

Master's Thesis

**Thermophilic Anaerobic Digestion of Source Separated
Institutional Food Waste and Kitchen Waste**

Jari Koponen



University of Jyväskylä

Department of Biological and Environmental Sciences

Environmental Science and Technology

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UNIVERSITY OF JYVÄSKYLÄ, Faculty of Mathematics and Science
Department of Biological and Environmental Science
Environmental Science and Technology

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Supervisors: Dr. Prasad Kaparaju & Dr. Leena Sivula
Inspectors: Prof. Jussi Kukkonen & Dr. Prasad Kaparaju
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ABSTRACT

Food wastage in Europe is increasing, and measures for the reduction and for the utilization of wastes should be taken in order to reduce economic and environmental impacts. Food waste is an attractive feedstock for anaerobic digestion as it has high methane potential, due to its composition with lipids and proteins. Present thesis work assesses thermophilic digestion (55 °C) of food wastes and kitchen wastes collected from University of Jyväskylä restaurant Ylistö. The produced waste quantities from the restaurant were estimated. The composition and the chemical characteristics of the wastes was analyzed, and the substrate performance was studied in batch and reactor (CSTR) experiments. Overall ca. 2780 kg of kitchen waste and ca. 9450 kg of food waste are estimated to be produced annually. Food waste had average total solids (TS) and volatile solids (VS) content of 28-32% and 27-30% and kitchen waste 16-23% and 15-22%, respectively. Methane yields for food waste and kitchen waste in batch assays performed at 55 °C were 174 and 186 mL gVS⁻¹, respectively. Digesters receiving wastes was monitored over period of 210 days, in order to identify the process performance, applicable organic loading rates (OLR) and methane yields in thermophilic process. The highest specific methane yield of food waste in reactor experiments with OLR 6 gVS L⁻¹ d⁻¹ and retention time (HRT) 30 d was 399 mL gVS⁻¹. At same OLR and HRT, highest specific methane yield for kitchen waste was 433 mL gVS⁻¹. Increased volatile fatty acid (VFA) concentrations and ammonia inhibition related to mono-digestion, elevated process temperature and the composition of feedstock occurred at OLR above 3 gVS L⁻¹ d⁻¹. With determined institution specific waste production ratio of 4.5:1 food to kitchen waste, present study had highest specific methane yield of 354 mL gVS⁻¹ at OLR 6 gVS L⁻¹ d⁻¹ and least signs of inhibition. Nevertheless, the maximum OLR that could sustainably be used in longer time period in order to maintain stable methane production under thermophilic process is around 3 gVS L⁻¹ d⁻¹ that in co-digestion of food waste and kitchen waste with HRT 30 d yields methane 150 mL gVS⁻¹.

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TIIVISTELMÄ

Ruokajätteen syntymäärä Euroopassa on kasvussa, toimia ruokahävikin ja ruokajätteen vähentämiseksi tulisi tehdä negatiivisten talous- ja ympäristövaikutusten minimoimiseksi. Ruokajätteillä on korkea energiasisältö, niiden sisältäessä paljon mm. rasvoja ja proteiineja. Tämän vuoksi ne ovat potentiaalisia syötemateriaaleja anaerobiseen käsittelyyn. Tässä työssä tutkittiin Jyväskylän Yliopiston kampusravintola Ylistössä syntyviä ruoka- ja keittiöjätteitä, sekä niiden soveltuvuutta termofiiliseen (55 °C) anaerobiseen käsittelyprosessiin. Keittiöstä syntyvien ruoanvalmistusjätteiden ja asiakkaiden tuottamien tähteiden syntymäärää ja koostumusta, sekä niiden kemiallisia ominaisuuksia analysoitiin. Jättemateriaalien metaanipotentiaalit arvioitiin panoskokein. Lisäksi tutkittiin jättemateriaalien metaanintuottoa eri syötemäärillä ja kuormitustasoilla laboratoriossa termofiilisessä lämpötilassa jatkuvatoimisilla täyssekoitteisilla reaktoreilla (CSTR). Vuodessa keittiöjätettä syntyy arviolta 2780 kg ja ruokajätettä 9450 kg. Ruokajätteen kuiva-ainepitoisuus (TS) oli 28-32% ja orgaanisen kuiva-aineen pitoisuus (VS) oli 27-30%. Keittiöjätteelle TS- ja VS -pitoisuudet olivat 16-23% ja 15-22%. Panoskokeissa metaanipotentiaali ruokajätteelle oli 174 mL gVS⁻¹ ja keittiöjätteelle 186 mL gVS⁻¹. Reaktorikokeissa korkein ominaismetaanintuotto ruokajätteelle kuormituksella (OLR) 6 gVS L⁻¹ d⁻¹ ja 30 vuorokauden viipymällä (HRT) oli 399 mL gVS⁻¹ ja keittiöjätteelle 433 mL gVS⁻¹. Reaktorikokeissa, jotka suoritettiin 210 päivän ajan, molemmat jätejakeet reaktoreissa toimivat epävakaasti 3 gVS L⁻¹ d⁻¹ korkeammilla syötemäärillä. Ruoka- ja keittiöjätettä syötettäessä rinnakkaissyöttönä, syötesuhteella 4.5:1, korkein ominaismetaanintuotto syötemäärällä 6 gVS L⁻¹ d⁻¹ oli 354 mL gVS⁻¹. Lisäksi haitalliset vapaan ammoniakkin ja haihtuvien rasvahappojen (VFA) pitoisuudet reaktorissa olivat matalammat verrattuna syötemateriaalien erikseen käsittelyssä. Tutkimuksessa havaittiin että termofiilisessä anaerobisessa prosessissa 3 gVS L⁻¹ d⁻¹ syötemäärää korkeammilla määrillä ammoniakki- ja VFA -pitoisuudet alkavat akkumuloidua, jotka pitkällä aikavälillä johtavat käsittelyprosessin kaatumiseen. Ruoka- ja keittiöjätteen yhteiskäsittely termofiilisessä prosessissa pitkällä aikavälillä on mahdollista korkeintaan 3 gVS L⁻¹ d⁻¹ kuormituksella ja 30 päivän viipymällä, jolloin ominaismetaanintuotto oli keskimäärin 150 mL gVS⁻¹.

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ABBREVIATIONS

FM	Fresh Matter
Free NH ₃	Free ammonia
HRT	Hydraulic Retention Time
LCFA	Long-Chain Fatty Acid
NH ₄ ⁺	Ammonium
NH ₄ -N	Ammonium nitrogen
OLR	Organic Loading Rate
SCOD	Soluble Chemical Oxygen Demand
TKN	Total Kjeldahl Nitrogen
TS	Total Solids
TVFA	Total Volatile Fatty Acids
VS	Volatile Solids
VFA	Volatile Fatty Acids
w/w	Wet weight

1 INTRODUCTION

One of the most significant environmental issues in Europe is continuously increasing waste production since society has grown and become wealthier. European Union produces up to 3 billion tonnes of waste yearly, with an average per capita waste generation of 6 tonnes (Eurostat 2010). The main types of waste streams vary across European countries that are mainly due to the economic structure of each country. For example the existence of a large mining sector in Luxembourg, Romania, Estonia, Finland and Sweden increases national averages due production of mineral waste (Eurostat 2010).

With overall waste production, also food wastage is increasing. It is estimated that 89 million tonnes of food waste is produced annually in the European Union. This amount at the current rate will increase 40% by 2020 if no measures are taken (European Parliament 2012). In Finland citizens (ca. 5.4 million), the food service sector, the retail sector and the food industry together waste up to 335-460 million kg of food, i.e. 62-85 kg per Finnish citizen every year (Silvennoinen et al. 2012). Food wastage has negative environmental and economic impacts. It is estimated that climate impacts of the food discarded annually results in high carbon dioxide (CO₂) emissions, equal to emissions of 100 000 cars. When considering the economic perspective of food wastage, average household uses €4 300 for purchasing food, of which the value of discarded foods is €220 annually. (Silvennoinen et al. 2012)

Waste collected by municipal authorities includes all the waste collected and disposed through the municipal waste management system. Municipal solid waste consists of waste generated by households and other wastes that are similar in nature and composition. Wastes are collected and managed by or on behalf of municipal authorities. Municipal waste stream is from households, though similar wastes from sources such as commerce, offices and public institutions are also included. In addition, municipal wastes include many different types of materials including paper, plastics, food, glass and household appliances. (Eurostat 2010)

Simplicity and financial reasons have made disposing solid wastes a common practice on sanitary landfills for many decades. The main negative impacts of landfilling of energy-rich organic waste are not only its health and environmental impacts, but also low recovery

of resources (European Commission 2013). Landfilling takes up valuable land space, and also causes air, water and soil pollution. Landfills discharge CO₂ and methane (CH₄) into the atmosphere. Earth and groundwater are exposed to leachate that may contain harmful contaminants, such as chemicals and pesticides (European Commission 2013). This in turn is harmful to human health, as well as to plants and animals. European Landfill Directive has obligated EU member states to develop more sustainable waste management practices that include collection, pretreatment and post-treatment methods. It has been shown that reduced landfilling in favor of increased recycling of energy and materials leads to lower environmental impact, lower consumption of energy resources, and lower economic costs (Eriksson et al. 2005).

Most commonly applied biological and mechanical-biological treatment methods for source-separated biodegradable wastes include aerobic composting and anaerobic digestion. These methods work as sustainable alternatives for landfilling. In composting air or oxygen is used to support metabolism of the aerobic micro-organisms degrading the substrate, whereas anaerobic digestion operates without free oxygen and results in fuel gas called biogas (Demirbas 2009). Compared to composting, anaerobic digestion has several advantages that include better handling of wet waste, less odor emissions and green house gases, nutrient recycling and the possibility of energy recovery in the form of CH₄ (Demirbas 2009, Valorgas 2010).

Anaerobic digestion has been proven to be an efficient and green technology for treatment of residual sludges and wastes, such as sewage sludges, crop residues, food waste and animal manure (Al-Seadi et al. 2008, Banks et al. 2011). Food waste is an attractive feedstock for anaerobic digestion as it has high methane potential, due to its composition with lipids and proteins (Banks et al. 2008, 2011). Proteins and lipids are reported to contribute significantly to methane production and anaerobic digestion of food wastes can achieve methane yield up to 450 m³ per one kilo of organic dry matter (Zhang R. et al. 2007, Banks et al. 2008, Forster-Carneiro et al. 2008, Li, C. et al. 2011, Pecorini et al. 2012, Zhang C. et al. 2013). On the other hand, problems related to anaerobic digestion of food wastes are the ammonia (NH₃) and volatile fatty acids (VFA) that are formed during degradation of proteins and lipids and that are inhibitory for microbial activity in high concentrations (Banks et al. 2008, 2010). It is also generally acknowledged that thermophilic process temperature (55 °C) results in larger degree of imbalance and higher

risk for ammonia inhibition than mesophilic process temperatures (35 °C) (Mata-Alvarez 2003, Banks et al. 2008, Demirbas 2009, Banks et al. 2010). Therefore thermophilic processes are more sensitive to temperature fluctuations and micro-organisms require longer time to adapt to a new temperature. Food waste feedstocks are also reported to perform more stable under co-digestion compared to mono-digestion due enhanced buffer capacity enabled by co-substrate such as cattle manure or sewage sludge (Marañón et al. 2012; Zhang, C. et al. 2013).

Present study focuses on anaerobic digestion of institutional food waste and kitchen waste under thermophilic (55 °C) conditions. Quantities, composition and chemical characteristics of waste fractions produced at restaurant Ylistö were determined and methane potential of food waste and kitchen waste were determined by batch experiments. In addition, process performance and methane yields of studied feedstocks were studied in anaerobic reactors over period of 210 days at 55 °C.

1.1 Background of the study

1.1.1 Food wastes for renewable energy production

Food that ends up as waste causes environmental impacts during its life cycle; from primary production and processing, to food manufacture and storage. Additionally food wastes may have to be transported long distances to treatment facilities. As a whole these impacts can be considered unnecessary, in case production of waste can be avoided. Discarded foods also cause the producer unnecessary costs.

The European Union has set waste prevention as the primary target of its waste management and in Waste Directive, 'prevention' is referred as "activities that are carried out before the end-product ends up as waste and that reduces the quantity of waste, their harmful effects and quantity of harmful contaminants". For food supplies such measures may include extension of life span by ensuring correct handling and storage conditions, careful design and dimensioning of food supply procurement. If discrediting is unavoidable, the produced wastes should be utilized as efficiently as possible. Waste Directive defines 'waste' as "any substance or object which the holder discards or intends or is required to discard". Moreover, 'biowastes' are defined as "biodegradable garden and park waste, food and kitchen waste from households, restaurants, retail premises and comparable waste from food processing plants".

The Waste Directive introduces a five step waste hierarchy where prevention is primary option for waste utilization and is followed by re-use, recycling and other forms of recovery of waste. Disposal, such as landfill, is considered as the last resort of waste. Member states should take measures for reduction of biodegradable waste that is deposited on landfills. Separating the municipal waste into source sorted organic fraction, recyclable fraction and residual waste and is a general practice of waste management adopted by member states to meet the requirements of the Directive. The Landfill Directive obliges Member States to reduce the amount of biodegradable municipal waste that they landfill to 35% of 1995 levels by 2016.

Food wastes can be separated into edible and inedible fraction. Edible food wastes are food materials that have initially at time of disposal been edible and inedible food wastes are parts of food materials that cannot be eaten, such as peels, guts and parings, bones and coffee grounds. In addition, contaminants such as paper and card, garden waste, plastic bags and containers, metals, and glass may end up in food waste stream. Compositional analyses of food wastes performed in Finland have shown that in general fruit and vegetable waste make the largest waste fraction from 25% to 45% (Heaven et al. 2010, Silvennoinen et al. 2012). These are followed by other discarded foods, e.g. home cooked food, milk products, breads etc. In addition, biodegradable wastes have been found to contain also contaminants or misplaced wastes; such as garden waste, paper and card, plastic containers, plastic bags, metals, glass and other miscellaneous wastes such as pet litter and textiles (Heaven et al. 2012).

Comparison of food waste compositional analysis in certain geographically distinct regions of Europe is presented in Table 1. Fruit and vegetable waste is the largest waste fraction in all countries (44.5-69%). In Finland drinks (27.5%), that consisted mainly coffee grounds, represent notably larger fraction compared to other countries (< 10%). Pasta, rice, flour and cereals are represented higher in Italy compared to other countries (12.4%). Despite some variation in the waste compositions, the values for key analytical parameters show higher degree of similarity. According to Heaven et al. (2010), it is considered that even food preferences and cuisine may vary from one country to another; the fundamental requirements of human diet and therefore the composition of domestic food waste are likely to remain similar.

