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1 **Anaerobic digestion of autoclaved and untreated food waste**

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8 **Abstract**

9 Anaerobic digestion of autoclaved (160 °C, 6.2 bar) and untreated source segregated  
10 food waste (FW) was compared over 473 days in semi-continuously fed mesophilic  
11 reactors with trace elements supplementation, at organic loading rates (OLRs) of 2, 3, 4  
12 and 6 kgVolatile solids(VS)/m<sup>3</sup>d. Methane yields at all OLR were 5-10 % higher for  
13 untreated FW (maximum 0.483 ± 0.013 m<sup>3</sup>CH<sub>4</sub>/kgVS at 3 kgVS/m<sup>3</sup>d) than autoclaved  
14 FW (maximum 0.439 ± 0.020 m<sup>3</sup>CH<sub>4</sub>/kgVS at 4 kgVS/m<sup>3</sup>d). The residual methane  
15 potential of both digestates at all OLRs was less than 0.110 m<sup>3</sup>CH<sub>4</sub>/kgVS, indicating  
16 efficient methanation in all cases. Use of acclimated inoculum allowed very rapid  
17 increases in OLR. Reactors fed on autoclaved FW showed lower ammonium and  
18 hydrogen sulphide concentrations, probably due to reduced protein hydrolysis as a  
19 result of formation of Maillard compounds. In the current study this reduced

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20 biodegradability appears to outweigh any benefit due to thermal hydrolysis of ligno-  
21 cellulosic components.

## 22 **Keywords**

23 Food waste, anaerobic digestion, autoclave treatment, organic loading rate, nitrogen  
24

## 25 **1. Introduction**

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

34 Although co-digestion of FW with sewage sludge and animal manures has been  
35 common practice, treatment of FW alone has often proved difficult (Banks et al. 2008,  
36 Neiva Correia et al. 2008, Zhang et al. 2012). These difficulties have been attributed to  
37 ammonia inhibition resulting from a high protein content (Gallert et al. 1998), and are  
38 often indicated by accumulation of volatile fatty acids (VFA) (Banks et al. 2012). To  
39 achieve stable anaerobic digestion with FW alone, organic loading rates (OLR) are  
40 usually maintained at low values: 2.25 kgVS/m<sup>3</sup>d at a hydraulic retention time (HRT) of  
41 80 days in Banks et al. (2011) and from 1-4 kgVS/m<sup>3</sup>d (HRT 14-30 days) as reported in

42 Cecchi et al. (2003). VFA accumulation at higher OLR has recently been linked to trace  
43 element (TE) deficiencies (Banks et al. 2012). When supplemented with TE successful  
44 FW digestion has been reported at OLRs of 5 kgVS/m<sup>3</sup>d (Banks et al. 2012) and 6.64  
45 kgVS/m<sup>3</sup>d (Zhang and Jahng 2012).

46 Thermal and hydrothermal pre-treatments have been widely studied as a means of  
47 hydrolysing recalcitrant components in a wide range of wastes to make them easier to  
48 degrade (Papadimitriou 2010, Ren et al. 2006, Takashima and Tanaka 2008); these  
49 techniques have also been used as pre-treatments before anaerobic digestion of mixed  
50 biowastes (Lissens et al. 2004, Sawayama et al. 1997). One such hydrothermal  
51 treatment is autoclaving, where water is used as a reagent at increased temperature and  
52 pressure, to hydrolyse and solubilise sugars, starch, proteins and hemicellulose  
53 (Papadimitriou 2010, Ren et al. 2006). Materials pre-treated by autoclaving under  
54 various conditions have shown increased methane production in batch tests: digested  
55 swine slurry autoclaved at 120 °C showed an increase in CH<sub>4</sub> yield of 115 % (Menardo  
56 et al. 2011) and autoclaving of mixed kitchen garbage (175 °C, 40 bar, 1 hour)  
57 increased CH<sub>4</sub> yield by 30 % (Sawayama et al. 1997). Improved methane production has  
58 also been observed in continuously-stirred tank reactors (CSTRs) treating waste  
59 activated sludge (WAS), with 12 % and 25 % increases after autoclaving at 135 °C and  
60 190 °C, respectively (Bougrier et al. 2007).

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

66 carbohydrates (Bougrier et al. 2008, Takashima and Tanaka 2008). Maillard compounds  
67 start to form at temperatures above 100 °C depending on the retention time (Müller  
68 2001, Nursten 2005), while the formation of more complex compounds, such as  
69 acrylamides and other vinylogous compounds, increases at higher temperatures (180 °C,  
70 Stadler et al. 2004).

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

## 76 **2. Materials and methods**

### 77 **2.1. Origin and characterization of FW and inocula**

78 The source segregated domestic FW used in the study was collected from the  
79 South Shropshire Biowaste digestion plant in Ludlow, UK. Biodegradable bags used for  
80 waste collection were removed and the FW material was mixed and divided into two  
81 equal portions. One portion was pre-treated at 160 °C and 6.2 bars in a novel double-  
82 auger autoclave (AeroThermal Group Ltd, UK) that provides improved mixing and  
83 steam penetration; the other portion was left untreated. Both portions were then passed  
84 through a macerating grinder (S52/010 Waste Disposer, IMC Limited, UK), packed into  
85 35-litre plastic boxes (7 untreated and 8 autoclaved), frozen and shipped at - 20 °C to  
86 MTT Agrifood Research, Finland.

