. Discussion

4.1. Energy consumption of AD, digestate liquid treatment and transportation

The present results, based on typical literature values from laboratory, pilot and full scale studies, show that the processing of digestate through solid–liquid separation and digestate liquid treatments into concentrated fertilizer products consumed less than 10% of the produced energy in an AD plant treating 60 kt/y of FW. In total AD, solid–liquid separation and digestate liquid treatment accounted for 26% of the produced energy, of which around 19% was used in the AD and separation of the digestate into solid and liquid fractions. The lower energy consumption of only 8–17% of the total energy produced was previously reported for a theoretical AD plant treating FW (90 kt/y) combined with ammonia stripping and hydrogen recovery, where the additional energy input from the assumed produced hydrogen most likely lowered the ratio between energy input and output (Babson et al., 2013). Compared with the present study, similar energy demand, 17% and 20% of the total energy production, was previously assumed in life cycle assessment studies for ADs combined with digestate solid–liquid separation treating 20–60 kt/y of an organic fraction of municipal solid waste and a mixture of municipal and agricultural substrates (Berglund and Börjesson, 2006; Pöschl et al., 2010; respectively). In the present study the differences in the total energy produced in AD and the lower ratio between energy demand and total energy production compared with a study by Berglund and Börjesson (2006) were due to the high energy content of the FW feedstock in the present study (450 m^3CH_4/tVS compared to around 300 m^3CH_4/tVS in Berglund and Börjesson, 2006). If similar digestate liquid treatment systems as those studied in this paper would be applied to an AD plant treating solely, e.g., manure, which produces less methane and has lower VS content (cow manure 148 m^3CH_4/tVS, VS 11%, Møller et al., 2004), the total energy production of the plant would be lower (9800 MWh/y) and digestate treatment (solids separation and liquid treatment) would require all of the feedstock's energy and the energy balance would be negative

Table 4
Energy production of the studied anaerobic digestion (AD) plant and the energy consumption during AD (including pretreatment and hygienization, biogas upgrading and digester), digestate separation and digestate liquid treatment. Energy consumption of the digestate liquid treatment is calculated towards the recovered concentrated fertilizer fractions (ammonium sulfate, concentrate), where stripping residue from S1 and retentate from S2 were not included. Reverse osmosis (RO), combined heat and power (CHP), electricity (el), thermal (th).

Process	Electricity (MWh_{el}/y)	Heat (MWh_{th}/y)	Total (MWh/y)	Total (kWh/kgN)	Electricity (kWh_{el}/kgN)
Energy production					
Primary energy production in AD	–	–	62100	–	–
Energy in CHP	23598	29187	52785	–	–
Energy consumption					
AD	5293	6142	11435	–	–
Solid–liquid separation	306	–	306	–	–
Energy consumption in digestate liquid treatment					
No treatment, S0	0	0	0	0	0
Stripping, S1	406	3727	4133	203	20
Stripping + RO, S2	590	3727	4317	213	29
Evaporation + RO, S3	551	3658	4209	148	19
Stripping + evaporation + RO, S4	922	3727	4649	153	30

–, not applicable.