Table 1 Comparison of results of food waste compositional analysis for samples from UK, Finland, Portugal and Italy (Heaven et al. 2010). Standard deviations are shown in parenthesis.

% (w/w)	UK	Finland	Portugal	Italy	Average
Fruit and vegetable waste	60.9	44.5	59.2	69	58.4 (10.2)
Pasta/rice/flour/cereals	1.5	0.4	0.2	12.4	3.6 (5.9)
Bread and bakery	9	3.8	3.1	2.8	4.7 (2.9)
Meat and fish	6.7	4.3	7.3	6.2	6.1 (1.3)
Dairy	1.7	2	0.7	1.4	1.4 (0.56)
Drinks	7.1	27.5	0.2	0	8.7 (13)
Confectionery. snacks etc.	0.7	3.2	0.3	0	1 (1.5)
Mixed meals	12.3	6.3	29	1.4	12.2 (12)
Other food	0.2	8	0	6.9	3.8 (4.2)
Total	100	100	100	100	100

Comparison of waste fractions produced in UK, Finland, Portugal and Italy is presented in Figure 1. Fruit and vegetable waste show largest fraction in the food waste fraction with 58.4% followed by mixed meals with 12.2%. Other waste fractions presented are below 10% of total food waste.

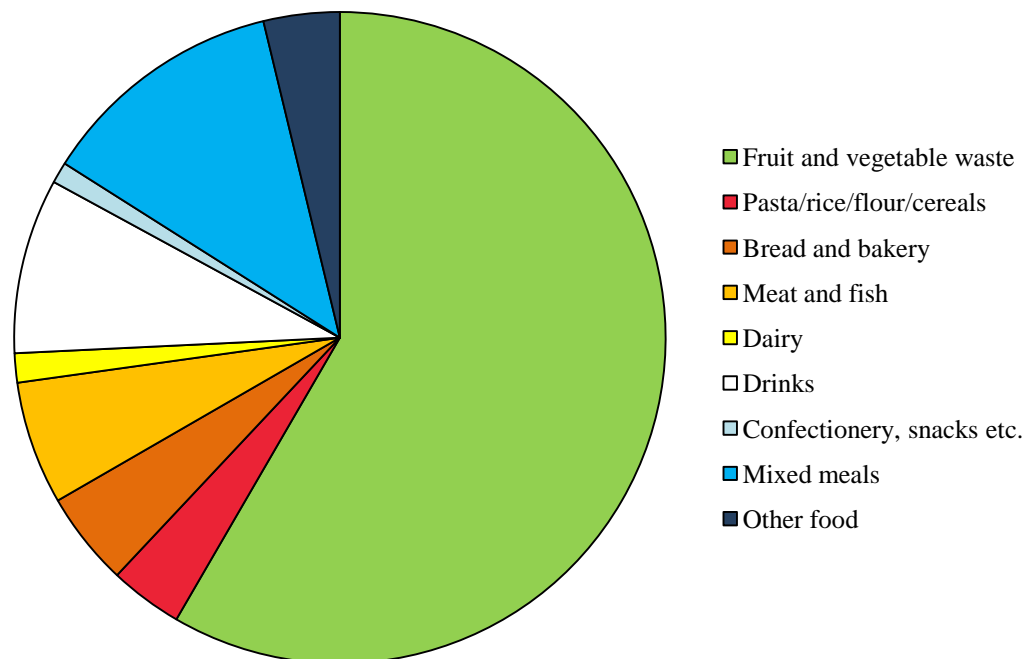


Figure 1 Comparison of results of food waste compositional analysis for samples from UK, Finland, Portugal and Italy (Heaven et al. 2010).

In the same study by Heaven et al. (2010) also the chemical composition of wastes was studied. Even the general composition of food wastes may vary between countries, the

chemical composition of food wastes remain more similar, where pH ranges 4.71-6.16, total solids (TS) 23.70-33.80%, volatile solids (VS) 20.16-26.83% and Total Kjeldahl Nitrogen (TKN) 6.45-8.12 g kg⁻¹ (w/w). Furthermore, the elemental analysis showed that food waste has nitrogen (N) 2.46-3.42 (%TS), carbon (C) 47.2-51.3 (%TS) hydrogen (H) 5.53-6.67 (%TS) sulphur (S) 0.15-0.23 (%TS) and oxygen (O) 29.3-34.7 (%TS). Similar chemical characteristics of food wastes have been reported in food waste digestion studies by Banks et al. (2008); Zhang, R. et al. (2007) and Forster-Carneiro et al. (2008). Food wastes that are produced in institutions and institutional restaurants generally have similar composition as the food wastes described above, with minor exceptions (Climenhaga et al. 2010, Zhang, C. et al. 2013). In a study by Climenhaga et al. (2010) that was conducted in the University of Southampton, it was estimated that over the course of 30 week academic year at average waste generation rate, food waste generation would be equal to 28 kg per student. The above generation rate was lower compared to the food waste produced in households i.e. 62-85 kg per Finnish citizen (Silvennoinen et al. 2012).

Present study focuses on institutional food wastes produced in university restaurant that includes the edible fraction (i.e. discarded food), and also inedible (i.e. peels and bones) fraction of food wastes. Large fraction of these food wastes include the paper napkins and other card or food packaging content, that are produced during eating and thrown to same biowaste bin as leftover food.

1.1.2 Anaerobic digestion of food wastes

Food wastes can be utilized in anaerobic digestion process that is a fermentation process where organic matter is degraded by micro-organisms under anaerobic conditions and results in the production of biogas. Biogas contains mostly 50-70% of CH₄ and 30-40% of CO₂, but is also carrying impurities such as moisture, hydrogen sulfide (H₂S) and particulate matter (Mata-Alvarez 2003, Al-Seadi et al. 2008, Weiland 2009). After appropriate gas clean-up, CH₄ can be used for heat and power production in combined heat and power plants, to produce heat in boilers, injected in natural gas grid or used as a vehicle fuel (Demirbas 2009, Kaparaju & Vijay 2010). The process effluent; i.e. leachate or digestate, can be used as fertilizer due high amounts of concentrated nutrients. Use of digestate as fertilizer reduces the need of chemical fertilizers. Hygienisation of process digestate is required prior to use as fertilizer (The Finnish Act on Fertilizer Products 539/2006).

1.1.3 Biochemistry of anaerobic digestion

Methane fermentation process can be divided into four phases that are hydrolysis, acidogenesis, acetogenesis and methanogenesis (Figure 2). These steps are carried out by a rather complex group of micro-organisms, but can be generalized as hydrolytic bacteria, acidogenic bacteria, acetogenic bacteria and two groups of methanogenic bacteria; hydrogenotrophic methanogens and acetoclastic methanogens (Al-Seadi et al. 2008, Demirbas 2009, Banks et al. 2010). Process is based on a close microbial association between acid-producing bacteria, acid-degrading bacteria and methanogens (Banks et al. 2010).

During hydrolysis, hydrolytic bacteria initially break polymers (i.e. proteins, carbohydrates and lipids) into smaller units (Al-Seadi et al. 2008). Proteins are mainly broken into amino acids, carbohydrates into sugars and lipids into long-chain fatty acids (LCFA). Both solubilization of insoluble particulate matter and biological decomposition take place (Demirbas 2009).

The hydrolysis products are further decomposed by the micro-organisms involved and used for their own metabolic processes. During acidogenesis, the products of hydrolysis are converted by acidogenic bacteria into methanogenic products. Simple sugars, amino acids and LCFAs are degraded into acetate, CO₂ and hydrogen (H₂), as well as into volatile fatty acids (VFA) and alcohols (Al-Seadi et al. 2008). The products of acidogenesis that cannot be directly converted into methane by methanogenic bacteria are further digested by acetogenic bacteria in acetogenesis. VFAs and alcohols are oxidized into methanogenic substrates such as CO₂, H₂ and acetic acid (Al-Seadi et al. 2008). H₂ increases the hydrogen partial pressure and inhibits metabolism of acetogenic bacteria (Al-Seadi et al. 2008).

Only a limited number of compounds can act as substrates in methanogenesis. Methanogenic bacteria produce methane from intermediate products; acetate, H₂ and CO₂, where 70 % of CH₄ is originating from acetate and remaining 30 % is produced from conversion of H₂ and CO₂ (Al-Seadi et al. 2008). Methanogenesis is the slowest biochemical reaction of the digestion process and for that reason also the most critical (Al-Seadi et al. 2008).

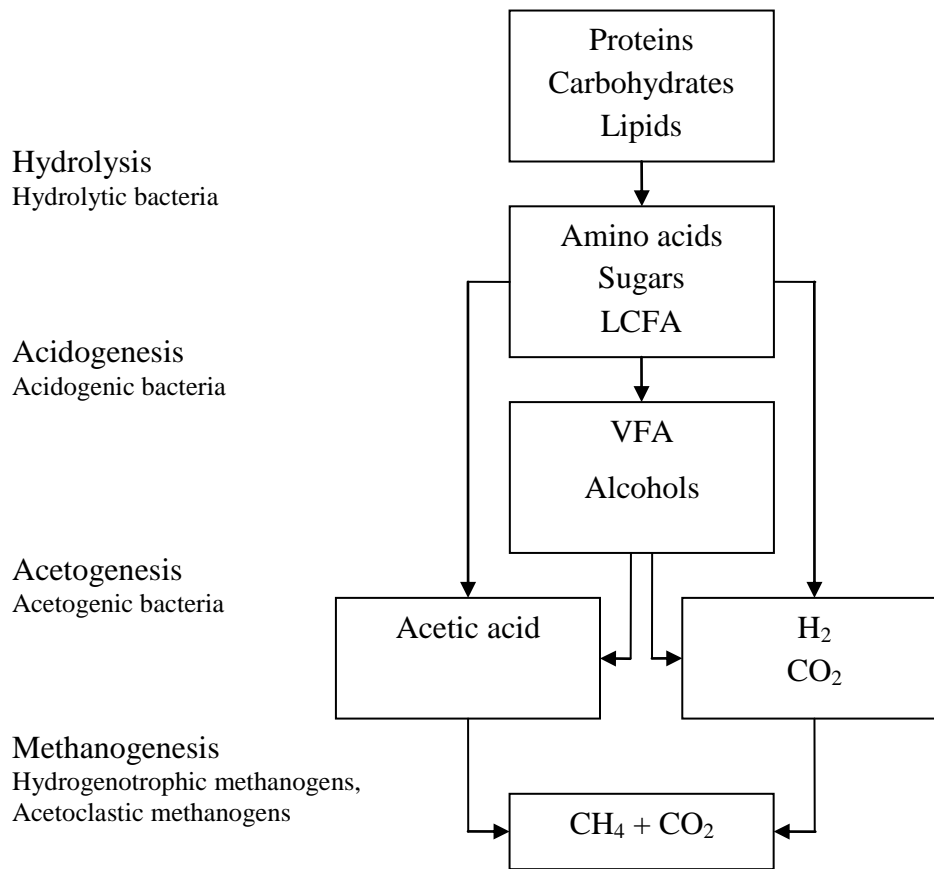


Figure 2 Conversion processes in anaerobic digestion (Redrawn according to Al-Seadi et al. 2008, Demirbas 2009 and Weiland 2009).

1.1.4 Factors affecting anaerobic digestion of food waste

Finding optimal control parameters is essential for the feasibility of anaerobic digestion process. Generally control parameters are selected based on how the highest possible methane yield can be achieved. Main operational parameters in process are pH, temperature, organic loading rate (OLR) and hydraulic retention time (HRT). Wide range of technologies can be applied, continuously stirred tank reactor (CSTR) being most commonly applied due to simplicity and financial reasons (Demirbas 2009, Weiland 2009).

Methane formation takes place within a relatively narrow pH interval, 6.5 to 8.5 while an optimum interval is 7 to 8. The process is severely inhibited if the pH decreases below 6 or rises above 8.5 (Weiland 2009).

When dimensioning the biogas digester, HRT should be taken into account, since the retention time is the average time interval (e.g. d) the substrate is kept inside the digester (Al-

Seadi et al. 2008). It is correlated to the digester volume (e.g. L, m³) and the volume of substrate fed per time unit (e.g. L d⁻¹).

OLR indicates how much organic dry matter can be fed into the digester per volume and time unit (Al-Seadi et al. 2008). Loading rate of volatile solids (VS) is commonly applied when determining loading rate (e.g. gVS L⁻¹ d⁻¹). In order to obtain the maximum biogas yield by complete digestion of the substrate and VS, longer retention time of the substrate inside the digester would be required and correspondingly larger digester size. As increasing the OLR increases the HRT, the retention time must be long enough to ensure that amount of micro-organisms removed with the effluent is not higher than the amount of reproduced micro-organisms. The duplication rate of anaerobic bacteria is usually 10 days or more (Al-Seadi et al. 2008). Shorter HRT provides good substrate flow rate, but also a lower biogas yield. Therefore it is important to adapt the HRT to the specific decomposition rate of the used substrates (Al-Seadi et al. 2008). Digesters operating on food wastes usually have HRT of 20-30 days (Banks et al. 2007; Zhang R. et al. 2007, Ghanimeh et al. 2012).

Anaerobic digestion is known to occur over a wide range of temperatures, from 10 °C to 71 °C. Mesophilic temperatures range is 35-42 °C and thermophilic temperature range 55-60 °C and both are most commonly applied (Al-Seadi et al. 2008, Demirbas 2009). Methanogenic micro-organisms grow at higher rate in thermophilic process (Weiland 2009). Operation at thermophilic temperature allows shorter HRT and a higher biogas production rate, making the process faster and more efficient than in mesophilic temperatures (Banks et al. 2008). For this reason thermophilic digester can be loaded with higher OLR or with lower HRT than mesophilic digesters. On the other hand, too high OLR causes accumulation of intermediate products and inhibition of the process, while low OLR give low methane yield and small amount of feedstock being treated at the same time (Mata-Alvarez 2003, Al-Seadi et al. 2008).