87 At MTT the frozen material was chopped into smaller portions corresponding to  
88 amounts required for weekly feeding of the digesters, and these smaller portions were

89 again stored at -20 °C. Each week portions of the autoclaved and untreated FW were  
90 thawed and stored at 4 °C and used as daily feed. The pH, total solids (TS), volatile  
91 solids (VS), ammonium nitrogen (NH<sub>4</sub>-N), total Kjeldahl nitrogen (TKN), soluble  
92 chemical oxygen demand (SCOD) and VFA content was determined for each new box  
93 of feed.

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

## 98 **2.2. Semi-continuous trials**

99 Four 11-litre stainless steel stirred tank reactors (STRs) (Metener Ltd, Finland)  
100 were operated at 37 °C. Stirring (32 rpm) was semi-continuous with 5 seconds on and  
101 60 seconds off. The reactors were fed manually five times a week through an inlet tube  
102 which extended below the digestate surface, and which was also used for digestate  
103 sampling. Digestate overflowed from the reactors by gravity through a u-tube trap to  
104 prevent gas escape. Between days 1-195 hourly gas volume and methane content were  
105 measured using an automatic system in which the produced biogas was collected into a  
106 small (~220 ml) gas storage vessel on top of the reactor. From day 195 onwards, due to  
107 break down of the automated system, gas volume was measured by water displacement  
108 in a volume-calibrated cylindrical gas collector, after which the gas was collected in  
109 aluminium gas bags.

110 Reactors were fed with untreated FW (R1) and autoclaved FW (R3). After 18  
111 days acclimation period with reduced feeding the experiments started at an OLR of 2

112 kgVS/m<sup>3</sup>day, corresponding to HRT of 117 and 94 days for R1 and R3 respectively. On  
113 day 151, after 1.1 (R1) and 1.4 (R3) HRTs, the OLR was raised to 3 kgVS/m<sup>3</sup>day and  
114 after 1.3 (R1) and 1.7 (R3) HRTs to 4 kgVS/m<sup>3</sup>day on day 256 (HRT 78 d and 58 d for  
115 untreated, 63 d and 47 d for autoclaved FW, respectively).

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

129 Grab samples of digestate (about 250 g) were taken every two weeks for analysis  
130 of TS, VS, SCOD, NH<sub>4</sub>-N, TKN, and samples for VFA analysis (about 50 g) were taken  
131 once a week. Digestate pH was measured weekly. Larger volumes of digestate were  
132 collected on days 130 (2 l), 214 (1 l), 287 (1 l) and 321 (1 l). After removal of these  
133 larger samples, daily feeding of the reactors was adjusted to compensate for the reduced  
134 volume until the normal operating level was restored.

### 135 **2.3. Biochemical and residual methane potential assays**

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

### 146 **2.4. Analyses and calculations**

147 TS and VS were determined according to SFS 3008 (Finnish Standard  
148 Association 1990) and NH<sub>4</sub>-N according to McCullough (1967). TKN was analysed by  
149 a standard method (AOAC 1990) using a Foss Kjeltac 2400 Analyzer Unit (Foss  
150 Tecator AB, Höganäs, Sweden), with Cu as a catalyst. For soluble COD analysis FW  
151 samples were diluted 1:10 with distilled water, and agitated for 1 hour. Diluted FW and  
152 raw digestate samples were centrifuged (2493 × g, 15 min) after which the supernatant  
153 was further centrifuged (16168 × g, 10 min) and stored in a freezer, then thawed before  
154 analysis according to SFS 5504 (Finnish Standards Association 2002). pH was  
155 determined using a VWR pH100 pH-analyzer (VWR International). Iron concentration  
156 was analysed according to Luh Huang and Schulte (1985) using inductively coupled

157 plasma emission spectrometry (ICP-OES) (Thermo Jarrel Ash Iris Advantage, Franklin,  
158 USA).

159 Samples for VFA analysis were centrifuged ( $1831 \times g$ , 10 min) and filtered with  
160 Chromafil GF/PET-20/25 filters. Concentrations of acetic, propionic, iso-butyric, n-  
161 butyric, iso-valeric, valeric and caproic acids were determined using a HP 6890 gas  
162 chromatograph with an HP 7683 autosampler (Hewlett-Packard, Little Falls, USA) and  
163 GC ChemStation Rev. B.03.02 software. The GC was fitted with a 10 m x 0.53 mm x 1  
164  $\mu\text{m}$  HP-FFAP capillary column (Agilent Technologies, USA) and a flame ionisation  
165 detector with helium as a carrier gas (9 ml/min). Oven temperatures were 60-78 °C (25  
166 °C/min), isothermal 1 min, 150 °C (7.5 °C/min) and 25 °C/min to 180 °C with 3 min  
167 final time. The injector and detector temperatures were 220 °C and 280 °C,  
168 respectively.

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

174 The reactor was fed for 5 days a week, but the OLR in  $\text{kgVS}/\text{m}^3\text{day}$  is expressed  
175 as the average daily weight of substrate fed to the reactor over a one-week period. HRT  
176 was calculated based on feedstock densities. All biogas and methane yields were  
177 converted to STP conditions (0 °C, 100 kPa) according to the ideal gas law. Methane  
178 yields in the RMP assays were calculated in two ways; by dividing the cumulative  
179 methane production by the 1) VS of the added digestate and 2) by the VS of the feed of

180 the semi-continuous reactors at the time of digestate sampling. The latter enables direct  
181 comparison of the methane yield in the RMP with that in the reactors. Free ammonia  
182 ( $\text{NH}_3\text{-N}$ ) concentrations were calculated according to Anthonisen et al. (1976):

$$183 \quad \text{NH}_3\text{-N} = (\text{NH}_4\text{-N} \times 10^{\text{pH}}) / ((K_b / K_w) + 10^{\text{pH}}), \quad (1)$$

184 where  $K_b$  is the ammonia ionisation constant and  $K_w$  the ionisation constant of water at  
185 37 °C.