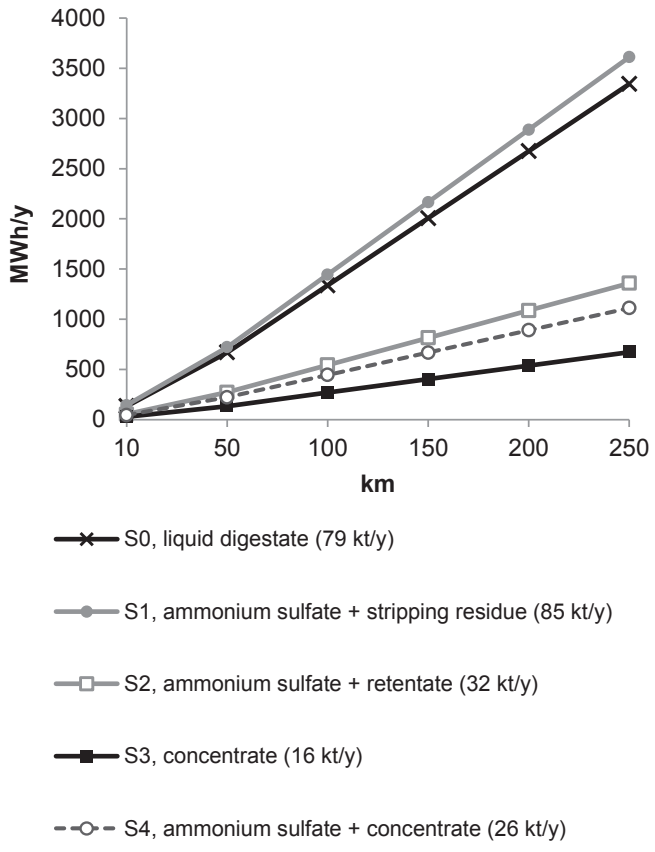

Fig. 3. Energy consumption of transportation (megawatt hours per year, MWh/y) of fertilizer products produced in each digestate liquid treatment system in relation to the transportation distance.

(900 MWh/y needed in addition to the AD plant's energy production). In this sense, the digestate liquid treatment systems are more rational options when feedstock has a high methane potential and initial organic matter and nutrient concentrations, such as FW.

Heat consumption of all the studied digestate liquid treatments accounted for 80–90% of the total energy demand, because with both stripping and evaporation increased process temperature (80 °C applied in this study) is needed to achieve efficient nutrient recovery (Mehta et al., 2015). As the electricity consumption between stripping and evaporation was similar (390–400 MWh/y), the total energy consumption was 4100 kWh/y with both stripping and evaporation, while the RO treatment consumed only electricity (150–180 MWh/y) as no heat was required. Similarly, evaporation treatment was reported to consume over 50% more energy compared with the treatment with combined microfiltration, reverse osmosis and ion exchange when digestate originated from a mixture of manure and plant materials (AD with the electric power of 186 kW_{el}, Rehl and Müller, 2011). Accordingly, due to the large heat demand of digestate liquid treatments with stripping and evaporation, the use of these treatments is rationalized by integration with AD, which enables the recovery of the plant's excess heat (Bonmatí et al., 2003; Fuchs and Drosg, 2013; Hjorth et al., 2010; Mehta et al., 2015), especially in situations where the AD plant's heat energy is not utilized in, e.g., district heating systems.

The results showed that the nitrogen recovery from the digestate liquid treatments consumed 150–210 kWh/kgN, as the consumption was allocated to the recovered nitrogen in the concentrated fertilizer products. This is over 15 times more than the energy consumption of mineral nitrogen fertilizer production in Europe (35.2 GJ/tNH_3 ~ 9.8 $kWh/kgNH_3$, Yara, 2015a). However, despite the higher energy consumption of the treatment of digestate liquids, the advantage with the use of liquid fertilizers from waste materials is the promotion of nutrient recycling and mitigation of greenhouse gas emissions through anaerobic digestion compared to the manufacturing of mineral fertilizers (Evangelisti et al., 2014). The energy demand in all four digestate liquid treatments combining two to three treatment technologies was also higher compared to a pilot-scale stripping treatment with urine (18.8–28.2 kWh/kgN recovered, Antonini et al., 2011), where the lower treatment temperature (40 °C vs. 80 °C in the present study) decreased energy consumption. Lower energy consumption was also reviewed with ion exchange, membrane distillation and chemical precipitation technologies (0.04–0.63 $kWh/kgNH_3$, Zarebska et al., 2015), where the treatments did not require heat energy for nutrient recovery. The energy consumption of the studied digestate liquid treatment technologies could be, however, reduced by using lower process temperatures. For example successful ammonia stripping (N recovery >80%) has also been reported at temperatures from 35 to 50 °C (Antonini et al., 2011; Laureni et al., 2013; Liu et al., 2015), while the use of lower temperatures (35–40 °C) with evaporation is possible with the increase of vacuum (pressure 5–7 bars, Bonmatí and Flotats, 2003b; Chiumenti et al., 2013). Additionally, the heat consumption of the treatment processes could be reduced by using heat exchangers to recycle the process heat, which was not taken into consideration in this study.