Food waste is energy-rich substrate but there are problems associated to its composition regarding anaerobic digestion. Main substances that can cause inhibition in digestion of food wastes are NH₃ and VFAs. Other toxic compounds in this environment are rare if source separation of wastes is carried out (Mata-Alvarez 2003). The high protein content in substrate usually gives out high nitrogen content during hydrolysis, while high nitrogen content usually generates elevated NH₃ concentrations in process especially during extend-

ed run times (Banks et al. 2008, 2010). NH_3 is an important compound and has significant function in the digestion process, as it is one of the most common reasons for the inhibition (Banks et al. 2008). Ammonium nitrogen ($\text{NH}_4\text{-N}$) in the process can exist in form of ammonium (NH_4^+) or free ammonia (NH_3) depending on environmental conditions (Banks et al. 2010). The concentration of NH_3 is directly proportional to the temperature and there is an increased risk of ammonia inhibition at thermophilic temperatures compared to mesophilic ones (Al-Seadi et al. 2008). There is no clear consensus over threshold levels for inhibition, but according to Mata-Alvarez (2003), inhibition occurs at total NH_3 concentrations of 1.2 g L^{-1} and above. In anaerobic digestion of organic fraction of municipal solid waste 50% inhibition of methane production has been observed at NH_3 concentrations of 215 and 468 mg L^{-1} in mesophilic and thermophilic digestion, respectively (Benabdallah El Hadj et al. 2009). Similarly, the methane generation under mesophilic and thermophilic conditions was reduced by 50% when NH_4^+ reached concentrations of 3860 and 5600 mg L^{-1} , respectively. High NH_3 concentrations are often associated high concentrations of VFA. NH_3 is considered to provide alkalinity through the formation of ammonium carbonate that helps to buffer the system allowing operation under these conditions (Banks et al. 2008). There is no clear consensus on threshold values of inhibitory levels of NH_3 or VFA concentrations and some reactors may perform well even under high concentrations (Banks et al. 2010).

VFA concentration is also important indicator, as increase in concentration causes decrease in pH. Elevated concentrations are characteristics of instability and indicate usually a developing problem. VFAs are intermediary compounds of the anaerobic degradation of organic matter and especially the undissociated species have been reported as more toxic as they can more easily diffuse to the inner parts of the micro-organism cell and among VFAs, propionic and butyric acids have been described as the most harmful (Mata-Alvarez 2003). In digestion of food wastes, a characteristic pattern of fatty acid production and accumulation has been observed on at different scales of operation (Banks et al. 2008). During the ammonia inhibition, increase in the VFA concentration will lead to a decrease in pH which partly counteracts with the effect of NH_3 . The accumulation of VFAs will often not always result in a pH drop, due to the buffer capacity produced by NH_3 . Many of the bacteria involved in anaerobic digestion process are pH sensitive and most intermediate products from different steps of the process can work as inhibitors (Weiland 2009). Acidogens, having better tolerance to acidity, may produce acids faster than increasingly

inhibited methanogens can consume. In conditions of low pH, this results in collapse of the whole process. Nevertheless, the actual cause may be less immediately obvious than mentioned above. Other factors may also foster increase in VFA concentration, such as inadequate mixing (Ghanimeh et al. 2012), excessive loading or poor temperature control.

Banks et al. (2008) assessed a pilot scale study for comparison of mesophilic and thermophilic mono-digestion of source segregated domestic food waste. The mesophilic reactor was initially fed at OLR $3.5 \text{ gVS L}^{-1} \text{ d}^{-1}$ and rate was gradually increased. It was found out that any increase above $4 \text{ gVS L}^{-1} \text{ d}^{-1}$ resulted in an increase in VFA and at OLR above $4.5 \text{ gVS L}^{-1} \text{ d}^{-1}$ and no additional biogas was produced. Digestion under thermophilic conditions at OLR $4 \text{ gVS L}^{-1} \text{ d}^{-1}$ showed more efficient process and also enhanced methane yields, but required a reduced loading to be applied due very high VFA levels. After OLR was reduced to $1 \text{ gVS L}^{-1} \text{ d}^{-1}$ and gradually increased back to $3 \text{ gVS L}^{-1} \text{ d}^{-1}$ the VFA levels stabilized. The mesophilic digester performed stable under OLR $4 \text{ gVS L}^{-1} \text{ d}^{-1}$ while loading for the thermophilic digester from 3.7 to over $5 \text{ gVS L}^{-1} \text{ d}^{-1}$ was not considered sustainable. The specific methane yield in the mesophilic digester was 390 mL gVS^{-1} and in the thermophilic digester 410 mL gVS^{-1} . Maximum biogas yields in the thermophilic digester were higher than in the mesophilic reactor, but the VFA levels in the mesophilic digester were more stable. Maximum VFA levels recorded in thermophilic digester were 44.6 g L^{-1} (range $14.1\text{-}44.6 \text{ g L}^{-1}$) and in mesophilic digester 28.3 mg L^{-1} (range $6.8\text{-}28.3 \text{ g L}^{-1}$). Mesophilic reactor found high ammonia concentration of around 5.2 g L^{-1} that was considered to increase the alkalinity of the system. This high alkalinity was considered to be sufficient to buffer the VFA resulting in a stabilized pH that in both reactors remained within narrow limits despite fluctuations in VFA concentrations. For this reason, pH proved to be a poor indicator of digester instability due to the delay in reaction time.

For growth and survival of specific groups of micro-organisms, several macro- and micro-nutrients are necessary. Macronutrients are carbon (C), phosphor (P) and sulfur (S). The need of macronutrients is low due to the fact that not much biomass is developed in the digestion process. Nutrient ratio C/N 20-30 is considered to be sufficient in anaerobic digestion (Li Y. et al. 2011). In the study by Banks et al. (2008) for mesophilic and thermophilic digestion of food wastes C/N ratio was 14. Similar values for food wastes are

reported in literature (Zhang R. et al. 2007, Heaven et al. 2010, Zhang C. et al. 2013). The optimal C/N ratio varies with the type of feedstock to be digested.

Micronutrients (i.e. trace nutrients), such as iron (Fe), manganese (Mn), copper (Cu), chlorine (Cl), molybdenum (Mo), zinc (Zn) and tungsten (W) are important for the growth rate of micro-organisms and maximum OLR can significantly be enhanced by trace nutrient addition (Banks et al. 2010). Especially selenium (Se) and cobalt (Co) has showed to be essential in order to prevent certain species (i.e. propionic acid) of VFA from accumulating. (Banks et al. 2012)

1.1.5 Improving the anaerobic digestion of food waste

Studies have shown that food wastes have high recoverable energy content. There are many examples of anaerobic digestion for treatment of organic municipal solid wastes. Especially co-digestion with sewage sludges or cattle manure has been widely applied due enhanced buffer capacity of the digestion process (Marañón et al. 2012; Zhang, C. et al. 2013). There are only few studies that have focused on the thermophilic mono-digestion of food wastes, let alone food waste arising from institutional sources, due to observed instability issues. The process characteristics play a major role in anaerobic digestion, but also the nature of organic substrate has an important influence on the biodegradation process and methane yield. For mixed food wastes, methane production potentials in mesophilic and thermophilic temperatures from 180 mL gVS⁻¹ up to even 484 mL gVS⁻¹ are reported in literature (Foster-Carneiro et al. 2008; Li, C. et al. 2011, Pecorini et al. 2012; Zhang, C. et al. 2013).

Zhang, C. et al. (2013) studied anaerobic co-digestion of cattle manure and food waste. It was considered that separate mono-digestion of food waste or cattle manure was hardly feasible, and addition of cattle manure was noticed to enhance buffer capacity of the process. Mono-digestion of food waste in mesophilic temperature with OLR 8 gVS L⁻¹ d⁻¹ gave methane yield of 347 mL gVS⁻¹. Increasing OLR resulted in decreased methane production and instability issues. Co-digestion with cattle manure resulted in enhanced methane production and also use of higher OLR was possible. Similarly, Marañón et al. (2012) assessed reactor performance under both mesophilic and thermophilic conditions with co-digestion of cattle manure, food waste and sewage sludge. Maximum methane yield was obtained with co-digestion of cattle manure, food waste and sewage sludge (in ratio of 70:20:10). Methane yield was 603 mL gVS⁻¹ at 36 °C, for an OLR 1.2 gVS L⁻¹ d⁻¹.

With same feed mixture in thermophilic conditions, yield was 440 mL gVS⁻¹. Also higher NH₃ and acidity values were found in thermophilic conditions.

Banks et al. (2012) investigated why anaerobic digesters treating food waste and operating at high NH₃ concentrations suffer from VFA accumulation, and especially propionic acid accumulation, which may result in process failure. The semi-continuous reactors were fed on source segregated food waste in mesophilic process conditions. The results showed deficiency of selenium that is essential for both propionate oxidation and syntrophic hydrogenotrophic methanogenesis and leads to process failure while supplementation allows operation at substantially higher OLR. Critical selenium and cobalt concentrations were established as 0.16 and 0.22 mg kg⁻¹ for fresh matter feed at moderate loading. At this dosage OLR could be raised and thus having increased biogas yields. Results were considered to represent a significant increase in process performance and operational stability. Thermophilic process does not respond to trace nutrient supplementation similarly compared to mesophilic process, although Uemura (2010) and Takashima et al. (2011) suggested that thermophilic digestion requires more trace nutrients than mesophilic digestion.

2 MATERIALS & METHODS

2.1 Source of food waste and kitchen waste

The feedstocks used for batch and reactor experiments were institutional food wastes and kitchen wastes. Both materials were collected from restaurant Ylistö at University of Jyväskylä (24.9.-19.10.2012). Another batch of food waste and kitchen waste was collected (3.6.-14.6.2013) for reactor experiments.

Sonaatti Ltd founded in 1997, serves as the company offering restaurant, cafeteria and catering services. It is joint enterprise of University of Jyväskylä, The Student Union of University of Jyväskylä (JYY) and Fazer Food Services Ltd. In total, Sonaatti Ltd has 12 service premises: 6 are restaurants and 6 are cafeterias (Sonaatti, 2013a). Of the 12 premises, 10 premises serve lunch; restaurants only prepare food (i.e. produce kitchen waste). Overall number of customers in Sonaatti restaurants (10) in the year 2012 was 670 824 (Sonaatti, 2013b). Restaurant Ylistö, that serves food throughout year, is located at Ylistönrinne campus of University of Jyväskylä. Monthly and overall customers in restaurant Ylistö are presented in Table 2. Generally summer months and December are quieter than fall and spring semesters, where average number of customers during June till August was ca. 7 000 and rest of the academic year ca. 12 000.

Table 2 Restaurant Ylistö lunch customers 2012 (Sonaatti, 2013a).

Month	Customers
January	11 520
February	12 434
March	12 108
April	10 002
May	10 792
June	7 407
July	6 132
August	7 742
September	12 519
October	14 764
November	14 550
December	7 159
Overall	127 129

Food wastes were produced by restaurant customers. In addition to leftover food waste, wastes also include paper napkins and sometimes other misplaced non-degradable waste (plastics, paper cups, cutlery etc.). All misplaced and non-degradable materials were removed before pretreatment. Kitchen wastes are wastes that are produced during the food cooking process. Wastes include vegetable wastes, perishable foods and desserts from previous days or week.

Lunch hours in the restaurant are 10:30-14:30. Wastes for present study were collected on weekdays, between 11:30-13:00 into garbage bags and taken into laboratory for further processing. Before sampling waste bins were weighed. After weighing, bins were mixed and grab samples were collected. Daily sample size of both kitchen wastes and food wastes were between 2-3 kg. After the sample collection, composition of food waste and kitchen waste was determined (Table 5 in the results section).

Furthermore, the number of customers was monitored daily. Customer numbers were acquired from restaurant customer register. Customers that at the time had not finished their meal (i.e. not returned the leftover food and trash) were excluded from the number of total customers. Consequently, the amount of waste produced by a single customer could be calculated based on the amount of waste generated at the restaurant (Table 5).

In the laboratory, the collected waste materials were grinded with a meat grinder (Talsa W22) into 5 mm particle size. The feedstocks before and after pretreatment are shown in Pictures 1 and 2. After daily grinding, both kitchen waste and food waste feedstock fractions were stored in plastic bags (Pirkka 1 L frozen-food-bags, HD-polyethene) and frozen at -20 °C for further use.



Picture 1 Food waste (right) and kitchen waste (left) collected from the Restaurant Ylistö, University of Jyväskylä (Photo: Jari Koponen).



Picture 2 Grinded waste feedstock materials (Talsa W22). Food waste (right) and kitchen waste (left) (Photo: Jari Koponen).

After the waste collection period, both collected kitchen waste and food waste feedstock daily portions were unfrozen and combined into two separate feedstock waste fractions, in order to get a descriptive monthly average well homogenized feedstock (Picture 3). Furthermore, chemical characteristics of food waste and kitchen waste determined. Characteristics of both food waste and kitchen waste are presented in Table 6 at the results section.



Picture 3 Food waste feedstock (right) and kitchen waste feedstock (left) after mixing the daily fractions. (Photo: Jari Koponen)

The ratio of food waste and kitchen waste produced was determined by the actual amounts that they were produced. For food waste, the waste amount was calculated by using the average number of customers visited the restaurant during in September and October 2012 i.e. 13642 (Table 2). The average number of customers per month was obtained by dividing the total number of business days (20). Thus, the mean number of customers per day in September and October was 682. The amount of food waste produced by a single customer per day was calculated from the monitored waste amount and the number of customers. Amount of food waste produced per customer was calculated to be 74.3 g d^{-1} . This results to overall 50.7 kg d^{-1} of food waste generated by all customers per day. Kitchen waste was considered to be produced at fixed rate, since the majority of food preparatory wastes were already produced by the time of waste monitoring commenced i.e. on average 11.6 kg d^{-1} . Based on these, the calculated ratio of food waste to kitchen waste was 4.4:1 (further rounded up to 4.5:1) and is further used in both batch and reactor experiments.

2.2 Source of inoculum

For batch and reactor experiments, digested material from a thermophilic digestion plant (Stormossen) treating both organic fraction of municipal solid waste and sewage sludge was used as inoculum. Inoculum was stored in canisters at $4 \text{ }^{\circ}\text{C}$ prior to use. In order to reactivate microorganisms and reduce background methane production, inoculum was incubated at $55 \text{ }^{\circ}\text{C}$ for 1 week. Characteristics of inoculum are also shown in Table 6 (results section).