### 186 **3. Results and discussion**

#### 187 **3.1. Material characterization**

188 The autoclaved FW appeared much darker than the untreated FW and had a  
189 pleasant caramel odour. TS and VS in the autoclaved FW were both about 15 % lower  
190 than in the untreated FW due to dilution by steam condensation during the autoclave  
191 treatment (Table 1). TKN on fresh matter basis was lower in the autoclaved FW ( $6.8 \pm$   
192  $0.3 \text{ g N/kg}$ ) than in untreated FW ( $7.4 \pm 0.3 \text{ g N/kg}$ ). The autoclaved FW had about 22  
193 % higher  $\text{NH}_4\text{-N}$  and 16 % higher SCOD, indicating that autoclaving had solubilised  
194 some organic nitrogen and carbon components. Total VFA concentrations were lower in  
195 the autoclaved material ( $2.2 \pm 0.2 \text{ g/l}$ ) than in the untreated FW ( $3.1 \pm 0.6 \text{ g/l}$ )  
196 suggesting either that some VFA had volatilised during or after autoclaving, or that  
197 some acidification of the untreated material had occurred.

198 Changes in the chemical composition of materials during autoclave treatment are  
199 dependent on the temperature as well as the materials used. In this study autoclaving  
200 conditions of 6.2 bars and 160 °C were used. Increased concentrations of  $\text{NH}_4\text{-N}$  and  
201 solubilisation of carbohydrates have previously been reported after autoclave treatment

202 of dewatered sewage sludge (175 °C, 20 bar), with an increase from 2.6 to 3.2 g NH<sub>4</sub>-  
203 N/l (Inoue et al. 1996); temperatures above 90 °C have also been reported to increase  
204 ammonia concentrations from 0.35 gN/l to 0.7 gN/l in WAS (Bougrier et al. 2008).

### 205 **3.2. BMP assay**

206 The 35-day BMP value for untreated FW was  $0.501 \pm 0.020$  m<sup>3</sup>CH<sub>4</sub>/kgVS, while  
207 that for autoclaved FW was  $0.445 \pm 0.001$  m<sup>3</sup>CH<sub>4</sub>/kgVS (Figure 1, Table 1). The lower  
208 methane yield of the autoclaved FW could be explained by Maillard reactions. Support  
209 for the occurrence of these is given by the darkening in colour of the autoclaved FW and  
210 the caramelised odour, while the increase in SCOD provides evidence of increased  
211 solubilisation of carbon compounds. Similar phenomena have also been observed with  
212 autoclaved WAS (Bougrier et al. 2008) and municipal solid waste (Takashima and  
213 Tanaka 2008). In other studies higher methane yields have been reported after similar  
214 thermal treatments (Lissens et al. 2004), but this can be attributed to the improved  
215 availability of the ligno-cellulosic materials; and when these form a large proportion of  
216 the waste the resulting increase may far exceed any decrease due to Maillard  
217 compounds. In contrast where ligno-cellulosic content is low, as in this type of food  
218 waste (Zhang et al. 2012) reductions in methane yield may result.

### 219 **3.3. Semi-continuous operation**

#### 220 *3.3.1. Effect of loading rate on methane yields*

221 Process parameters from the whole experimental period (days 1-473) are shown in  
222 Figure 2 and detailed results from the last four weeks of stable operation at each OLR  
223 are presented in Table 2. Operation was considered stable when variations were < 0.2  
224 units in pH, < 90 mg/l in VFA and < 1.8 % in CH<sub>4</sub>.

225 Throughout the experimental period specific methane yields were 5-10 % higher  
226 for untreated FW than for autoclaved FW. The methane yields at OLR 2 kgVS/m<sup>3</sup>day  
227 were on average  $0.443 \pm 0.038$  and  $0.373 \pm 0.037$  m<sup>3</sup>CH<sub>4</sub>/kgVS for untreated (R1) and  
228 autoclaved FW (R3), respectively. The highest yield for untreated FW was observed at  
229 OLR 3 kgVS/m<sup>3</sup>day ( $0.483 \pm 0.013$  m<sup>3</sup>CH<sub>4</sub>/kgVS) while autoclaved FW produced the  
230 highest yield at OLR 4 kgVS/m<sup>3</sup>day ( $0.439 \pm 0.020$  m<sup>3</sup>CH<sub>4</sub>/kgVS). When the OLR was  
231 further increased to 6 kgVS/m<sup>3</sup>day methane yields decreased by 12 % and 11 % in  
232 untreated FW and autoclaved FW, respectively. The specific methane yield for  
233 autoclaved FW was lower at OLR 2 kgVS/m<sup>3</sup>day than at higher OLRs, which could  
234 possibly indicate some acclimatisation. This was not seen in the untreated FW where  
235 the lowest specific methane yield occurred at OLR 6 kgVS/m<sup>3</sup>day, which could indicate  
236 retarded hydrolysis as no increased SCOD nor VFA was detected. At OLR 6  
237 kgVS/m<sup>3</sup>day the difference in methane yields between the parallel (R2 and R4) and  
238 original (R1 and R3) reactors was < 7 % (Table 2).