As the energy consumption during transportation was affected by the mass of fertilizer products (from 16 to 87 kt/y), up to an 80% decrease in the energy consumption during transportation of the fertilizer products was possible using the studied digestate liquid treatment systems when compared to the transportation of the untreated digestate liquid. Energy savings of 80%, 67% and 59% during transportation were possible with the following treatment systems, respectively: evaporation combined with RO (S3); combined stripping, evaporation and RO (S4); stripping combined with RO (S2). This was due to the lower transportable mass compared with the untreated digestate liquid in the reference system (S0). Decreased energy consumption of transportation supports the use of these digestate liquid treatments in AD plants treating FW, which are usually located far from agricultural lands (Babson et al., 2013) and where the reuse of the FW nutrients is challenging due to long transportation distances. Similar energy savings from transportation of municipal waste-based digestate have been reported after solid–liquid separation, where the energy consumption of transportation of solid digestate after separation decreased 50% (distance 5 km, Pöschl et al., 2010), thus the transportation of the liquid digestate was not discussed. The advantage with the digestate liquid treatment systems applying RO treatment is the reduction of the total mass of the transportable products compared with the untreated digestate liquid, as some mass exits the system as treated water and retentate. With stripping only (S1), where no RO was included in treatment of digestate liquid, the mass of ammonia sulfate was low, but with the stripping residue also aimed for use in agriculture, the total mass of products was high (87 kt/y), which is feasible to transport only by minimizing the distance between the AD plant and the fields to be fertilized. A high total mass of 87 kt/y with stripping (S1) was due to the addition of chemicals during stripping which also led to 10% higher energy consumption during transportation compared to the reference system (S0).

4.2. Characteristics of fertilizer products

The results, based on typical literature values, showed that liquid digestate treatment with evaporation, combined with RO (S3), produced the most concentrated nutrient product by

concentrating the original FW mass of 60 kt/y into 16 kt/y. The high nitrogen and potassium and low phosphorus concentrations within the concentrate (18 kgN/tFM, 12 kgNH_4-N/tFM, 0.3 kgP/tFM, 9 kgK/tFM) compared with the untreated digestate liquid (4 kgN/tFM, 2.7 kgNH_4-N/tFM, 0.1 kgP/tFM, 1.8 kgK/tFM), were dependent on the mass and nutrient recovery and characteristics of the feedstock. Previously similar (18.7 kgN/tFM) and slightly higher (>20 kgN/tFM) nitrogen concentrations have been reported after acid evaporation with pig slurry digestate (Chiumenti et al., 2013; Bonmatí and Flotats, 2003b; respectively). Compared with the concentrate from evaporation, commercial mineral fertilizers in solid form have remarkably higher nutrient concentrations (e.g. 303 kgN/tFM, 114 kgP/tFM, 245 kgK/tFM, Abubaker et al., 2012, 266 kgN/tFM, 13 kgP/tFM, 43 kgK/tFM, Yara, 2015b), thus, the characteristics of N and K were in line with commercial liquid fertilizers intended for, e.g., vegetables fertilization (24 kgN/tFM, 55 kgP/tFM, 40 kgK/tFM, Yara, 2015c). Hence, the concentrate from evaporation could potentially replace liquid mineral fertilizers, especially in cases where phosphorus fertilization is not needed.