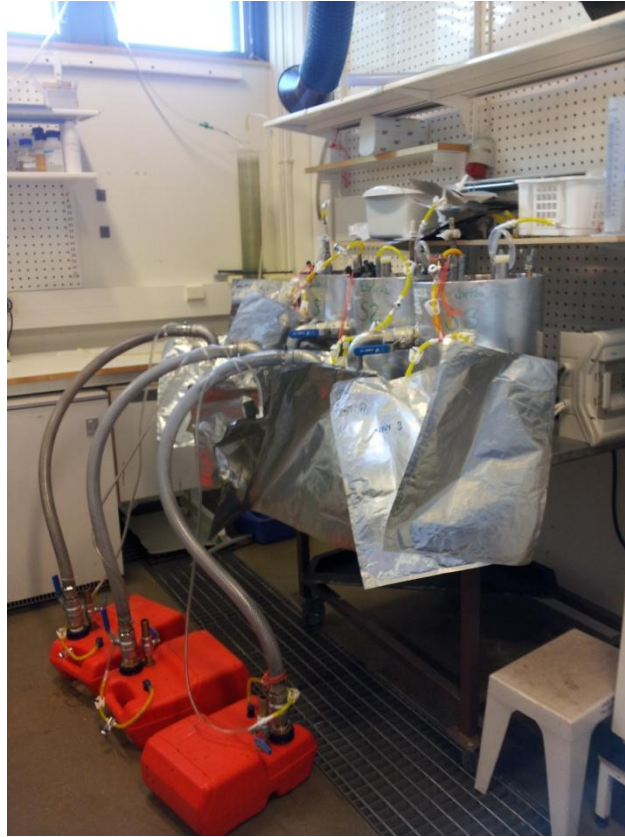
2.3 Batch experiment

Methane production potentials of food waste and kitchen waste were determined in batch experiment at 55 °C. Triplicate sets of batch assays were prepared for control (inoculum), kitchen waste and food waste. Experiment was conducted in 1000 mL glass bottles with working volume of 700 mL. To each assay, substrate (4 gVS bottle⁻¹) and inoculum (400 mL) were added to achieve a substrate to inoculum VS ratio of 0.5.

Each bottle was filled up to 750 mL with tap water. Bottles were closed with butyl rubber seals and air was removed flushing the bottles with N₂/CO₂ –gas mixture (70% N₂ and 30% CO₂). The produced gas was collected in aluminum bags. Experiment was conducted in triplicates. Methane production potential of inocula was used as a control and was subtracted from those of the sample assays.

2.4 Reactor experiment

The effect of organic loading rate on the process performance and methane yields from food waste and kitchen waste alone and as co-digestion of both substrates was studied at 55 °C. Three similar stainless steel continuously stirred tank reactors (CSTR) were operated with a working volume of 10 L (Picture 4). Reactor 1 was fed with food waste (R1), Reactor 2 was fed with kitchen waste (R2) and Reactor 3 was fed with food waste and kitchen waste (R3) at a ratio of 4.5:1 (w/w).



Picture 4 Reactor experimental set-up.

During the start-up, each reactor (R1, R2 and R3) was filled with 10 L of inoculum. An initial feed of 27.3 g of food waste for R1, 48.4 g of kitchen waste for R2 and 24.2 g of food waste and 5.5 g of kitchen waste (ratio 4.5:1 w/w) R3 were loaded.

Methane concentration in biogas was awaited to reach 50% before daily feeding was started. This took 5 days with exception of R3 as it had a leak in reactor lid that was noticed due long-lasting start-up time. Leak was fixed after 5 days and feeding with half feedstock amount ($0.38 \text{ gVS L}^{-1} \text{ d}^{-1}$) was used until 50% CH_4 -concentration was reached in 15 days.

During the experiment 6 different OLRs were tested and were divided as 6 different loading periods. Operation lasted for R1 and R3 for 207 days and for R2 for 210 days. After the initial start-up, reactors were operated at an OLR of $0.75 \text{ gVS L}^{-1} \text{ d}^{-1}$ with HRT of 50 d (days 1-50). OLR was increased in a step-wise manner from $1.5 \text{ gVS L}^{-1} \text{ d}^{-1}$ to $6 \text{ gVS L}^{-1} \text{ d}^{-1}$. It should be noted that after the first OLR of $0.75 \text{ gVS L}^{-1} \text{ d}^{-1}$, HRT was reduced from 50 to 30 d. For remaining experimental period, the HRT was between 27-31 days. During days 174-179, feeding was withheld for 1 week for all three reactors due to process instability. Feeding was resumed on day 180 with same OLR of $6 \text{ gVS L}^{-1} \text{ d}^{-1}$ in R1 and R3

while the OLR of R2 was reduced to $4.5 \text{ gVS L}^{-1} \text{ d}^{-1}$. Same volume of effluent was removed as feed was added daily, up to day 113 (loading period 4), as reactors were opened and significant water vaporization was noticed. After day 113 only samples for chemical analyzes were taken.

Feed was prepared once a week by thawing the frozen materials. Prepared feed was stored at $4 \text{ }^{\circ}\text{C}$ for further use (Picture 3). On each day, reactors were fed with 333.3 ml of feed to attain a HRT of 30 days. At the same time, an equal amount of effluent was removed by means of over-flow from the reactor. Feed was diluted with tap water to attain feed rate. Reactors were fed on weekdays only (Monday through Friday). Starting from day 50 (OLR $1.5 \text{ gVS L}^{-1} \text{ d}^{-1}$) onwards once a week, feed was supplemented with trace nutrients at the rate of 140, 245 and 150 μl (Banks et al. 2012). For further loading periods with higher OLR, trace nutrient supplementation was correlated with the amount of fresh substrate fed. Supplementary trace nutrient concentrations are shown below in Table 3. The amount of trace nutrient supplementation for the remaining loading periods was calculated based on substrate loading rate (w/w).

Table 3 Trace nutrient concentrations in trace nutrient supplement solution.

Element	Compound used	Element concentration (mg L^{-1})
Aluminium (Al)	$\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$	0.1
Boron (B)	H_3BO_3	0.1
Cobalt (Co)	$\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	1
Copper (Cu)	$\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$	0.1
Iron (Fe)	$\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$	5
Manganese (Mn)	$\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$	1
Molybdenum (Mo)	$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$	0.2
Nickel (Ni)	$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	1
Selenium (Se)	Na_2SeO_3	0.2
Tungsten (W)	$\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$	0.2
Zinc (Zn)	ZnCl_2	0.2

2.5 Chemical analyzes and calculations

pH was measured immediately after sampling using pH meter (Mettler Toledo Seven Easy or Methrohm 774). Meters were calibrated before each experiment. TS and VS were analyzed according to standard SFS 3008 (Finnish Standard Association 1990). $\text{NH}_4\text{-N}$ and TKN were analyzed by using Kjeltac 1002 distillation unit (Tecator AB) and according to protocol published elsewhere (Perstop Analytical/ Tecator AB,13/10/99/SM). Soluble

chemical oxygen demand (SCOD) was analyzed according to Finnish Standards SFS 5504 (Finnish Standard Association, 2002).

Biogas volume was determined by water-displacement method (column volume 10 L). The methane concentration in the biogas was determined by using gas chromatograph (Perkin-Elmer Arnel Clarus 500, with Alumina column 30 m * 0.53 mm) equipped flame-ionization detector (FID). Operation conditions were 100 °C for oven, 225 °C for detector and 250 °C for injection port. Carrier gas that was used was argon with flow rate of 14 mL min⁻¹ (Bayr et al. 2012a). VFA were analyzed by gas chromatograph equipped with FID (Perkin-Elmer Autosystem XL, using Perkin-Elmer Elite FFAP column 27.5 m * 0.32 mm with film thickness 0.25 µm). Operation conditions for column oven were 130°C (initial) – 250 °C, 250 °C for detector and 230 °C for injection port. Helium (He) was used as carrier gas with pressure of 63.2 kPa with split ratio of 50 (Bayr et al. 2012a). VFA was determined from filtrate that was first centrifuged (Sanyo Harrier 18/80 centrifuge at 3500 rpm for 15 min) and then filtrated through glass microfiber filter (VWR GF/A Grade 691, 90 mm, 1.6 µm filter). VFA concentrations were converted to SCOD equivalents with the following coefficients: 1.066 for acetic acid, 1.512 for propionic acid, 1.816 for iso-butyric and butyric acid, 2.036 for iso-pentanoic (iso-valeric) and pentanoic (valeric) acid and 2.204 for hexanoic (caproic) acid (Bayr et al. 2012a).

Trace element concentration analyzes are based on accredited ISO-standard SFS-EN ISO 11885:09 (modif.), except B and Se are based on SFS:EN ISO 17294-2:05 (modif.), and were analyzed by outside party. Prior to trace element analyses, samples were pretreated using the ultrasound-assisted digestion method (Bayr et. al. 2012b). A sample volume of 20 mL was used throughout. The digestion solution of 6 mL aqua regia and 4 droplets of hydrogen fluoride (HF) was introduced into the sample vessel (50 mL centrifuge tube, Sarstedt) and placed into ultrasonic water bath. Sonication procedure was performed at 40°C that contains three steps: 3 minutes of sonication, 15 minutes of standing and 2 * 3 minutes sonication. After the sonication procedure, samples were centrifuged for 15 minutes at 3500 rpm and filtered (VWR GF/A Grade 691, 90 mm, 1.6 µm filter).

VS-removal (%) in reactor experiments was calculated in the end of each loading period using equation 1:

$$VS_{removal} = \frac{(VS\%_{feed} - VS\%_{effluent})}{VS\%_{feed}} * 100 \quad (1)$$

where VS_{removal} = amount of VS removed from effluent during digestion, $VS\%_{\text{feed}}$ = VS (%) in the substrate and $VS\%_{\text{effluent}}$ = VS (%) in effluent.

The concentration of NH_3 in process effluent was calculated using $NH_4\text{-N}$ concentration, pH and reactor temperature, by using equation 2 below (Bayr et al. 2012b):

$$f_{NH_3} = (1 + 10^{(pK_w - pK_b - pH)^{-1}})^{-1} \quad (2)$$

where f_{NH_3} = fraction of NH_3 , K_w = dissociated constant of water, K_b = ionization constant of NH_3 , $pK_w = 13.152$ (at 55 °C) and $pK_b = 4.721$ (at 55 °C) (Housecroft and Constable 2002).

3 RESULTS

3.1 Food waste and kitchen waste generation in the restaurant

The total number of customers and the amount of food waste and kitchen waste collected from the Ylistö restaurant are presented in Table 4. Overall, the number of customers during the sample period of 20 days was 4 979. The amount of kitchen waste and food waste collected during these 20 days was 232.1 kg and 308.6 kg, respectively. Based on the number of customers monitored during the waste collection period, the average amount of food waste produced per customer was 74.3 g d⁻¹. For kitchen waste, which was considered to be produced at fixed rate, the average produced waste amount is 11.6 kg d⁻¹.

Table 4 Number of customers and amount of wastes during monitoring period 24.9.-19.10.2012.

Date	No. of customers	Kitchen waste (kg)	Food waste (kg)	Food waste (g customer ⁻¹)
24.9.2012	165	5.6	9.2	55.8
25.9.2012	231	11.3	15.2	65.9
26.9.2012	330	6.4	14.4	43.5
27.9.2012	310	14.2	23.9	77.2
28.9.2012	210	4.6	11.8	56
1.10.2012	261	16.2	15.8	60.6
2.10.2012	252	9.6	11	43.7
3.10.2012	390	8.9	21.4	54.8
4.10.2012	334	11.2	20.4	61
5.10.2012	204	16.7	13.8	67.7
8.10.2012	292	15.8	15.2	52.2
9.10.2012	48	12.1	10.4	216.3
10.10.2012	347	11.5	18.3	52.8
11.10.2012	335	13.6	17.3	51.7
12.10.2012	205	16.0	24.8	121.1
15.10.2012	72	15.7	13.9	192.8
16.10.2012	235	25.1	17.1	72.6
17.10.2012	313	6.3	12.8	40.9
18.10.2012	215	5.1	11.8	55.1
19.10.2012	230	6.3	10	43.7
Overall	4979	232.1	308.6	74.3*

Note: * = average value.

3.2 Composition of food waste and kitchen waste

The composition of food waste and kitchen waste that were sampled from the Ylistö restaurant was determined during a 2 week period is presented in Table 5. Overall ca. 27 kg kitchen waste and 25 kg of food waste was collected during the 2 week sampling period. In general, fruit and vegetable waste dominated (43%) in the kitchen waste while mixed meal waste (70.8%) account for the highest waste fraction in the food waste.

Table 5 Composition of food waste and kitchen waste generated during 2 week period at the Ylistö restaurant, University of Jyväskylä in 2012.

Component	Kitchen waste		Food waste	
	Amount (kg)	Percentage value (%)	Amount (kg)	Percentage value (%)
Fruit and vegetable waste	11.6	43	4.7	19
Pasta and rice	7.8	29.1	1	4
Potato	2.2	8.2	1	4
Bread and bakery	3	11.3	0.5	2.2
Mixed meal wastes	2.3	8.4	17.4*	70.8

Note: * = includes paper napkins

3.3 Feedstock and inoculum chemical characteristics

The chemical composition and trace element concentrations of the substrates are presented in Table 6. Food waste had TS and VS content of 28.6% and 27.5% respectively with a pH of 5.1. The total volatile fatty acid (TVFA) and $\text{NH}_4\text{-N}$ content were 35.0 mg L^{-1} and 50.9 mg L^{-1} , respectively. Kitchen waste on the other hand had a TS and VS content of 16.7 and 15.5%, with pH 5.3. The TVFA and $\text{NH}_4\text{-N}$ content in kitchen waste was 24.0 mg L^{-1} and 32.1 mg L^{-1} , respectively. Inoculum had TS and VS content of 2.3% and 1.6%, with pH 8. TVFA and $\text{NH}_4\text{-N}$ concentration in the inoculum was 39.6 mg L^{-1} and 844 mg L^{-1} .

Table 6 Characteristics of substrates and inoculum used in the study. Standard deviations are shown in parenthesis.

