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.

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Keywords

Food waste, digestate, autoclave treatment, characterisation, ammonium nitrogen, 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 (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 kgVS/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 kgVS/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 kgVS/m^{3}d (total organic carbon was analysed during organic loading rate 4 kgVS/m^{3}d) while digestate samples for the hygiene analyses were collected during organic loading rates of 4 kgVS/m^{3}d (4 samples) and 6 kgVS/m^{3}d (3 samples) (Table 1). The food waste samples were collected simultaneously with the digestates (6 to 7 samples) and thawed and stored in a freezer (4 °C) for 1-5 days prior to analyses.

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).

2.3. 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 one minute

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.

2.4. 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 (NH4-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 hour. Diluted FW 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, 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 et al., 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 to 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 100-260 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 NH4-N/TKN ratio was ~30% lower (Table 2). The reduced NH4-N and NH4-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 NH4-N concentration in the AFW digestate increased by 0.95 g/kgFM while with the other two digestates NH4-N increase was ca 0.3 g/kgFM (Figure 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 NH4-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 NH4-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 NH4-N formation with AFW along with low NH4-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 NH4-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 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

278 The hygienic quality of the FW (7 samples) and digestate (6 samples) were tested
279 with hygiene indicators *E. coli*, other coliforms, total coliforms, enterococci and
280 sulphite-reducing clostridia and Salmonella (Figure 2). No Salmonella was detected in
281 any of the feed or digestate samples (data not shown). In one of the six FW samples a
282 few colonies of *E. coli* were discovered, while both enterococci (average $2.79 \times 10^4 \pm$
283 2.74×10^4 cfu/g) and clostridia ($2.24 \times 10^3 \pm 1.86 \times 10^3$ cfu/g) were also discovered. In
284 AFW all hygiene indicators were under the detection limit (5 cfu/g). In both digestates,
285 high enterococci concentrations (6.77×10^8 cfu/g $\pm 7.40 \times 10^8$ and $3.71 \times 10^8 \pm 4.64 \times$
286 10^8 in the FW and AFW digestate) were detected, while the clostridia concentration
287 remained lower (6.14×10^2 cfu/g $\pm 4.98 \times 10^2$ and $6.48 \times 10^3 \pm 6.29 \times 10^3$ cfu/g in the
288 FW and AFW digestates, respectively).

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

298 The results show that the studied autoclave treatment effectively reduced all the
299 hygiene indicator concentrations in AFW due to high temperature and pressure, which
300 are widely used for sterilisation. However, the observed increase in concentrations of
301 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 24-34 g/kgTS, total-P between 2.7-6.4 and total-K between 8.6-14.3 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 & Morton, 1999). However, when considering the low NH4-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 NH4-N (4 g/kgFM), which supports its use as a fertiliser in agriculture. Also the hygienic quality, nutrient concentrations (NH4-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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513 Table 1. Source of digestates used for characterisation, hygiene analyses and batch
514 assays. Organic loading rate (OLR) and hydraulic retention time (HRT) of the reactors
515 and supplementation of trace elements (TEs) are shown for time of sampling as well as
516 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

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526 Table 2. Characteristics of food waste (FW) and autoclaved food waste (AFW) as well
527 as FW and AFW digestates. Averages and standard deviations are shown, feed N=3-4,
528 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

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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

538 Table 4. Initial TS, VS and NH4-N (g/kgFM) during batch assays with different inocula
539 (FW, AFW and reference digestates) and food waste (FW) and autoclaved food waste
540 (AFW) as substrates. Residual methane potentials (RMPs) and biochemical methane
541 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
NH4-N initial (g/kg)[a]	3.02	3.09	3.11	1.03	1.05	1.07	1.31	1.35	1.35
NH4-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