All four studied digestate liquid treatment systems produced fertilizer products containing either N (ammonium sulfate) or N, P and K (concentrate, retentate, stripping residue) in different proportions, which affect their use as fertilizers in agriculture and also affect the amount of fertilizers spread on agricultural lands. The proportion of mineral nitrogen in total N (NH_4-N/Ntot) was 100% in the ammonium sulfate from stripping (S1, S2, S4) and 68% in the concentrate from evaporation (S3), indicating the high availability of N to plants and fast growth response after fertilization (Abubaker et al., 2012). In concentrate from combined stripping, evaporation and RO (S4), retentate from stripping and RO (S2) and stripping residue from stripping alone (S1) the NH_4-N/Ntot was 10%, 9% and 7%, respectively, indicating slower N release in soils. When considering the NPK ratios, potassium was the dominant nutrient in all fertilizer products (except ammonium sulfate), as the NPK ratios (per FM) were around 100:5:130 in the stripping residue from stripping (S1), the retentate from stripping combined with RO (S2) and the concentrate from combined stripping, evaporation and RO (S4). The concentrate from the system combining evaporation and RO (S3) showed the most balanced NPK ratio of 100:2:50, which was also somewhat similar to the NPK need of herbaceous plants, 100:14:68 (Knecht and Göransson, 2004), thus with remarkably lower P content. The low share of phosphorus was due to the digestate solid–liquid separation, which distributed only 10% of the P in FW into the liquid digestate, while the water-soluble N and K were the predominant nutrients. As follows, all the studied fertilizer products can be used in agriculture supplementing especially N or N and K fertilization in situations where the soil P content is already high.

The low nutrient concentration within especially stripping residue (from stripping, S1, 1.5 kgN/tFM, 0.1 kgNH_4-N/tFM, 0.1 kgP/tFM, 1.9 kgK/tFM) increases the fertilizer application amounts per hectare and discourages the use of the residue in agriculture. The amount of the stripping residue to be spread during fertilization (assumed N fertilization rate 170kgN/ha) was high, over 100 t/ha, due to the low nutrient concentrations, which could affect the soil properties due to soil wetting/water logging (Rigby and Smith, 2011). More practical volumes were achieved with fertilizer products from other treatment systems, as the amount of mass applied to soils was 4 t/ha for ammonium sulfate from stripping (S1, S2, S4), 9 t/ha for concentrate from evaporation and RO (S3) and around 25–30 t/ha for concentrate from stripping, evaporation and RO (S4) and retentate from stripping and RO (S2).

All in all, the most suitable digestate liquid treatment system for concentrated fertilizer products considering nutrients, energy and transportability was the concentrate from the system with evaporation and RO (S3), thus, the production of a different fertilizer product (e.g. ammonium sulfate) is possible with slightly larger energy input with a system combining stripping, evaporation and RO (S4) or stripping and RO (S2). However, along with the recovery of useful nutrients the evaporation and also RO treatment are able to concentrate some undesired components, such as heavy metals, into the concentrate, which could affect the fertilizer use of the products (Mehta et al., 2015). Furthermore, the addition of chemicals to the digestate liquid during the treatment, and their effects on soil after fertilization, should be noticed. E.g. sulfuric acid additions during/after stripping (S1, S2, S4) and before evaporation (S3, S4), both lower the pH value and increase the salinity, which are likely to cause corrosion (Vaneeckhaute et al., 2013b). Like sulfur, also sodium acts as micronutrient in plant nutrition. However, in large doses sodium increases the soil salinity and affects the soil structure (reviewed in Kronzucker et al., 2013; Vaneeckhaute et al., 2013a). The Na concentration in the stripping residue from system S1 was calculated to be around 6 g/kgFM which is about double the amount of Na in manure and manure based digestate (Vaneeckhaute et al., 2013a,b). It is thus important to measure the pH and salinity of the produced liquid fertilizer products and monitor the effect of these products in soils after fertilization.

5. Conclusions

This theoretical study showed the feasibility of FW nutrient recovery through AD and digestate liquid treatment and the production of transportable fertilizer products with the energy produced in AD. Despite the use of heat-demanding treatments, such as evaporation and stripping, the energy produced in AD was sufficient for digestate liquid treatment consuming fewer than 10% of the total energy produced in AD. The studied digestate liquid treatment systems were mostly considered as nitrogen concentration methods which are able to concentrate up to 67% of feedstock nitrogen into liquid fertilizer products with low mass. Of the studied digestate systems evaporation combined with RO was evaluated as the most efficient nutrient recovery technology for the production of transportable fertilizer products for agricultural application due to the highest concentration of nutrients, nutrient availability, the low mass of the product and low energy consumption of the treatment. Stripping was an efficient technology for the recovery of nitrogen, however, the high mass of the residue containing the remaining K and P should be further managed sustainably. Overall, the selection of the treatment technology is dependent on the location of the AD plant relative to agricultural lands and the type of fertilizer products needed (N fertilizer, NPK fertilizer).