Parameter	Kitchen waste		Food waste		Inoculum
	Batch 1	Batch 2	Batch 1	Batch 2	
TS (%)	16.7 (0.3)	22.3 (0.2)	28.6 (0.8)	31.3 (0.2)	2.3 (0.2)
VS (%)	15.5 (0.2)	21.3 (0.2)	27.5 (0.8)	29.9 (0.1)	1.6 (0.2)
VS/TS (%)	93.4	95.4	96.1	95.8	68.4
pH	5.3	5.4	5.1	5.6	8
TVFA (mg L ⁻¹)	24	N.D.	35	N.D.	39.6
TVFA (mgCOD L ⁻¹)	40.5	N.D.	45.1	N.D.	48.1
SCOD (mg L ⁻¹)	17.7	N.D.	24.3	N.D.	19.3
NH ₄ -N (mg L ⁻¹)	32.1	N.D.	50.9	N.D.	844
TKN (g L ⁻¹)	4	N.D.	7.1	N.D.	2.2
Al (mg kg ⁻¹)	19 (4)	N.D.	20 (4)	N.D.	4000 (810)
Bo (mg kg ⁻¹)	9 (2)	N.D.	6 (1)	N.D.	44 (9)
Co (mg kg ⁻¹)	< 1	N.D.	< 1	N.D.	4 (1)
Cu (mg kg ⁻¹)	4.0 (1)	N.D.	4 (1)	N.D.	55 (11)
Fe (mg kg ⁻¹)	49.0 (10)	N.D.	31 (6)	N.D.	7100 (1400)
Mn (mg kg ⁻¹)	13.0 (3)	N.D.	11 (2)	N.D.	370 (74)
Mo (mg kg ⁻¹)	< 1	N.D.	< 1	N.D.	3 (1)
Ni (mg kg ⁻¹)	< 1	N.D.	< 1	N.D.	10 (2)
Se (mg kg ⁻¹)	<0.2 (0.06)	N.D.	< 0.2	N.D.	4.1 (0.8)
Zn (mg kg ⁻¹)	24.0 (5)	N.D.	22 (4)	N.D.	410 (82)

Note: N.D. = not determined.

3.4 Batch experiments

Methane production potential of kitchen waste and food waste are presented in Figure 3. Results show that methane production started immediately in all assays without any lag phase. In both batches, about 90 % of methane was formed in 3 days (Figure 3). Methane production potentials for food wastes and kitchen wastes are presented in Table 7. For food waste methane production potential is 174 mL gVS_{added}⁻¹, 167 mL gTS⁻¹ and 110 m³ per tonne of fresh matter (FM). For kitchen waste methane production potentials are 186 mL gVS_{added}⁻¹, 173 mL gTS⁻¹ and 71 m³ per tonne of FM (w/w).

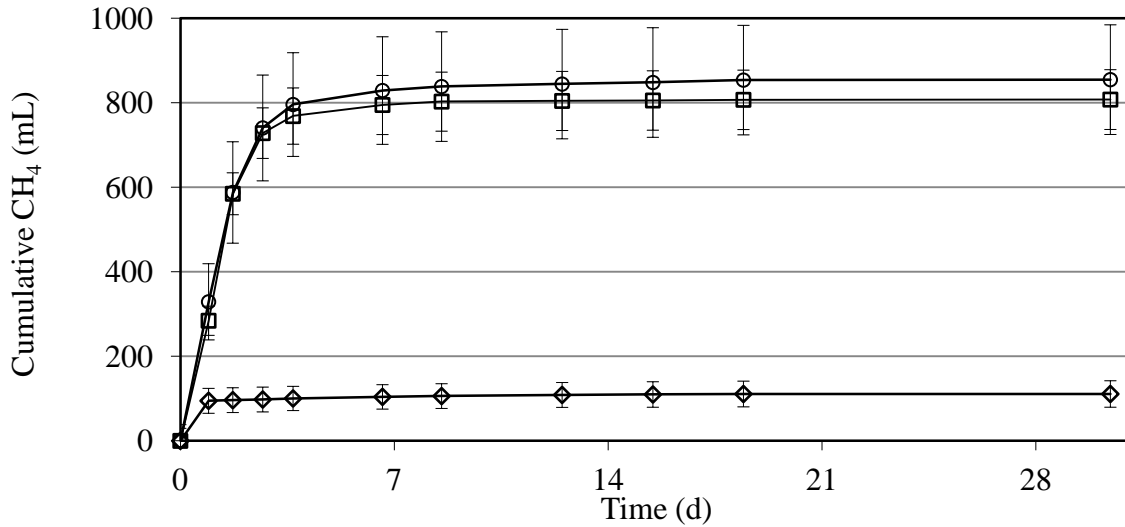


Figure 3 Cumulative methane production (mL) from inoculum alone (control, ◇), food waste (□) and kitchen waste (○) in batch assays incubated at 55 °C.

Table 7 Methane production potentials for food and kitchen wastes in batch assays. Calculated per VS, TS and fresh matter of substrate as wet weight (w/w).

	mL CH ₄ ⁻¹ gVS _{added}	mL CH ₄ ⁻¹ gTS	m ³ CH ₄ tonneFM ⁻¹ (w/w)
Food waste	174 ± 18	167 ± 17	110 ± 20
Kitchen waste	186 ± 32	173 ± 30	71 ± 12

Digestate characteristics for batch assays after the experiments are presented in Table 8. TS, VS and pH remained similar in all assays. Kitchen waste had lower TVFA content of 15.0 mg L⁻¹ compared to food waste (19.4 mg L⁻¹). Also NH₄-N concentration in kitchen waste assay (708.8 mg L⁻¹) was lower compared to food waste assay (768.3 mg L⁻¹).

Table 8 Digestate characteristics after batch experiments.

Parameter	Food waste	Kitchen waste	Inoculum
TS (%)	1.3	1.3	1.1
VS (%)	0.8	0.8	0.7
VS/TS (%)	60	59.7	59.8
pH	7.8	7.9	8.1
TVFA (mg L ⁻¹)	19.4	15	10.9
NH ₄ -N (mg L ⁻¹)	768.3	708.8	675.4

3.5 Reactor experiments

The effect of OLR on the process performance and methane yield during the thermophilic anaerobic digestion of food waste and kitchen waste was investigated in CSTR at 55 °C. The overall results for the food waste reactor (R1) are presented in Figure 4 and in Table 9, for the kitchen waste reactor (R2) in Figure 5 and in Table 10 and for the food waste and kitchen waste reactor (R3) is presented in Figure 6 and in Table 11. The Tables 9, 10 and 11 represent the operational parameters, methane production and digestate characteristics in the reactors obtained during the experiments.

3.5.1 Food waste

After the initial start-up for first loading period, R1 was fed with an OLR of 0.75 gVS L⁻¹ d⁻¹ and HRT of 50 d was used (days 1-50). Methane production responded to this OLR and mean methane yield of 72 mL gVS⁻¹ was obtained (Table 11). Thereafter for the loading period 2, OLR was increased to 1.5 gVS L⁻¹ d⁻¹ and HRT was reduced to 30 d (days 51-81). R1 responded well to this higher OLR with stable process and mean methane production of 87 mL gVS⁻¹ was obtained. Upon further increase in OLR to 3 gVS L⁻¹ d⁻¹ (days 82-112), a similar process performance and higher methane production was noticed. Nevertheless, daily methane yields started to show variation after OLR 3 gVS L⁻¹ d⁻¹ was introduced and after day 112, OLR 3 gVS L⁻¹ d⁻¹ was decided to be maintained in all three reactors for another 30 days (113-144) for the loading period 4. The mean methane yield in R1 was 139 mL gVS⁻¹. Further increase in OLR to 6 gVS L⁻¹ d⁻¹ (days 145-175) resulted almost doubling the methane yields. Similar process performance was noticed with the mean methane yield of 330 mL gVS⁻¹. Feeding in R1 was decided to be withheld for one week due unstable process performance in the end of loading period. Upon resumption of feeding on day 180, R1 was continued at the previous OLR of 6 gVS L⁻¹ d⁻¹ and R1 started to show more unstable process with increased TVFA concentrations. Still increased methane yields were obtained from R1 than before the unfed period with methane yield 399 mL gVS⁻¹.

NH₄-N concentration started to increase steadily after feeding at an OLR of 6 gVS L⁻¹ d⁻¹ (day 145) to reach 2.2 g L⁻¹ on day 204. pH followed more or less the same trend as that of free NH₃, reaching a maximum pH of 8.2 on day 204. The corresponding free NH₃ concentration on day 204 is 180 mg L⁻¹. NH₄-N concentration in R1 remained between 0.7 and 1.2 g L⁻¹ between days 1 and 143.

TVFA levels in R1 were below 200 mg L^{-1} throughout the experiment indicating no process inhibition due to VFA build-up, until the last loading period of the experiment when TVFA concentration increased to 2245 mg L^{-1} (day 188). On day 201 TVFA concentrations in R1 had decreased to 433 mg L^{-1} and increased to 920 mg L^{-1} on day 204. This type of accumulation is quite genuine, where TVFA concentration suddenly drops. This can be explained by change in sampling date. If VFA analyzes were executed right after the weekend, when no feeding occurred, TVFA concentrations seemed to have decreased. But if feeding was done on Monday and TVFA analyzes on next day, also increased VFA concentrations was noticed.

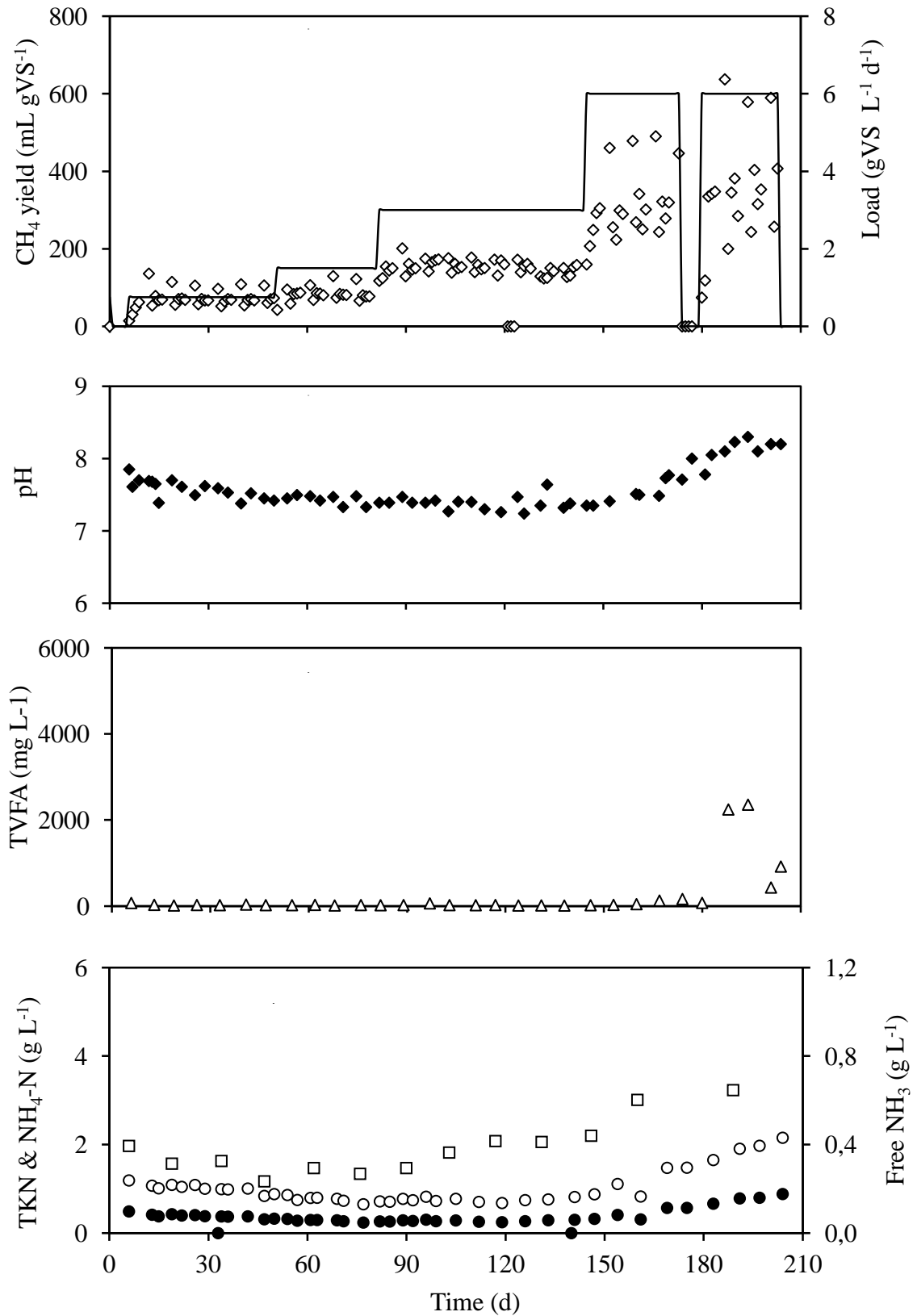


Figure 4 Process performance of food waste (R1) reactor at 55 °C. Load (-), TKN (□) NH₄-N (○) and free NH₃ (●).

Table 9 Experimental set-up, methane production and chemical characteristics in food waste reactor (R1). Standard deviations are shown in parenthesis.

Parameter	R1 Food waste					
Loading period	1	2	3	4	5	6
Days	1-50	51-81	82-112	113-144	145-175	180-207
OLR (gVS L ⁻¹ d ⁻¹)	0.75	1.5	3	3	6	6
HRT (d)	50	30	30	31	30	27
CH ₄ prod. (mL gVS ⁻¹)*	72 (2)	87 (4)	161 (7)	139 (7)	330 (2)	399 (28)
CH ₄ prod. (m ³ tonneFM ⁻¹)**	19 (4.4)	23.5 (0.9)	42.4 (2.7)	43.8 (2.8)	90.1 (12.0)	95.7 (21.9)
Vol. CH ₄ prod. (L d ⁻¹)**	3.8 (0.9)	7.8 (0.3)	14.1 (0.9)	14.6 (0.9)	27.6 (6.5)	33.3 (6.6)
CH ₄ conc. (%)*	56 (1)	58 (2)	58 (1)	56 (2)	61 (4)	66 (4)
VS-removal (%)***	96.4	97.3	95	94.7	93.4	92.5
TS (%)**	2 (0.4)	1.4 (0.2)	1.7 (0.1)	2.1 (0.1)	2.2 (0.4)	2.6 (0.1)
VS (%) **	1.2 (0.3)	0.8 (0.1)	1.2 (0.2)	1.7 (0.1)	1.8 (0.3)	2.0 (0.14)
VS/TS (%)	63.1	57.4	74.9	78.4	82.8	76.5
TVFA (mg L ⁻¹)**	33 (19)	24 (6)	32 (18)	18 (6)	79 (65)	1206 (1044)
TVFA (mgCOD L ⁻¹)**	41 (26)	29 (8)	44	25 (9)	96 (81)	1666 (1460)
NH ₄ -N (g L ⁻¹)**	1 (0.1)	0.8 (0.1)	0.7 (0)	0.7 (0.1)	1.1 (0.3)	1.9 (0.2)
TKN (g L ⁻¹)**	1.4 (0.3)	1.4 (0.1)	1.6 (0.2)	2.1 (0.1)	2.8 (0.5)	3.4 (0.2)
pH*	7.4 (0.1)	7.4 (0.1)	7.4 (0.1)	7.4 (0.1)	7.6 (0.1)	8.2 (0.1)

Note: *= Calculated from the last 2 weeks average at the end of the loading period, **= Average of the whole loading period, ***= Result calculated at the end of each loading period. Standard deviations are shown in parenthesis.