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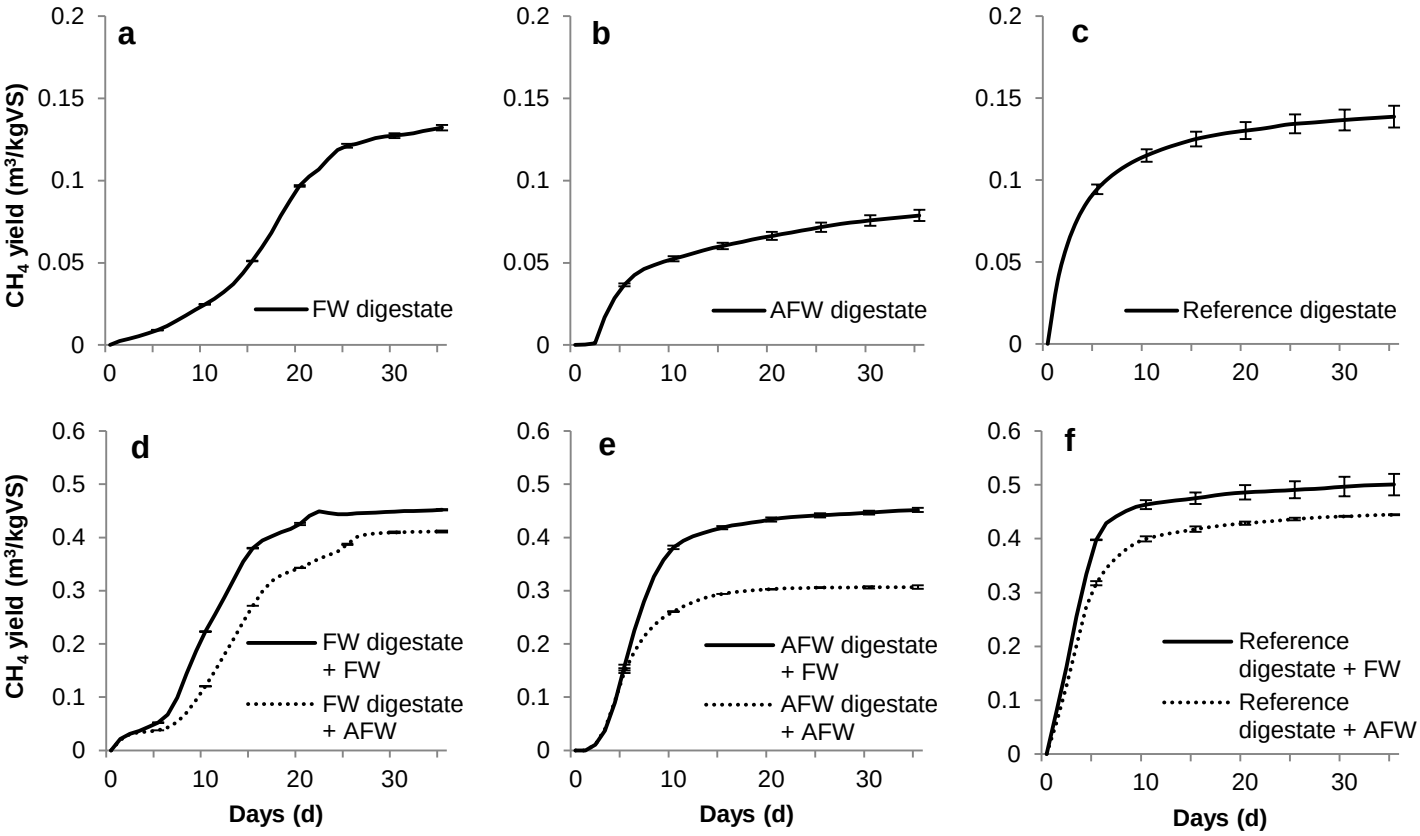
Figure Captions

Figure 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.

Figure 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.

557 Fig.1.



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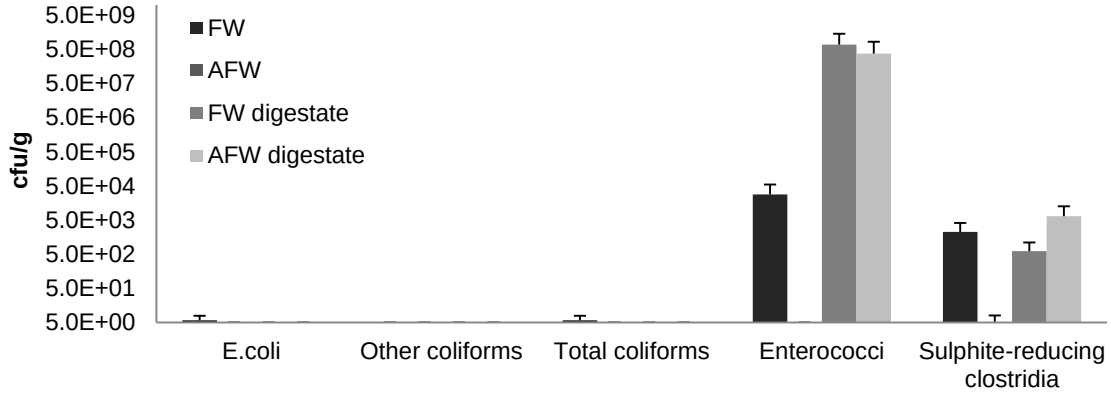
571 Fig. 2.


5.0E+09
5.0E+08
5.0E+07
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5.0E+05
5.0E+04
5.0E+03
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5.0E+00
cfu/g
FW
AFW
FW digestate
AFW digestate
E.coli
Other coliforms
Total coliforms
Enterococci
Sulphite-reducing clostridia

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