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

248 As far as is known, this is the first study to report anaerobic digestion of  
249 autoclaved food waste in a semi-continuously fed system. Methane yields of  $0.483 \pm$   
250  $0.013$  and  $0.423 \pm 0.002$   $\text{m}^3\text{CH}_4/\text{kgVS}$  for the untreated and autoclaved FW at OLR 3  
251  $\text{kgVS}/\text{m}^3\text{day}$  are in good agreement with previous studies, where a full-scale digester  
252 fed on the same type of source-segregated household food waste at an average OLR of  
253  $2.5$   $\text{kgVS}/\text{m}^3\text{day}$  yielded  $0.402$   $\text{m}^3\text{CH}_4/\text{kgVS}$  (Banks et al. 2011). Earlier pilot-scale  
254 studies gave an average of  $0.390$   $\text{m}^3\text{CH}_4/\text{kgVS}$ , but using a different source of source-  
255 segregated domestic food waste at higher OLR ( $3.5$  to  $4$   $\text{kgVS}/\text{m}^3\text{day}$ ), and without TE  
256 supplementation (Banks et al. 2008). Laboratory-scale FW digestion with TE  
257 supplementation was reported to yield  $0.352$ - $0.439$   $\text{m}^3\text{CH}_4/\text{kgVS}$  at an OLR of  $6.64$   
258  $\text{kgVS}/\text{m}^3\text{day}$  by Zhang and Jahng (2012); while in the study by Banks et al. (2012) the  
259 methane yield for TE supplemented FW was  $0.435$   $\text{m}^3\text{CH}_4/\text{kgVS}$ .

260 The maximum methane yields for untreated and autoclaved FW in the semi-  
261 continuous trials were  $0.483 \pm 0.013$  and  $0.439 \pm 0.020$   $\text{m}^3\text{CH}_4/\text{kgVS}$  respectively.  
262 These were slightly lower than the BMP values in each case. The results therefore  
263 strongly indicate that even after long periods of operation no significant acclimatisation  
264 that could improve the biodegradability of compounds produced in the autoclaving  
265 process had taken place.

266 With mixed biowastes, the benefits of increased biogas production due to  
267 improved degradation of ligno-cellulosic materials may outweigh any losses in  
268 biodegradability as a result of formation of recalcitrant compounds during thermal  
269 treatment. FW, however, has a relatively low ligno-cellulosic fibre content compared to  
270 other municipal biowaste components (e.g. garden or yard waste, paper and card), and  
271 in the present study the net effect of treatment was a reduction in specific methane yield.

272 This balance may however change with different autoclaving conditions, and in  
273 particular a lowering of temperature may produce more favourable results.

### 274 3.3.2. *Digestion parameters*

275 Results for pH, VFA, TS, VS, SCOD, NH<sub>4</sub>-N, TKN are presented in Table 2 and  
276 Figure 3. pH in the untreated FW reactor remained around 7.8 throughout the  
277 experimental period, while with autoclaved FW the pH decreased from pH 7.6 at OLR 2  
278 kgVS/m<sup>3</sup>day to 7.3 at OLR 6 kgVS/m<sup>3</sup>day.

279 At an OLR of 2 kgVS/m<sup>3</sup>day, total VFA concentration in both reactors remained  
280 under 250 mg/l. When the OLR was increased to 3 kgVS/m<sup>3</sup>day, VFA in the untreated  
281 FW reactor increased to 2400 mg/l by day 153, and consisted mainly of acetic (about 85  
282 %) and propionic acids (about 10 %). In the autoclaved FW reactor VFA concentration  
283 showed smaller increases with peaks of 1500 mg/l on day 139 (consisting 98 % of  
284 acetic acid) and 910 mg/l on day 160 (27 % acetic acid and 65 % propionic acid). The  
285 relatively large samples (2 litres) taken from the reactors on day 130 could have  
286 contributed to these increases in VFA concentration, but similar removals of digestate at  
287 later stages in the experimental run did not have this effect. VFA concentrations reduced  
288 to under 200 mg/l in both reactors by day 214, shortly after the introduction of trace  
289 element additions of selenium and cobalt on day 179 and full TE supplementation on  
290 day 199. This behaviour is consistent with previous reports of responses to TE  
291 supplementation where the VFA increase was linked with the loss of electron transfer  
292 interspecies during digestion (Banks et al. 2012).

293 TS, VS and TKN contents in both reactors gradually increased during the  
294 experimental period, with TS increasing from under 70 to over 80 g/kg. Despite the

295 lower feedstock solids concentration, the solids content in the autoclaved FW reactor  
296 was slightly higher than in the untreated FW up to the end of OLR 4 kgVS/m<sup>3</sup>day. After  
297 OLR was increased to 6 kgVS/m<sup>3</sup>day there was an increase in solids concentrations in  
298 the untreated FW reactor, which was not apparent with the autoclaved FW. The initial  
299 TKN concentration in both reactors was 4.9 g N/kg and showed a similar increase to ~8  
300 g N/kg by around day 200. TKN in the untreated FW reactor continued to increase until  
301 around day 300 at which point it stabilised at ~9 g N/kg, whereas for the autoclaved FW  
302 it remained at ~8 g N/kg. The differences in TKN reflected the differences in feedstock  
303 concentrations. The increases in solids content were most likely associated with the  
304 increase in loading, although it is possible that some accumulation was due to  
305 stratification despite the intermittent mixing, as surplus digestate was discharged from  
306 an overflow at the top of the reactor. Mass balance calculations affirmed, in the  
307 beginning of OLR 4 kgVS/m<sup>3</sup>day, that accumulation of TKN was taking place.