3.5.2 Kitchen waste

R2 was fed with similar loading strategy as R1. After initial start up, R2 was fed with an OLR of $0.75 \text{ gVS L}^{-1} \text{ d}^{-1}$ and HRT of 50 d was used (days 1-50). Mean methane yield of 131 mL gVS^{-1} was obtained for the loading period 1 (Table 12). For the loading period 2, OLR was increased to $1.5 \text{ gVS L}^{-1} \text{ d}^{-1}$ and HRT reduced to 30 d (days 51-81) and mean methane yields of 139 mL gVS^{-1} was obtained. During loading period fluctuation in daily methane yields started to occur compared to previous OLR. After further increase in OLR to $3 \text{ gVS L}^{-1} \text{ d}^{-1}$ (days 82-112), more fluctuation occurred with mean methane yields of 299 mL gVS^{-1} . Similarly to R1, after day 112 OLR $3 \text{ gVS L}^{-1} \text{ d}^{-1}$ was decided to be maintained for another 30 days for the loading period 4 (days 113-144). Daily methane yields started to slightly stabilize; though mean methane yield of 236 mL gVS^{-1} was obtained, that was lower than obtained from the previous loading period. For the loading period 4 (days 145-175), OLR $6 \text{ gVS L}^{-1} \text{ d}^{-1}$ was used. From the beginning of loading period R2 continued to show variation between daily methane yields, though nearly doubled mean methane yield 433 mL gVS^{-1} was obtained. In the end of loading period, feeding in R2 was decided to be withheld for one week due unstable process performance. For the last loading period (days 180-210), OLR $4.5 \text{ gVS L}^{-1} \text{ d}^{-1}$ was decided to put in use in order to maintain the process and to prevent the collapse of the process due increased ammonia and TVFA concentrations.

TVFA concentration in R2 remained below 1 g L^{-1} between days 1 and 138 increased sharply with the introduction of OLR of $6 \text{ gVS L}^{-1} \text{ d}^{-1}$ to reach a value of 2.7 g L^{-1} (day 174). During unfed period (days 174-179), TVFA concentration decreased to 1.6 g L^{-1} . Feeding kitchen waste at an OLR $4.5 \text{ gVS L}^{-1} \text{ d}^{-1}$ after unfed period (days 180-210) resulted in restoring the process, where TVFA concentration dropped from 4.2 g L^{-1} to 2.7 g L^{-1} in the end of the experiment on day 211.

After start-up, $\text{NH}_4\text{-N}$ concentration of 1.3 g L^{-1} was measured in R2. This concentration decreased below 1 g L^{-1} after day 30 and remained there until day 89. At OLR $3.0 \text{ gVS L}^{-1} \text{ d}^{-1}$ $\text{NH}_4\text{-N}$ concentration steadily increased to 1.3 g L^{-1} again. After introducing OLR $6 \text{ gVS L}^{-1} \text{ d}^{-1}$, concentration reached 2.2 g L^{-1} . In the last loading period $\text{NH}_4\text{-N}$ concentration slightly decreased from 2.5 g L^{-1} (day 183), having concentration of 2.4 g L^{-1} in the end of experiment (day 211). $\text{NH}_4\text{-N}$ and corresponding NH_3 concentrations are presented in Figure 5 below.

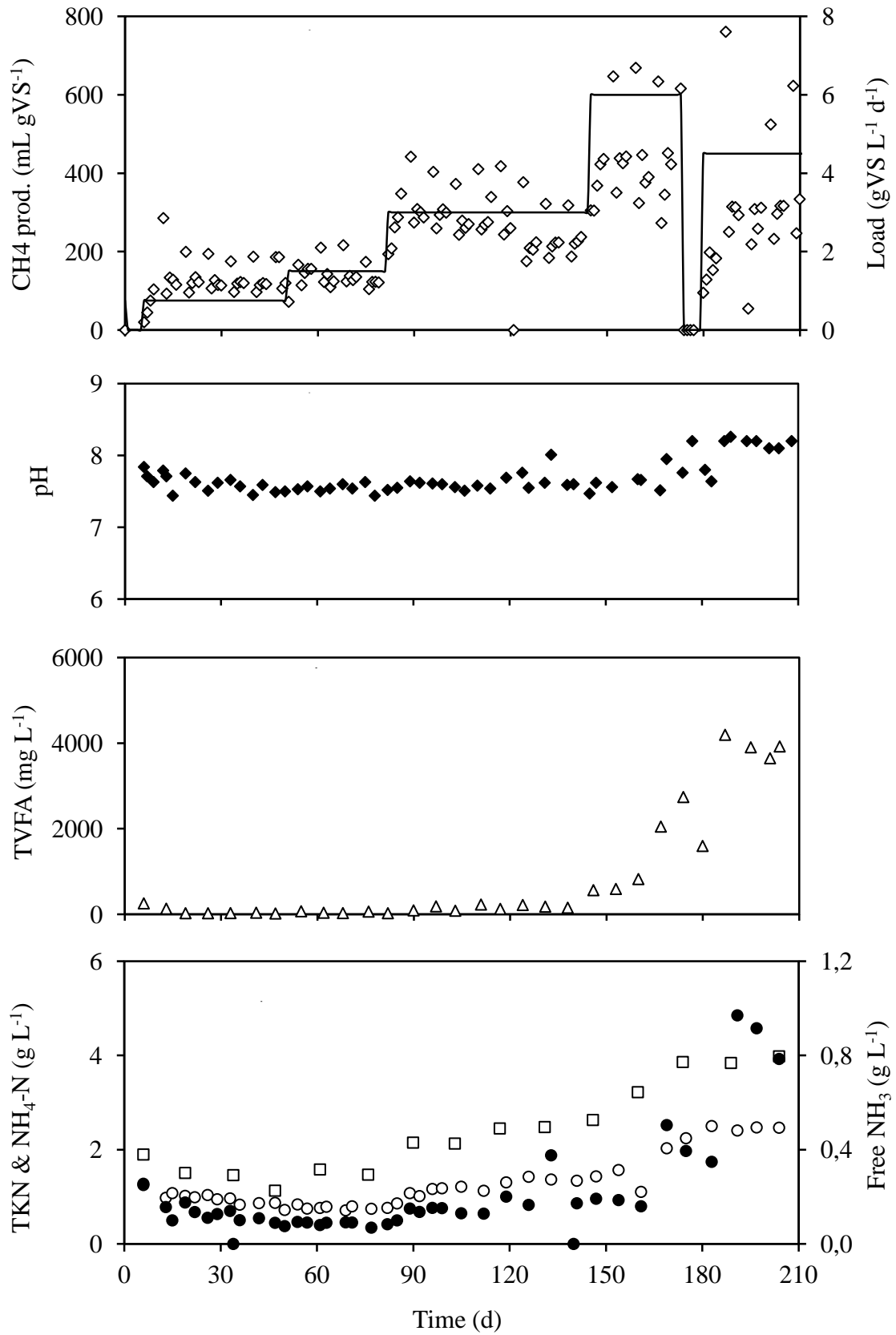


Figure 5 Process performance of kitchen waste (R2) reactor at 55 °C. Load (—), TKN (□) NH₄-N (○) and free NH₃ (●).

Table 10 Experimental set-up, methane production and chemical characteristics in R2. Standard deviations are shown in parenthesis.

Parameter	R2 Kitchen waste					
	1	2	3	4	5	6
Loading period						
Days	1-50	51-81	82-112	113-144	145-175	180-210
OLR (g VS L ⁻¹ d ⁻¹)	0.75	1.5	3	3	6	4.5
HRT (d)	50	30	30	31	30	27
CH ₄ prod. (mL gVS ⁻¹) *	131 (5)	139 (14)	299 (20)	236 (3)	433 (11)	284
CH ₄ prod. (m ³ tonneFM ⁻¹)**	18.9 (5)	22.0 (1.4)	46.3 (3.9)	50.7 (1.0)	90.3 (8.6)	61.2
Vol. CH ₄ (L d ⁻¹)**	3.8 (1)	7.3 (0.5)	15.4 (1.3)	16.9 (0.3)	27.5 (6.3)	25.4
CH ₄ conc. (%)*	55 (1)	55 (3)	59 (1)	61 (2)	63 (3)	63
VS-removal (%)***	92.9	92.4	92.6	93.8	91.8	90.6
TS (%)**	2.1 (0.3)	1.6 (0)	1.6 (0.1)	1.9 (0)	2 (0.5)	2.6 (0.5)
VS (%) **	1.4 (0.3)	1.1 (0.1)	1.1 (0.1)	1.3 (0)	1.5 (0.4)	1.9 (0.1)
VS/TS (%)	66.8	69.3	66.5	70.1	73.5	74.8
TVFA (mg L ⁻¹)**	77 (89)	52 (20)	125 (82)	172 (39)	1356 (986)	3454 (1055)
TVFA (mgCOD L ⁻¹)**	98 (105)	62 (24)	167 (117)	216 (51)	1794 (1332)	4910 (1553)
NH ₄ -N (g L ⁻¹)**	1.0 (0.1)	0.8 (0)	1.1 (0.2)	1.4 (0.1)	1.7 (0.5)	2.5 (0)
TKN (g L ⁻¹)**	1.5 (0.3)	1.5 (0.1)	2.1 (0)	2.5 (0)	3.2 (0.6)	3.9 (0.1)
pH*	7.5 (0.1)	7.6 (0.1)	7.6 (0)	7.7 (0.2)	7.7 (0.2)	8.2 (0.1)

Note: *= Calculated from the last 2 weeks average at the end of the loading period, **= Average of the whole loading period, ***= Result calculated at the end of each loading period. Standard deviations are shown in parenthesis.

3.5.3 Co-digestion of food waste and kitchen waste

R3 with ratio 4.5:1 food waste to kitchen waste had same feeding strategy as R1, with mean methane yields for 6 loading periods of 76, 90, 172, 150, 327 and 354 mL gVS⁻¹. Results are presented in Figure 6 and Table 11. The daily methane yields started to show variation after OLR 3 gVS L⁻¹ d⁻¹ was introduced.

As can be seen from Figure 6, NH₄-N concentrations of 1.10 g L⁻¹ were measured in R3 after initial start-up. Also pH increased to 8.5 during this time. High pH and ammonia concentrations were because the reactor was not sealed properly. But after start-up problems were solved, both NH₄-N and pH started to decrease. pH remained rather steady during days 51-112. During the loading period 4 (days 113-144) pH steadily decreased and dropped temporarily below 7 in the end of loading period. From this point it started to increase, reaching 8 at the end of experiment (day 207). TVFA concentration maintained below 150 mg L⁻¹ throughout the process and basically no inhibition due to VFA build-up occurred. TVFA concentration in R3 was less than 70 mg L⁻¹ in the end of experiment (day 204). However, the NH₄-N and NH₃ concentrations of 1.84 g L⁻¹ and 274 mg L⁻¹ was measured in the end of last loading period that had been steadily increasing throughout the experiment.

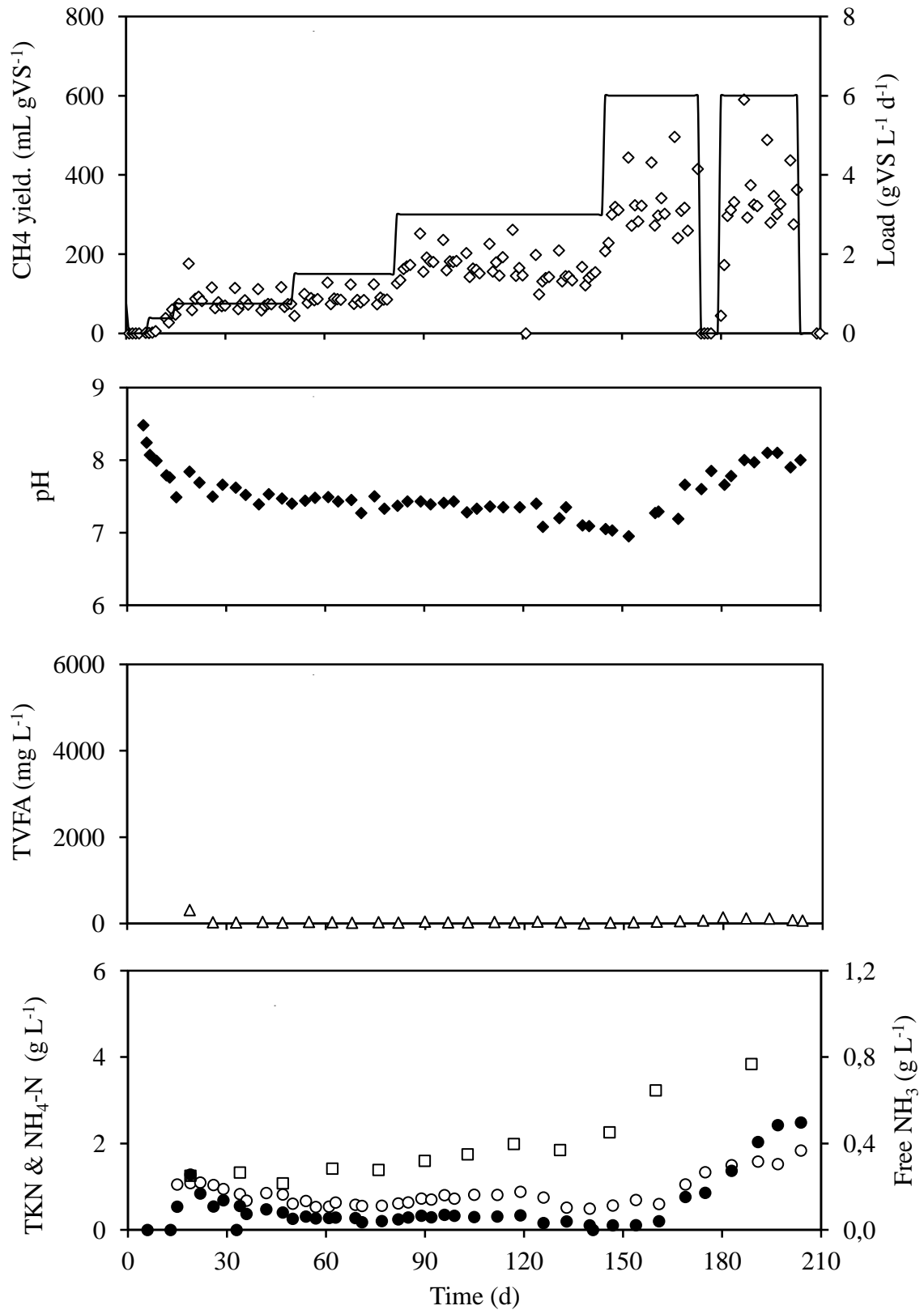


Figure 6 Process performance of food and kitchen waste (in ratio of 4.5:1) reactor (R3) during at 55 °C. Load (—), TKN (□) NH₄-N (○) and free NH₃ (●).