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jclepro.2016.03.127.

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Supplementary Material for

Liquid fertilizer products from anaerobic digestion of food waste: mass, nutrient and energy balance of four digestate liquid treatment systems

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1. Hygienization and digester

1.1 Mass balances

The volume of methane produced during anaerobic digestion per year (AD, m^3/y) was calculated by multiplying the amount of volatile solids (VS) fed to the reactor (t/y) with the methane potential of the feedstock (m^3/tVS). The mass of methane was calculated according to equation:

$$m = V \times \rho / 1000$$

where the m is the mass of CH_4 (tFM/y), V is the volume of CH_4 (6.21 Mm^3/y) and ρ is the density of CH_4 (0.72 kg/m^3).

The volume of CO_2 annually produced in the AD was calculated using the carbon dioxide content (40%), the assumed methane content (60%) and the volume of methane (6.21 Mm^3/y). The mass of CO_2 was calculated the same way as the mass of CH_4 using the CO_2 density of 1.96 kg/m^3. Subsequently, the digestate mass was calculated by subtracting the biogas mass (CH_4 + CO_2, 12 586 t/y) from the feedstock mass.

For the calculation of the mass balances of total solids (TS) and VS, it was assumed that the produced biogas was produced from TS and VS. Subsequently, the TS and VS contents of the produced digestate were calculated as the difference between feedstock TS or VS (t/y) and biogas mass (t/y). The nutrient content in digestate was assumed to be the same as in the feedstock except in the case of NH_4-N, which concentration was assumed to increase from 0.4 g/kg fresh matter (FM) (feedstock) to 4 g/kgFM (digestate) according to Tampio et al. (2014, 2015).

1.2 Energy balances

In the hygienization unit the feedstock (60 kt of food waste) was heated to 75 °C. The amount of energy needed for feedstock mixture heating was calculated with the specific heat capacity of water according to the equation:

$$\Delta E = c \times m \times \Delta t$$

where ΔE is the energy needed for heating the feedstock mixture, c is the specific heat capacity of water (c_{water} = 4.18 kJ/kg°C), m is the mass of the mixture (kg) and Δt is the change of the temperature (from 15 to 75 °C). For the digester, the amount of energy needed was allocated only for the dilution water (40 kt/y). This is because the heat from the hygienization unit was assumed to be sufficient for the heating of the FW feedstock (60 kt/y) (Berglund and Börjesson 2006, Prapaspongsa et al. 2010). The heating of water from 15 to 40 °C was calculated with the specific heat capacity of water as above. A similar method, based on the heat capacity of water for the calculation of the heat demand, was also used e.g. by Bacenetti et al. (2013), Rapport et al. (2011) and Smyth et al. (2009).

For the biogas production the conversion factor 1 $m^3(CH_4)$ = 10 kWh was used for the calculation of the energy content of the produced biogas.

Heat loss from the hygienization and digester was assumed to be 15% of the heat demand according to Smyth et al. (2009). Also higher values for digester heat loss have been reported, e.g., 20% of the heat demand in Rapport et al. (2011).

The electricity and heat consumption during feed pretreatment and hygienization as well as during digestion and gas upgrading in CHP-unit (combined heat and power) were calculated according to the literature values (Table S1). The CHP efficiency was also chosen according to the literature information (Table S1).

Table S1. Energy consumption of hygienization and pretreatment, anaerobic digestion and gas upgrading in CHP obtained from the literature and values chosen for the calculations.