308       The SCOD concentration in both reactors increased from around 10 g/l to over 20  
309 g/l during the first 300 days of operation, then stabilised in the autoclaved FW reactor.  
310 In the untreated FW reactor the SCOD increased sharply to ~36 g/l for over 50 days  
311 then decreased equally sharply in the end of the run: these variations did not correspond  
312 to changes in OLR and were not accomplished with changes in methane yield nor  
313 digestate VFA. Total VFA concentrations accounted for only 0.5-2 % of the SCOD. A  
314 probable explanation for the general increase in SCOD in both reactors is an increase in  
315 the quantity of soluble microbial products present in the digestate; this phenomenon has  
316 previously been observed with solid substrates and at long retention times (Kuo et al.  
317 1996, Rinçon et al. 2012).

318 *3.3.3. Ammonium and ammonia*

319           NH<sub>4</sub>-N concentration in the untreated FW reactor increased during the first ~170  
320 days from 2.4 (inoculum) to 4 g/kg and then showed a very gradual decrease to around  
321 3 g/kg by the end of the experimental run. This decrease could be associated with the  
322 increase in microbial biomass (Lindorfer et al. 2011) or in soluble microbial products  
323 caused by the increasing OLR. In the autoclaved FW reactor, however, NH<sub>4</sub>-N  
324 decreased from 2.4 to about 1.2 g/kg by the end of the experimental period. The low  
325 NH<sub>4</sub>-N concentrations in the autoclaved FW reactor were probably mainly due to the  
326 effect of autoclaving and the formation of Maillard compounds from the reaction of  
327 proteins with carbohydrates (Bougrier et al. 2007, 2008). Free ammonia concentrations  
328 in the reactors were calculated, but NH<sub>3</sub> remained below 0.30 g/kg in untreated FW and  
329 below 0.10 g/kg in the autoclaved FW reactor.

330           The pH value in the untreated FW reactor rose to around 7.8 by day 55 and  
331 remained relatively stable until the OLR was raised to 6 kgVS/m<sup>3</sup>day, at which point it  
332 fell very slightly. In the autoclaved reactor after a slight initial rise pH decreased during  
333 the experimental run to a final value of around 7.3. These pH values reflect the relative  
334 NH<sub>4</sub>-N concentrations in each case, as NH<sub>4</sub>-N provides buffering capacity (Procházka et  
335 al. 2012). High NH<sub>4</sub>-N concentration can also inhibit the digestion process, but this is  
336 greatly dependent on the feedstock materials and acclimation times (Chen et al. 2008,  
337 Procházka et al. 2012). In the present study, after TE supplementation was introduced,  
338 there was no evidence of the VFA accumulation that is often associated with ammonia  
339 toxicity, and the free ammonia concentrations were similar to those previously observed  
340 in FW digestion (Zhang et al. 2012).

341 *3.3.4. Gas composition*

342 The biogas methane content in both autoclaved and untreated FW digesters was  
343 similar and ranged between 55-63 % during the experiment, with an average of around  
344 58% (Table 2, Figure 2). It did not appear to be affected by changes in applied OLR. In  
345 contrast, in a study by Zhang and Jahng (2012) on FW digestion the methane content  
346 was found to decrease from 53 % to 48 % as the OLR was gradually increased from  
347 2.19 to 6.64 kgVS/m<sup>3</sup>day.

348 Hydrogen sulphide concentration was monitored between days 166-313 while the  
349 reactors were operated at OLR 3 and 4 kgVS/m<sup>3</sup>day (Figure 4). H<sub>2</sub>S concentrations at  
350 OLR 3 kgVS/m<sup>3</sup>day were < 100 ppm in the untreated FW reactor and < 75 ppm in the  
351 autoclaved FW reactor. Shortly before the OLR was increased to 4 kgVS/m<sup>3</sup>day the H<sub>2</sub>S  
352 concentration in the untreated FW reactor began to increase, and reached 480 ppm by  
353 day 314 at which point monitoring ceased; while in the autoclaved FW reactor H<sub>2</sub>S  
354 content remained < 60 ppm. H<sub>2</sub>S was also monitored at the OLR of 6 kgVS/m<sup>3</sup>day  
355 (days 448-473) and concentrations were 751 ± 182 ppm in the untreated FW reactors  
356 (R1 and 2) compared to 63 ± 4 ppm in the autoclaved FW reactors (R3, R4).

357 In the autoclaved FW reactors H<sub>2</sub>S concentrations remained low, probably due to  
358 the effect of autoclaving on proteins in the food waste, which may have reduced the  
359 availability of sulphur. The low H<sub>2</sub>S concentration could also be due in part to  
360 precipitation through the formation of iron sulphides. The iron content in the autoclaved  
361 FW was 170 times higher than in the untreated FW (Table 1), possibly due to metal  
362 contamination from the autoclaving apparatus. O'Flaherty et al. (1998) showed that  
363 sulphate-reducing bacteria (SRBs) have an optimum pH slightly higher than that of  
364 methanogenic archaea, and hence the higher pH in the untreated FW reactors may have

365 favoured the growth of SRBs causing increased H<sub>2</sub>S concentrations. Decreasing HRT  
366 will also give SRB an additional competitive advantage.