Table 11 Experimental set-up, methane production and chemical characteristics in R3. Standard deviations are shown in parenthesis.

Parameter	R3 Food waste and kitchen waste (4.5:1)					
	1	2	3	4	5	6
Loading period	1	2	3	4	5	6
Days	1-50	51-81	82-112	113-144	145-175	180-207
OLR (g VS L ⁻¹ d ⁻¹)	0.75	1.5	3	3	6	6
HRT (d)	50	30	30	31	30	27
CH ₄ prod. (mL gVS ⁻¹)*	76 (2)	90 (2)	172 (11)	150 (4)	327 (3)	354 (7)
CH ₄ prod. (w/w) (m ³ tonneFM ⁻¹)**	15.7 (9.6)	22.8 (0.6)	44.4 (4.2)	42.6 (2.3)	88.8 (7.6)	91.0 (14.0)
Vol. CH ₄ prod. (L d ⁻¹)**	3.3 (1.7)	7.6 (0.2)	14.8 (1.4)	14.2 (0.8)	26.9 (6.3)	29.9 (6.0)
CH ₄ conc. (%)*	56 (2)	58 (1)	56 (2)	55 (2)	57 (2)	59 (1)
VS-removal (%)***	95.4	96.1	94.8	93.7	89.7	89.0
TS (%)**	2 (0.3)	1.5 (0.1)	1.6 (0.2)	2 (0.4)	2.9 (0.9)	3.8 (0)
VS (%) **	1.3 (0.2)	1 (0.1)	1.2 (0.2)	1.5 (0.4)	2.3 (0.7)	3.1 (0)
VS/TS (%)	63.9	68.4	72.6	78.2	80.9	80.6
TVFA (mg L ⁻¹)**	83 (127)	27 (8)	29 (9)	25 (18)	42 (19)	102 (31)
TVFA (mgCOD L ⁻¹) **	117 (188)	33 (9)	34 (11)	28 (20)	52 (23)	126 (45)
NH ₄ -N (g L ⁻¹)**	0.9 (0.2)	0.6 (0.1)	0.7 (0.1)	0.7 (0.2)	0.9 (0.3)	1.7 (0.2)
TKN (g L ⁻¹)**	1.2 (0.1)	1.4 (0)	1.7 (0.1)	1.9 (0.1)	3.0 (0.7)	4.0 (0.2)
pH*	7.4 (0.1)	7.4 (0.1)	7.4 (0.1)	7.2 (0.1)	7.4 (0.2)	8.0 (0.1)

Note: *= Calculated from the last 2 weeks average at the end of the loading period, **= Average of the whole loading period, ***= Result calculated at the end of each loading period. Standard deviations are shown in parenthesis.

3.5.4 Volatile fatty acids

In Figure 7 is presented VFA profiles of each reactor. Acetic acid and propionic acid showed largest concentrations among VFAs. In R1 acetic acid peaked between 807-816 mg L⁻¹ (860-870 mgCOD L⁻¹) during days 189-196 in the last loading period, though concentrations decreased from this. Similarly concentrations 1271-1362 mg L⁻¹ (1925-2062 mgCOD L⁻¹) of propionic acid was noticed at the time. In R2 acetic acid peaked at 720-1384 mg L⁻¹ (767-1475 mgCOD L⁻¹) between days 168-212. Propionic acid reached its peak concentration on day 202 with 3028 mg L⁻¹ (4585 mgCOD L⁻¹). In R3 TVFA concentrations remained below 126 mgCOD L⁻¹ throughout the experiment.

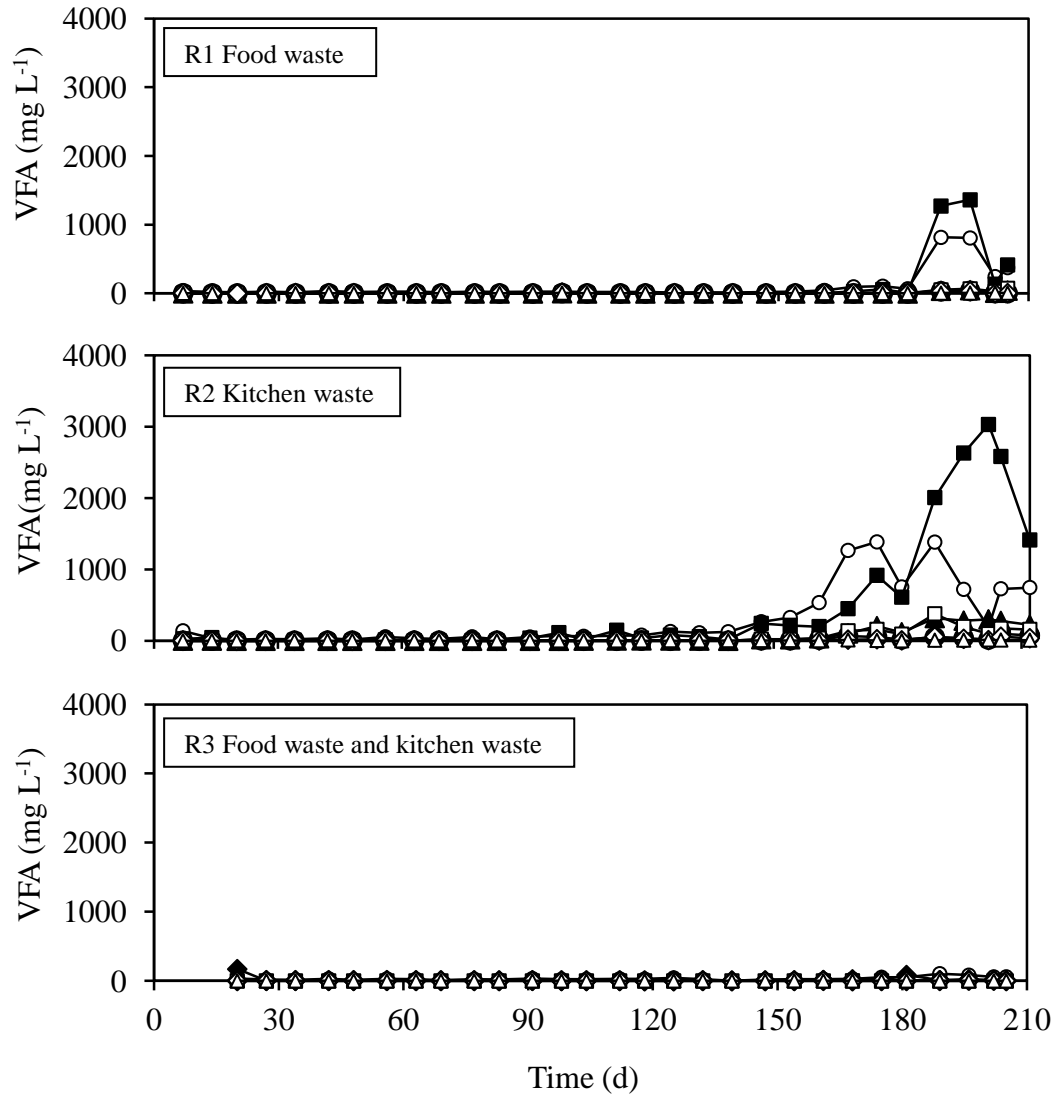


Figure 7 VFA profile in R1, R2 and R3 digesting food waste, kitchen waste and 4.5:1 mixture of the substrates, respectively. Acetic acid (\circ), propionic acid (\blacksquare), isobutyric acid (\blacktriangle), butyric acid (\bullet), isovaleric acid (\square), valeric acid (\diamond) and caproic acid (\triangle).

3.5.5 Trace element concentrations in digestate

Results of trace element concentration in feedstocks and inoculum are presented in Table 8. Table 14 presents effluent trace element concentrations during operation in each reactor at the end of loading periods 2 (day 81) and 4 (day 144). The concentration of each trace element decreased during the experimental period from that of inoculum. Al, Fe, Mn and Zn concentrations decreased most in all reactors. Al concentration in inoculum was 4000 mg kg^{-1} , while monitored concentrations in all reactors was $13\text{-}22 \text{ mg kg}^{-1}$. Fe concentration decreased from 7100 mg kg^{-1} to $8\text{-}37 \text{ mg kg}^{-1}$. Mn concentration dropped from 370 mg kg^{-1} to $0.96\text{-}2.3 \text{ mg kg}^{-1}$. Zn concentration in inoculum was 410 mg kg^{-1} while in reactors

4.1-7.1 mg kg⁻¹. Also other elements, i.e. Bo, Co, Cu, Mo, Ni and Se that were found in inoculum in smaller concentrations, had decreased concentrations during the experiment had increased in all reactors between loading periods 2 and 4.

Table 12 Reactor effluent trace element concentrations during the process. Standard deviations are shown in parenthesis.

Parameter	R1 Food waste		R2 Kitchen waste		R3 Food waste and kitchen waste	
	2	4	2	4	2	4
Loading period	2	4	2	4	2	4
OLR	1.5	3	1.5	3	1.5	3
Day	81	140	81	140	81	140
Al (mg kg ⁻¹)	22 (3)	15 (2)	22 (3)	14 (2)	23 (3)	13 (2)
Bo (mg kg ⁻¹)	2.8 (0.4)	4.1 (0.6)	2.1 (0.3)	2.8 (0.4)	1.6 (0.2)	2.2 (0.3)
Co (mg kg ⁻¹)	0.043 (0.007)	0.18 (0.03)	0.07 (0.01)	0.30 (0.05)	0.05 (0.01)	0.21 (0.03)
Cu (mg kg ⁻¹)	0.61 (0.01)	0.48 (0.01)	0.54 (0.08)	0.49 (0.07)	1.2 (0.2)	0.5 (0.08)
Fe (mg kg ⁻¹)	35 (5)	11 (2)	27 (4)	13 (2)	37 (6)	7.6 (1)
Mn (mg kg ⁻¹)	1.9 (0.3)	1 (0.1)	1.9 (0.3)	1.3 (0.2)	2.3 (0.3)	0.96 (0.1)
Mo (mg kg ⁻¹)	0.046 (0.007)	0.06 (0.01)	0.041 (0.006)	0.095 (0.014)	0.052 (0.01)	0.06 (0.01)
Ni (mg kg ⁻¹)	0.83 (0.12)	0.40 (0.06)	0.23 (0.03)	0.49 (0.07)	0.35 (0.05)	0.32 (0.05)
Se (mg kg ⁻¹)	0.003 (0.0003)	0.010 (0.001)	0.004 (0.0004)	0.015 (0.002)	0.003 (0.0003)	0.012 (0.001)
Zn (mg kg ⁻¹)	7.1 (1)	7 (1)	4.7 (0.7)	5.8 (0.9)	4.9 (0.7)	4.1 (0.6)

4 DISCUSSION

4.1 Food waste and kitchen waste generation and characteristics

Waste generation of food waste and kitchen waste was studied between 24.9.-19.10.2012 in the Ylistö restaurant. The composition of both feedstocks was analyzed from the sampled wastes. The results of present study show that the amount of waste generated and the composition of waste can vary depending upon the source or type of restaurant, food menu and number of customers.

4.1.1 Food waste and kitchen waste generation and composition

Variation between daily food waste amounts (Table 4) to average food waste produced per customer was caused by higher weight waste fractions such as fruit and vegetable peels, chicken bones and skins, but also leftover pizza crust etc. Restaurant produces monthly 232 kg of kitchen waste i.e. ca. 2780 kg of kitchen waste yearly (Table 4). Compared with overall customers in Ylistö in 2012, ca. 9450 kg of food waste is generated annually (Tables 2 and 4). According to study by Silvennoinen et al. (2012), the amount and type of leftovers vary noticeably from one restaurant to another, depending on the restaurant's business model and type, which in turn is determined by the portion sizes and the menu. It is estimated that workplace restaurants and canteens serve 14% of all food in the Finnish restaurant sector and in these establishments 24% of food became waste divided as follows: kitchen waste 3%, service waste 17%, and leftovers 4% (Silvennoinen et al. 2012). In the present study, no absolute values with respect to the overall quantities of food supplies used in the Ylistö restaurant were available. According Silvennoinen et al. (2012), workplace restaurants and canteens produce 13-16 million kg of food waste annually. In a similar study by Climenhaga et al. (2010) it was estimated that 28 kg of food waste is produced per student over the course of a 30 week academic year. In the present study, average food waste produced per customer in the Ylistö restaurant over the same time period (30 weeks) would be 11 kg per student and 18 kg per student if kitchen waste is also included. The amount of waste generated at the University of Jyväskylä was substantially lower, compared to students at the University of Southampton. In Finnish society including the families, schools and universities, it is a common practice to educate children and students "you take what you eat" or "mind the waste"- practices, where discouraging food wastage is common and it is quite uncommon to see much leftover food on anyone's plate at the end of lunch.

Nevertheless, in order to reduce waste production, proper waste management practices should be followed accordingly. Not only reminding students of refraining from the food wastage and reducing quantity of the paper napkin waste produced, but also kitchen personnel from reducing amount of food preparatory wastes if possible are some of the measures to achieve this.