Energy consumption	Electricity	Heat	Reference
Pretreatment + hygienization			
	150 kWh/tTS	10% of heat in CHP	reviewed in Pöschl et al. 2010
	37.5 kWh/t	Calculated value	Present study
Digester			
	3% biogas	9.6% biogas	
	(18.6 kWh/t[a])	(55.8 kWh/t)[a]	reviewed in Pöschl et al. 2010
	7.5% of electricity in CHP	20% of heat in CHP	
	(17.7 kWh/t)[a]	(47.2 kWh/t)[a]	reviewed in Pöschl et al. 2010
	18 (15-22) kWh/t	31 (19-50) kWh/t	reviewed in Berglund and Börjesson 2006
	5 kWh/t biomass	34 kWh/m^3 raw material	DEA 2005
	18 kWh/t	Calculated value	Present study
CHP-unit			
	4.5 % of electricity in CHP	-	Pöschl et al. 2010
	6.3% of electricity in CHP	-	Havukainen et al. 2014
	8.5-8.7% of electricity in CHP	-	Naegele et al. 2012
	4.2% of electricity in CHP	-	Banks et al. 2011
	5 % of electricity in CHP	-	Present study
CHP efficiency			
	40.9 %	44 %	Bacenetti et al. 2013
	37 %	47.1 %	Bacenetti et al. 2013
	35.7 %	51 %	Bacenetti et al. 2013
	40 %	48 %	Poeschl et al. 2012
	38 %	47 %	Present study

[a]Calculated with the results from the present study
-, not available

2. Digestate separation

The digestate solid-liquid separation efficiency and energy consumption of a decanter centrifuge were based on the literature values (Table S2).

Table S2. Centrifuge separation efficiencies and energy consumption obtained from the literature and values chosen for the calculations. Separation efficiency presented as the percentage in the liquid fraction. Digestate (D), digestate liquid (DL), manure (M).

Separation efficiency, %							Energy	Material	Reference
Mass	TS	VS	N	NH_4-N	P	K	Electricity (kWh/m^3 digestate)		
-	31-46	-	69-76	-	9-48	-	3.1-5.6	D	Møller et al. 2002
91	36	-	72	89	10	99	-	DL_{pig}	Ledda et al. 2013
76	17	-	48	81	4	77	-	DL_{cow}	Ledda et al. 2013
22.5	-	-	87	-	19	33.5	1.8-2.2	M_{pig}	Melse and Verdoes 2005
-	38-67	-	71-87	-	34-40	-	4.3-6.3	M_{pig}	Møller et al. 2002
-	35-45	-	51-73	-	18-22	-	4.3-7.3	M_{cow}	Møller et al. 2002
75-95	5-66	-	46-99	72-92	9-52	-	-	$M_{pig,\ cow}$	reviewed in Hjorth et al. 2010
-	-	-	-	-	-	-	2-4	-	Flotats et al. 2011
-	-	-	-	-	-	-	3	-	Møller et al. 2000
90	20	20	70	81	10	85	3.5	-	Present study

-, not available

3. Stripping of digestate liquid

The digestate liquid stripping efficiency and energy consumption were based on the literature values (Table S3). The heat demand of the stripping was calculated with the temperature change between digester (40 °C) and stripper (80 °C) using the heating capacity of water as in Chapter 1.2 of the Supplementary material.

Table S3. Recovery efficiency, process parameters and energy consumption of stripping obtained from the literature and values chosen for the calculations. Recovery efficiency presented as percentage in ammonium sulfate. Digestate (D), manure (M), digestate liquid (DL), urine (U).

Recovery efficiency (%)		Process parameters		Consumption	Scale	Material	Reference
N	NH_4-N	pH	Tempera-ture (°C)	Energy (kWh/kgN recovered)			
-	97	-	35	-	lab	D	Liu et al. 2015
-	>96	8.5-11.5	80	-	lab	D	Bonmatí and Flotats 2003a
-	>80	9.5	40-50	-	lab	D, M_{pig}	Laureni et al. 2013
65-80	80-92.2	8.5-11	30-70	-	pilot	DL	Guštin and Marinšek-Logar 2011
65-76	-	9-9.5	60	-	full	DL	Morales et al. 2013
94	-	10	40	18.8-28.2[a]	pilot	U	Antonini et al. 2011
-	92	12	16	-	lab	U	Basakcilardan-Kabakci et al. 2007
-	65-98.8	7.7-11.5	80	-	lab	M_{pig}	Bonmatí and Flotats 2003a
90	-	9.3	60	11-14	full	-	reviewed in Morales et al. 2013
-	-	-	-	7.3 (aeration)	-	-	reviewed in Mauer et al. 2003
-	-	-	-	0.8-23 (electricity)	-	-	reviewed in van Eekert et al. 2012
-	95	9-10	<100	-	-	-	Flotats et al. 2011
-	95	-	80	2 + heat[b]	-	-	Present study

[a] kWh/kg NH_4-N

[b]Calculated with the specific heat capacity of water

-, not available

4. Reverse osmosis treatment for stripping residue and condensate

The recovery efficiency and energy consumption of the reverse osmosis (RO) treatment were based on the literature values (Table S4).