### 367 **3.4. Residual methane potential assays**

368 50-day RMP values were determined at the end of each period of reactor  
369 operation at OLRs 2, 4 and 6 kgVS/m<sup>3</sup>day (Table 3). The RMPs increased with the  
370 increasing OLRs and decreasing HRTs from  $0.069 \pm 0.005$  m<sup>3</sup>CH<sub>4</sub>/kgVS to  $0.105 \pm$   
371  $0.002$  m<sup>3</sup>CH<sub>4</sub>/kgVS with the untreated FW and from  $0.063 \pm 0.002$  m<sup>3</sup>CH<sub>4</sub>/kgVS to  
372  $0.095 \pm 0.012$  m<sup>3</sup>CH<sub>4</sub>/kgVS with the autoclaved FW (OLR 2 to 6 kgVS/m<sup>3</sup>day).  
373 However, RMPs after operation with OLR 4 kgVS/m<sup>3</sup>day were 6 and 10 % lower in  
374 untreated and autoclaved FW compared to OLR 2 kgVS/m<sup>3</sup>day reflecting the highest  
375 CH<sub>4</sub> yields obtained with OLRs 3 and 4 kgVS/m<sup>3</sup>day in STRs. Also few days longer  
376 storage time might have affected the RMPs after OLR 4 kgVS/m<sup>3</sup>day allowing  
377 materials to slightly degrade before the RMP start.

378 Overall, when results were calculated per VS of FWs fed to the STRs, RMP<sub>original</sub>  
379 increased total methane yield of the semi-continuous reactors by 2.9-4.7 % with the  
380 untreated FW and by 4.3-5.2 % with the autoclaved FW (Table 3). The calculated total  
381 methane yield with the untreated FW was, after OLRs 2, 4 and 6 kgVS/m<sup>3</sup>day, 3.6-12.6  
382 % lower than the BMP value ( $0.501$  m<sup>3</sup>CH<sub>4</sub>/kgVS) being closest after OLR 4  
383 kgVS/m<sup>3</sup>day and thus reflecting the specific yields in STRs. Autoclaved FW showed  
384 similar STR reflecting behavior but after OLR 4 kgVS/m<sup>3</sup>day the calculated total  
385 methane yield was 3.1 % higher than the BMP value ( $0.445$  m<sup>3</sup>CH<sub>4</sub>/kgVS). The VS  
386 removals were not cohesive with the calculated total methane yields, which could partly  
387 be explained with deviations between samples. The results suggest that in both materials

388 there was still a small part of biodegradable material after semi-continuous reactors and  
389 the amount increased with the increasing OLRs and decreasing HRTs.

#### 390 **4. Conclusions**

391 Stable digestion of untreated and autoclaved FW was possible in TE-  
392 supplemented mesophilic reactors at OLRs up to 6 kgVS/m<sup>3</sup>d, with yields of 0.435 and  
393 0.393 m<sup>3</sup>CH<sub>4</sub>/kgVS, respectively. Using an acclimated inoculum allowed rapid  
394 increases in OLR without process disturbance. Untreated FW showed a higher specific  
395 methane yield than autoclaved FW at all OLRs and in batch assays. This difference may  
396 be due to the formation of Maillard compounds, with the resulting reduction in  
397 biodegradability apparently outweighing any benefits from thermal hydrolysis of ligo-  
398 cellulosic components under the autoclaving conditions used. Biogas H<sub>2</sub>S  
399 concentrations were much lower in reactors treating autoclaved FW.

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501

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

	Control FW	Autoclaved FW	Inoculum
pH	4.96 ± 0.16	5.01 ± 0.12	N/A
TS (g/kg)	247.5 ± 4.7	210.9 ± 18.6	77.3
VS (g/kg)	229.9 ± 4.5	194.6 ± 17.6	43.1
VS/TS (%)	92.9	92.3	55.8
SCOD (g/l)	98.2 ± 6.5	117.5 ± 10.3	11.9
TVFA (g/l)	3.1 ± 0.6	2.2 ± 0.2	2.4
TKN (g/kg)	7.4 ± 0.3	6.8 ± 0.3	4.9
NH <sub>4</sub> -N (g/kg)	0.32 ± 0.12	0.41 ± 0.10	2.4
Fe (g/kg <sub>TS</sub> )	0.13 ± 0.01	22.73 ± 12.54	N/A
SMP (m <sup>3</sup> CH <sub>4</sub> /kg <sub>VS</sub> )	0.501 ± 0.020	0.445 ± 0.001	N/A
SMP (m <sup>3</sup> CH <sub>4</sub> /kg <sub>TS</sub> )	0.462 ± 0.019	0.408 ± 0.001	N/A
SMP (m <sup>3</sup> CH <sub>4</sub> /kg <sub>FM</sub> )	0.112 ± 0.005	0.084 ± 0.0001	N/A
Density (kg/l)	1.064 ± 0.0042	1.063 ± 0.0002	N/A

N=24 for pH, N=8 for TS, VS, SCOD, TVFA, TKN, NH<sub>4</sub>-N, N=2 for specific methane potentials (SMPs) and Fe, N=3 for density

N/A, not available

503

Table 2. Reactor characteristics during the last 4 weeks of each organic loading rate (OLR, kgVS/m<sup>3</sup>d) periods.