The composition of kitchen waste is quite similar as reported by Heaven et al. (2010) and Silvennoinen et al. (2012), with large fruit and vegetable waste fraction. High fraction of pasta and rice (29.1%), potatoes (8.2%) and bread and bakery products (11.3%) in the kitchen waste show in higher fractions compared to Heaven et al. (2010) since this type of foods are much utilized in university canteen type businesses. At the present study, food waste fraction composed largely of paper napkins. Exact percentage was not studied as napkins were smeared with leftover food and had attracted moisture, which made separation difficult. More exact food waste composition would be achieved with separation of food waste from paper waste when leftover food is disposed. Comparable results from other studies of the food waste fraction collected from institutional sources, where paper waste was included, was not found. Aim of the present research was to study suitability of current waste fractions for anaerobic digestion that are more dependent on nutrient composition and chemical characteristics of the feedstock.

4.1.2 Food waste and kitchen waste chemical characteristics

Characteristics of substrates and inoculum used in the study are presented in Table 6. As previously pointed out, even the general composition of food wastes may have regionally or culturally distinct characteristics, the chemical composition of food wastes remain more similar due to the fundamental requirements of human diet (Heaven et al. 2010). Food waste had similar content to that reported by Heaven et al. (2010) and Zhang et al. (2007). TKN in kitchen waste (4 g L^{-1}) was lower ($6.45\text{-}8.12 \text{ g kg}^{-1}$) than reported in study by Heaven et al. (2010). On the other hand, the characteristics of kitchen waste used in the present study were comparable to that of the results reported by Zhang et al. (2007). There are some differences between chemical characteristics obtained from different studies and therefore it is more practical to compare feedstock characteristics between food waste and kitchen waste of the present study.

At the present study, the higher moisture content in the kitchen waste fraction compared to the food waste fraction is due absence of paper napkins and other paper packaging waste.

The TVFA and $\text{NH}_4\text{-N}$ content for food waste were 35 mg L^{-1} ($45.1 \text{ mgCOD L}^{-1}$) and 50.9 mg L^{-1} , respectively. TVFA and pH for kitchen waste were more or less similar to that of food waste. However, the $\text{NH}_4\text{-N}$ in kitchen waste was lower than that of food waste with concentration of 32.1 mg L^{-1} . This difference in chemical characteristics of the two studied substrates is obviously due to the variation in the composition of the waste fractions. Kitchen waste is generated prior to actual cooking of food and is mainly composed of vegetable wastes and perishable foodstuffs from previous days. Also TKN in kitchen waste was lower than in food waste, with values 4.0 g L^{-1} and 7.1 g L^{-1} , respectively.

4.2 Methane production

Methane production potential of kitchen waste and food waste was assessed in batch assays. The effect of OLR on the process performance and the methane yield during the thermophilic anaerobic digestion of food waste and kitchen waste was investigated in CSTR at $55 \text{ }^\circ\text{C}$.

4.2.1 Batch assays

The higher methane yields obtained for kitchen waste than for food waste (Figure 3 and Table 7) in the present study was due to fact that the chemical characteristics of both materials are different. High paper napkin content in the food waste presumably increases the C/N-ratio of the substrate and lower methane potential compared to the kitchen waste was expected (Zhang R. et al. 2007, Banks et al. 2008, Heaven et al. 2010, Li Y. et al. 2011).

Methane yields of food wastes obtained in literature are presented in Table 15. The methane yields obtained for both kitchen and food wastes in the present study were lower than those reported in literature. However, methane yields were similar to those reported by Forster-Carneiro et al. (2008) for food waste collected from university campus restaurant. Moreover, the chemical characteristics of food waste used in the present study were similar to that used in the above, with exception of higher pH that was 8 and better C/N-ratio of 23.4. On the other hand, studies by Zhang R. et al. (2007), Pecorini et al. (2012) and Li Y. et al. (2011) food waste was synthetic or from municipal sources, while in the studies by Forster-Carneiro et al. (2008) and Zhang C. et al. (2013) food waste was collected from university canteens similar to present study. None of the studies assessed composition of waste feedstocks in their studies. Overall, the chemical characteristics of the feedstock and the nutrient composition in anaerobic digestion are most eminent.

The results show that methane production started immediately in all assays without any lag phase. The digestate characteristics (Table 10) suggest that no inhibition occurred during the batch experiments. pH in batch assays remained similar and $\text{NH}_4\text{-N}$ and TVFA concentrations were lower compared to the inoculum.

Table 13 Mono-digestion of food wastes in present study and other literature.

Experimental set-up	Feedstock	T (°C)	OLR	HRT (d)	Methane yield (mL gVS ⁻¹)	Reference
Batch	Food waste	55	-	30	174	Present study
Batch	Kitchen waste	55	-	30	186	Present study
Batch	Food waste	50	-	28	435	Zhang R. et al. (2007)
Batch	Food waste	55	-	90	180	Forster-Carneiro et al. (2008)
Batch	Food waste	35	-	27	410	Zhang C. et al. (2013)
Batch	Food waste	37.5	-	21	520-542	Pecorini et al. (2012)
Batch	Fruit & Vegetable	37.5	-	21	353	Pecorini et al. (2012)
Batch	Dirty paper	37.5	-	21	422	Pecorini et al. (2012)
Batch	Synthetic kitchen waste	37	-	50	219-286	Li, C. et al. (2011)
CSTR	Food waste	55	3	30	139	Present study
CSTR	Food waste	55	6	30	399	Present study
CSTR	Kitchen waste	55	3	30	236	Present study
CSTR	Kitchen waste	55	6	30	433	Present study
CSTR	Domestic food waste	36.5	4	28	390	Banks et al (2008)
CSTR	Domestic food waste	56	4	28	410	Banks et al (2008)

As shown in Table 7, where methane production potentials for food and kitchen wastes in batch assays are presented, methane potential for food waste and kitchen waste are 110 and 71 m³ per tonne of waste (w/w), respectively. With calculated total waste production quan-

tities, from food waste (w/w) ca. 1 040 m³ and from kitchen waste (w/w) ca. 200 m³ of CH₄ could be produced annually.

4.2.2 Semi-continuous reactor experiments

Among the three feedstocks, thermophilic digestion of kitchen waste produced highest methane yields (per gVS) compared to food waste and mixture of food waste and kitchen waste. Compared to the methane yields reported by Banks et al. (2008) at OLR 4 gVS L⁻¹ d⁻¹ in mesophilic conditions, present study had lower methane yields (Table 15). Food wastes in study by Banks et al. (2008) were from domestic sources and at present study from institutional sources.

The maximum mean methane yields at OLR 6 gVS L⁻¹ d⁻¹ for food waste was 399 mL gVS⁻¹, kitchen waste 433 mL gVS⁻¹ and 4.5:1 mixture ratio of food waste and kitchen waste 354 mL gVS⁻¹ in thermophilic process conditions. Nevertheless, the variation between daily methane yields after introducing OLR 3 gVS L⁻¹ d⁻¹ suggests that OLRs above this may be too high, especially under longer time period. As can be seen in Figure 6 for the food waste and kitchen waste reactor the maximum OLR that could sustainably be used in order to maintain stable methane production under thermophilic process is around 3 gVS L⁻¹ d⁻¹. In the study by Banks et al. (2008) digestion under thermophilic conditions at OLR 4 gVS L⁻¹ d⁻¹ showed more efficient process and enhanced methane yields, but required a reduced loading to be applied due very high VFA levels. Furthermore, OLR was reduced to 1 gVS L⁻¹ d⁻¹ and gradually increased back to 3 gVS L⁻¹ d⁻¹ that stopped VFA from accumulating and decreased the TVFA concentration. Results of the present study similarly show that too high OLR causes accumulation of intermediate products and inhibition of the process, while lower OLR give low methane yield and smaller amount of feedstock being treated at the same time. As seen from the studies by Marañón et al. (2012) and Zhang C. et al. (2013), addition of cattle manure or sewage sludge was noticed to enhance the buffer capacity of the process. Mono-digestion of food wastes was considered hardly feasible, but co-digestion with either co-substrates resulted in enhanced methane production and also use of higher OLR was possible in both mesophilic and thermophilic conditions.

High and stable VS-removal was noticed in all reactors throughout the experiment, which suggests high waste biodegradation (Tables 9, 10 and 11). The waste generation quantities that are ca. 2780 kg of kitchen waste and ca. 9450 kg of food waste per year, results to overall 12 tonnes of wastes per year to be treated. In a digester these could be treated with

OLR $3 \text{ gVS L}^{-1} \text{ d}^{-1}$ and HRT of 30d, while methane yield is ca. 44 m^3 per tonne wastes (w/w) fed. This is an encouraging result, especially when it is considered that transporting co-substrates i.e. sewage sludges or cattle manure to on-site biogas plants is not always economically or environmentally feasible.

4.2.3 Stability and inhibition in CSTR process

The experiments with semi-continuous anaerobic reactors show that institutional food and kitchen waste are well suited substrates for biogas production. Nevertheless; inhibition related to mono-digestion, elevated process temperature and composition of feedstock occurred at OLRs above $3 \text{ gVS L}^{-1} \text{ d}^{-1}$. During the experiment, digester fed with food waste and kitchen waste had only minor VFA accumulation but there was increased pH owing to NH_3 build-up. Kitchen waste digester had highest concentrations of $\text{NH}_4\text{-N}$ and VFA-concentrations measured, while food waste digester showed similar signs but in lower concentrations. TVFA-levels in a thermophilic process can reach as high as 45 g L^{-1} (Banks et al. 2008), while the present study had highest TVFA measured at 4.2 g L^{-1} . TVFA-levels were significantly lower than reported, and while accumulation of VFA and NH_3 was noticed in the present study, neither of the processes completely failed.

As previously mentioned, it is considered that especially the undissociated VFA species are more toxic as they can more easily diffuse to the inner parts of micro-organism cell. The most inhibitory VFAs are propionic and butyric acid (Mata-Alvarez 2003). According to Banks et al. (2012) mesophilic food waste digesters without trace element supplementation VFA accumulation occurs and the main component of accumulation is propionic acid. Moreover, propionic acid and other longer chain length acids initially show in lower concentrations (Banks et al. 2012). The present study similarly shows that initially acetic acid was predominant in the reactors and the build-up of propionic acid could be seen while digesting kitchen waste from day 100 and more significantly from day 140 onwards.

Accumulation of VFA is a known consequence of ammonia toxicity, and the point this started to occur when the free NH_3 concentration had reached around 0.5 g L^{-1} while digesting kitchen waste. Highest level of NH_3 measured in kitchen waste reactor was 2.5 g L^{-1} , that is significantly over the threshold level (1.2 g L^{-1}) suggested by Mata-Alvarez (2003). Furthermore, 50% inhibition of CH_4 production has been observed at NH_3 concentrations of 468 mg L^{-1} in thermophilic processes (Benabdallah El Hadj et al. 2009). In both food waste reactor and food waste and kitchen waste reactor, NH_3 concentration remained

below 0.5 g L^{-1} throughout the process, and only in the food waste reactor substantial TVFA concentrations could be seen in the end of experiment. The concentration of NH_3 is directly proportional to the temperature and there is an increased risk of ammonia inhibition at thermophilic temperatures compared to mesophilic ones. The high protein content in substrate usually gives out high nitrogen content during hydrolysis, while high nitrogen content usually leads to elevated NH_3 concentrations in process especially during extended run times (Banks et al. 2008, 2010)

Both selenium and cobalt have been found to be present only at very low concentrations in source segregated food waste (Banks et al. 2012). Although typically present in inoculum taken from municipal wastewater digestion, these trace elements would be digested over a period of time. Selenium is an essential trace element for food waste digestion. It is also confirmed that without trace element supplementation an accumulation of VFA occurs in mesophilic processes, and the main component in this accumulation is propionic acid (Banks et al. 2012). In the study by Banks et al. (2012), acetic acid was initially found predominant, while propionic acid and other longer chain length VFA were at low concentrations. According to Banks et al. (2012) the non-reversible accumulation of propionic acid was considered to occur due to deficiency of trace elements. Trace elements are required for synthesis of the enzymes needed in syntrophic hydrogenotrophic methane production, even though the results did not prove this hypothesis. Nevertheless, the supplementation with selenium and cobalt allowed the OLR of the system to be increased to $5 \text{ gVS L}^{-1} \text{ d}^{-1}$ resulting in a higher specific methane yield and almost three fold the volumetric biogas production.

The present study did not have a control reactor operating without trace element supplementation that would make the comparison of the possible effects of trace element supplementation to the VFA accumulation possible. Furthermore, for thermophilic digestion minimum supplementary trace nutrient concentrations that could prevent process failure and improve the process performance have not been established to date. Also, there are yet unpublished reports that trace nutrient supplementation may not have an effect in the thermophilic conditions. Nevertheless, as suggested by Uemura (2010) and Takashima et al. (2011), it may be that thermophilic digestion requires more trace nutrients than mesophilic digestion or supplementation of trace elements in thermophilic process may delay the onset of VFA accumulation only to a limited extent.

5 CONCLUSIONS

Restaurant Ylistö at University of Jyväskylä produces monthly 232 kg of kitchen waste i.e. ca. 2780 kg of kitchen waste yearly. Furthermore, the average amount of food waste produced per customer is 74.3 g d^{-1} that generates ca. 9450 kg of food waste annually. Produced kitchen waste is mainly composed of fruit and vegetable waste (43%), while the mixed meal waste (70.8%) account for the highest waste fraction in food waste. Significant proportion of mixed meal waste consists of paper napkins. Food waste had average TS and VS content of 28-32% and 27-30% and kitchen waste 16-23% and 15-22%, respectively.

Batch experiments showed that the methane potential for food waste and kitchen waste are 110 and 71 m^3 per tonne of waste, respectively. With calculated waste production quantities, from food waste ca. 1040 m^3 and from kitchen waste ca. 200 m^3 of CH_4 could be produced annually.

The results of this experimental study also shows different behavior patterns from organic matter biodegradation in thermophilic CSTR process. The highest specific methane yield for food waste at $\text{OLR } 6 \text{ gVS L}^{-1} \text{ d}^{-1}$ is 399 mL gVS^{-1} and for kitchen waste 433 mL gVS^{-1} . Co-digestion of both waste feedstocks had the highest specific methane yield of 354 mL gVS^{-1} . The present study shows that too high OLR causes accumulation of intermediate products and inhibition of the process. The maximum OLR that could sustainably be used in longer time period in order to maintain stable methane production under thermophilic process is around $3 \text{ gVS L}^{-1} \text{ d}^{-1}$ that in co-digestion of food waste and kitchen waste with HRT 30 d yields methane 150 mL gVS^{-1} .

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