Table S4. Recovery efficiency, process parameters and energy consumption of reverse osmosis obtained from the literature and values chosen for the calculations. Recovery efficiency presented as percentage in retentate. Digestate (D), digestate liquid (DL), urine (U), sewage reject water (S), manure (M).

Separation/recovery efficiency, %							Process parameters			Consumption	Scale	Material	Reference
Mass	TS	VS	N	NH_4-N	P	K	pH	Tempera-ture (°C)	Pres-sure (bar)	Energy (kWh/m^3 stripping residue/condensate)			
-	-	-	-	-	-	-	-	-	10-30	2.5-10	lab	D	Carretier et al. 2015
28	86-100	-	99.7	99.6	72	99.5	-	-	-	-	full	DL	Ledda et al. 2013
29	97-100	-	97	97	100	99	-	-	-	-	full	DL	Ledda et al. 2013
28	-	-	-	-	-	-	-	-	-	-	full	DL	Chiumenti et al. 2010
-	-	-	95	-	90	99	6-9.2	10-45	-	8 (electricity) 4 (heat)	lab	U	Ek et al. 2006
-	-	-	90	-	92	97	6-9.2	10-45	-	5 (electricity) 0 (heat)	lab	S	Ek et al. 2006
-	92.3	98-100	-	66	-	-	8.8	22.5	55		lab	M_{pig}[a]	Mondor et al. 2008
-	-	99	-	99.5	-	-	-	-	10-100	1.5-10	-	-	Flotats et al. 2011
28	100	100	-	95	95	99	-	-	-	2.5	-	-	Present study

[a]After electrodialysis treatment

-, not available

5. Evaporation treatment for digestate liquid and stripping residue

The digestate liquid evaporation efficiency and energy consumption were based on the literature values (Table S5). The heat demand of evaporation was calculated as the temperature change between the digester (40 °C) and evaporator (80 °C) using the heating capacity of water as in Chapter 1.2 of the Supplementary material. In system stripping + evaporation + RO (S4) no heat was allocated for evaporation, thus it was assumed that heat from the stripping (80 °C) was sufficient for the evaporation as was presented in Ervasti et al. (2011).

Table S5. Recovery efficiency, process parameters and energy consumption of evaporation obtained from the literature and values chosen for the calculations. Recovery efficiency presented as percentage in concentrate. Digestate (D), digestate urine (U), sewage reject water (S).

Recovery efficiency, %				Process parameters			Consumption	Scale	Material	Reference
Mass	N	P	K	pH	Temper-ature (°C)	Pres-sure (bar)	Energy (kWh/m^3 digestate liquid or stripping residue)			
-	80-84	84-96	90-99	5.9-6.5	40	6.7	-	lab	D	Bonmatí and Flotats 2003b
20.2	99.2	-	-	3.5-5	35	5.3	-	-	D	Chiumenti et al. 2013
10	-	-	-	-	78	0.2	1.9[a] (electricity) 3[a] (fuel)	-	U	Mauer et al. 2003
-	95	100	99	4.5-5.5	>30	-	30 (electricity) 0 (heat)	lab	U	Ek et al. 2006
-	95	100	100	4.5-5.5	>30	-	30 (electricity) 0 (heat)	lab	S	Ek et al. 2006
15-20	98	-	-	<5.5	-	-	21 (electricity) 107-353 (heat)	-	-	Flotats et al. 2011
20	90	100	100	-	80	-	30 + heat[b]	-	-	Present study

[a]kWh/kgN

[b]Calculated with the specific heat capacity of water

-, not available

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