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

N/A, not available

N=2-5, for pH N=15

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

OLR	Reactor	RMP (m <sup>3</sup> /kgVS)	RMP <sub>original</sub> (m <sup>3</sup> /kgVS <sub>feed</sub> ) <sup>a</sup>	Total CH <sub>4</sub> yield in STR+RMP (m <sup>3</sup> /kgVS <sub>feed</sub> ) <sup>a</sup>	VS removal in STR+RMP (%)
2	R1	0.069 ± 0.005	0.013 ± 0.0009	0.456	85.1
	R3	0.063 ± 0.002	0.017 ± 0.0006	0.390	75.3
4	R1	0.065 ± 0.001	0.017 ± 0.0004	0.482	80.9
	R3	0.057 ± 0.002	0.020 ± 0.0006	0.459	67.4
6	R1	0.105 ± 0.002	0.032 ± 0.0005	0.437	76.9
	R3	0.095 ± 0.012	0.034 ± 0.0045	0.427	69.3

<sup>a</sup> Results calculated according to VS fed to STRs

N=2-3

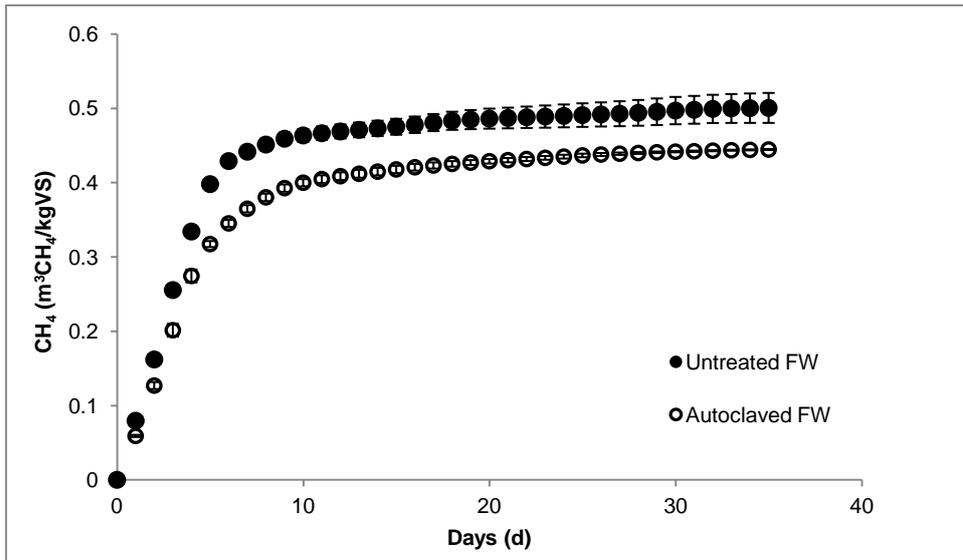


Fig. 1. Biochemical methane potential (BMP) and standard deviation of untreated and autoclaved food waste (FW) in 35-day assays.

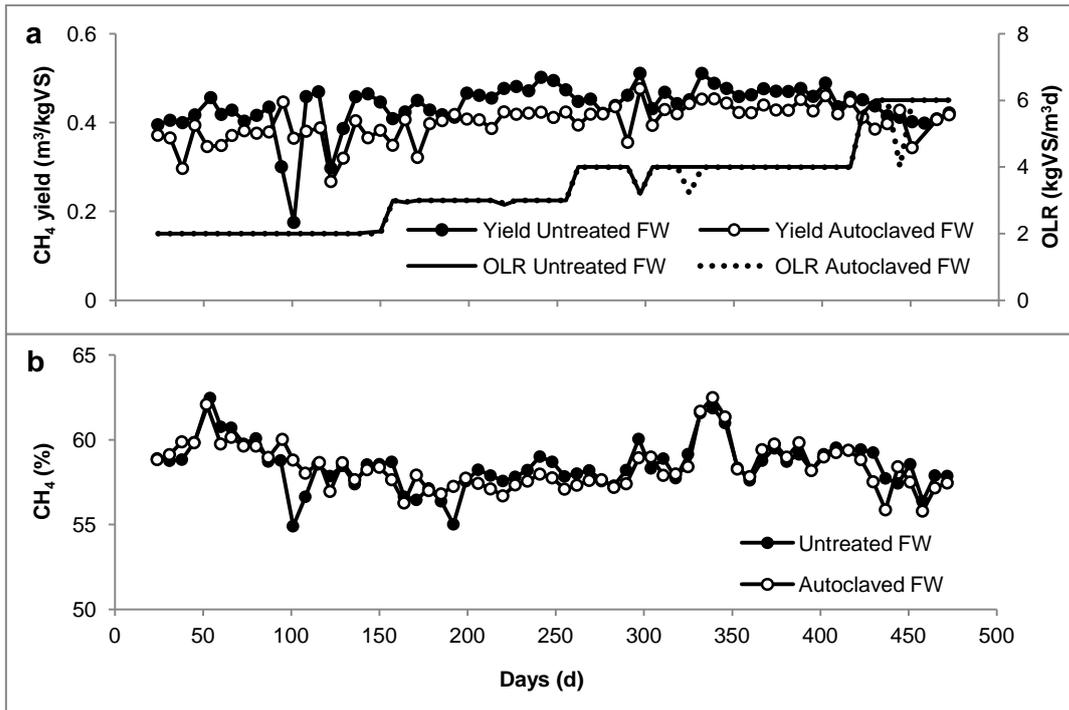


Fig. 2. Methane yields and contents in reactors treating untreated food waste (FW) and autoclaved FW during the semi-continuous operation.

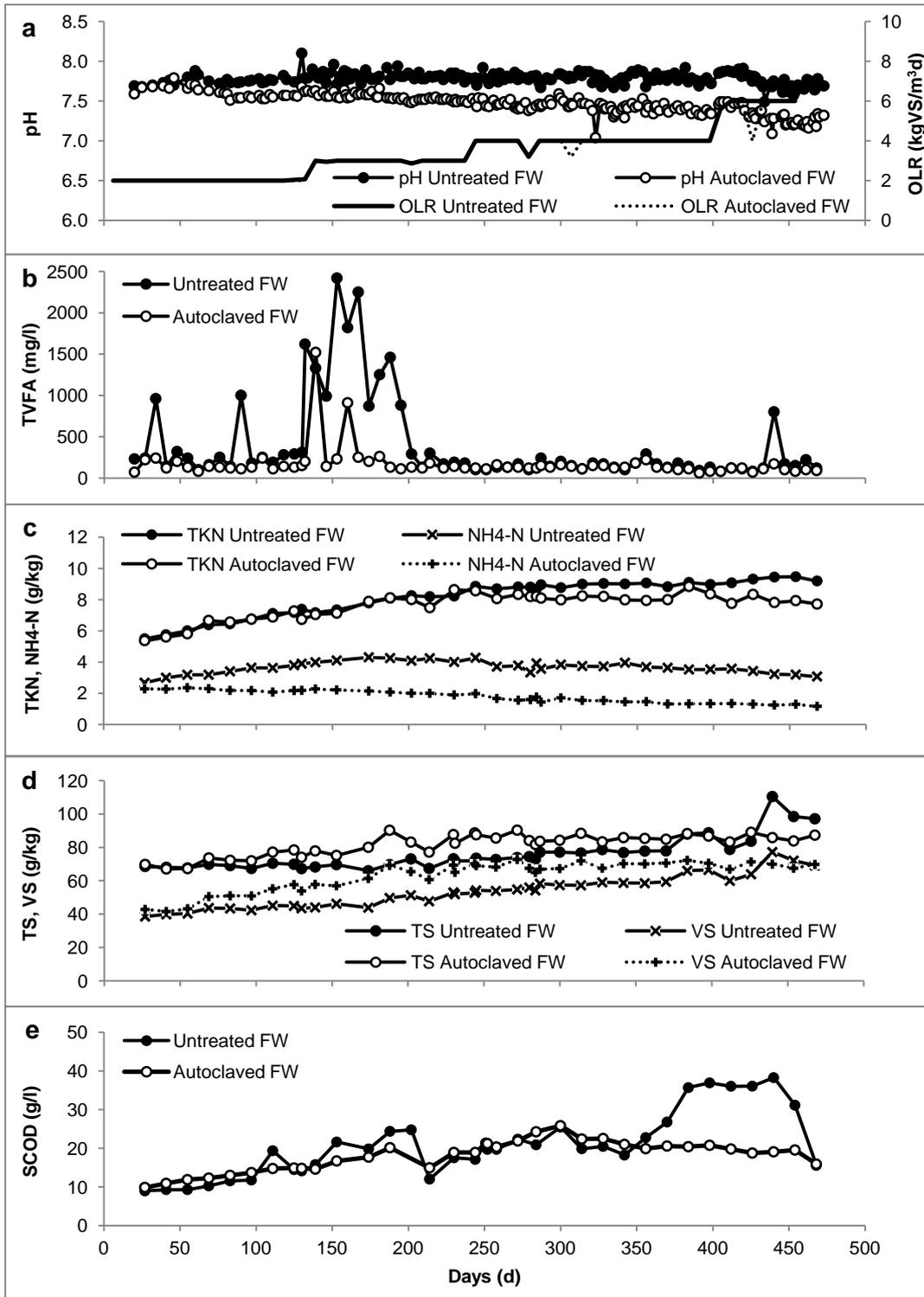


Fig. 3. Chemical characteristics (pH, TVFA, TKN, NH<sub>4</sub>-N, TS, VS, SCOD) of untreated food waste (FW) and autoclaved FW reactor contents during the semi-continuous operation.

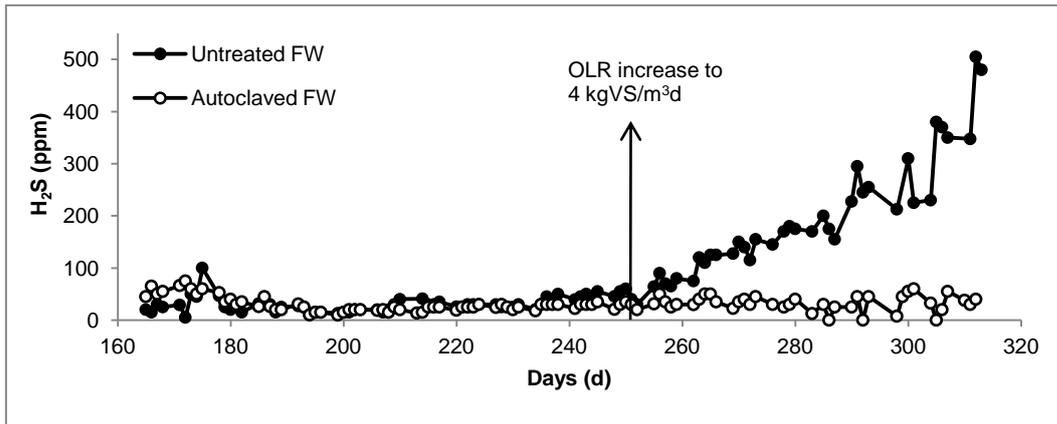


Fig. 4. H<sub>2</sub>S contents in reactors treating untreated food waste (FW) and autoclaved FW during days 166-314.