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Additional Information

1 **Potential use of the organic fraction of municipal solid waste in anaerobic co-**
2 **digestion with wastewater in submerged anaerobic membrane technology.**

3
4 **P. Moñino ^a, E. Jiménez ^b, R. Barat ^a, D. Aguado ^{a*}, A. Seco ^b, J. Ferrer ^a**

5
6 ^a Institut Universitari d'Investigació d'Enginyeria de l'Aigua i Medi Ambient, IIAMA, Universitat
7 Politècnica de València, Camí de Vera, s/n, 46022 València, Spain (e-mail: *patmoiam@upv.es*;
8 *rababa@dihma.upv.es*; *daaggar@hma.upv.es*; *jferrer@hma.upv.es*)

9 ^b Departament d'Enginyeria Química, Escola Tècnica Superior d'Enginyeria, Universitat de València,
10 Doctor Moliner 50, 46100 Burjassot, València, Spain (e-mail: *Emerita.Jimenez@uv.es*;
11 *aurora.seco@uv.es*)

12 * Corresponding author

13
14 **Abstract**

15 Food waste was characterized for its potential use as substrate for anaerobic co-digestion in a
16 submerged anaerobic membrane bioreactor pilot plant that treats urban wastewater (WW). 90% of
17 the particles had sizes under 0.5 mm after grinding the food waste in a commercial food waste
18 disposer. COD, nitrogen and phosphorus concentrations were 100, 2 and 20 times higher in food
19 waste than their average concentrations in WW, but the relative flow contribution of both streams
20 made COD the only pollutant that increased significantly when both substrates were mixed. As
21 sulphate concentration in food waste was in the same range as WW, co-digestion of both substrates
22 would increase the COD/SO₄-S ratio and favour methanogenic activity in anaerobic treatments.
23 The average methane potential of the food waste was 421±15 mL CH₄·g⁻¹ VS, achieving 73%
24 anaerobic biodegradability. The anaerobic co-digestion of food waste with WW is expected to
25 increase methane production 2.9-fold. The settleable solids tests and the particle size distribution
26 analyses confirmed that both treatment lines of a conventional WWTP (water and sludge lines)
27 would be clearly impacted by the incorporation of food waste into its influent. Anaerobic processes
28 are therefore preferred over their aerobic counterparts due to their ability to valorise the high COD
29 content to produce biogas (a renewable energy) instead of increasing the energetic costs associated
30 with the aeration process for aerobic COD oxidation.

31
32 **Keywords:** AnMBR, characterization, co-digestion, food waste, methane production, resource recovery.

33
34 **1. Introduction**

35
36 Wastewater (WW) and municipal solid waste (MSW) from household activities are
37 constantly growing due to the ever expanding worldwide population. To protect the
38 environment, stricter regulations have been imposed requiring innovations and/or
39 optimization of existing treatments. The European Directive 2008/98/CE has encouraged
40 the recovery of resources from household waste and other materials in order to conserve
41 natural resources. The target is that by 2016 EU countries should reduce the quantity of
42 organic waste sent to landfills by 35% of the total amount of biodegradable municipal
43 waste produced in 1995 (1999/31/CE Directive). Untreated biodegradable waste is known
44 to cause many environmental problems, such as contamination of soil, water, and air

1 during collection, transportation and final landfill disposal due to its degradation (Han
2 and Shin, 2004).

3
4 A considerable reduction in the organic matter currently sent to landfills could be
5 achieved by more efficient handling of domestic organic waste. Source control systems
6 constitute an interesting potential solution for increased biogas production as well as
7 nutrient recovery (Kjerstadius *et al.*, 2015). Different technical solutions are available to
8 take advantage of domestic organic waste collection, transportation and treatment for its
9 valorisation. One of these options is to incorporate the organic fraction of municipal solid
10 waste (OFMSW) into the sewage system for joint treatment with urban wastewater in
11 wastewater treatment plants (WWTP) (Kujawa-Roeleveld and Zeeman, 2006). The
12 combined process could lead to improved treatment, savings in MSW transportation,
13 together with the environmental benefits of reduced fossil fuel consumption and landfill
14 volumes. According to these authors, food waste is one of the main constituents of
15 OFMSW.

16
17 The increased influent organic load due to OFMSW incorporation will have different
18 impacts according to the wastewater treatment scheme involved (Evans *et al.*, 2010).
19 Aerobic-based wastewater treatment schemes are energy intensive, produce significant
20 quantities of sludge and do not recover the potential resources available in wastewater
21 (Tchobanoglous *et al.*, 2003). In these systems the higher the organic content of the
22 influent, the higher the energetic cost of aeration (Serralta *et al.*, 2002). In contrast,
23 anaerobic treatment schemes can recover energy by converting organic matter into
24 methane-rich biogas besides other advantages such as low sludge production, fewer
25 pathogens and the possibility of recovering nutrients from wastewater for reuse in
26 agriculture (Fang and Zhang, 2015).

27
28 The low growth rate of the microorganisms involved in anaerobic processes without
29 biomass retention require high sludge retention times (SRT) and thus high reaction
30 volumes, which rules out the use of this technology as a mainstream process. However,
31 the application of membrane technology allows the hydraulic retention time (HRT) to be
32 decoupled from the solids retention time (Giménez *et al.*, 2011), making it possible to
33 operate anaerobic processes at high SRT while keeping reactor volumes low. Submerged

1 MBR technology has been reported as a successful application for anaerobic wastewater
2 treatment (Huang *et al.*, 2011).

3
4 Although a few systems have been investigated for the separate collection of food waste
5 on both experimental and full scales in different countries (Battistoni *et al.*, 2007; Evans
6 *et al.*, 2010; Bernstad *et al.*, 2013) no previous study has focused on the potential benefits
7 of the co-digestion of food waste together with wastewater for valorisation with
8 submerged anaerobic membrane bioreactor technology (AnMBR).

9
10 A study of the feasibility of AnMBR technology for the joint treatment of OFMSW and
11 urban wastewater requires the previous comprehensive characterization of the new
12 wastewater influent (OFMSW+WW) in order to determine whether the chemical,
13 physical and biological characteristics are appropriate for the proposed treatment. These
14 characteristics include particle size distribution, COD concentration, anaerobic
15 biodegradability, nutrient concentration, sulphur concentration, etc. The aim of this study
16 was therefore to thoroughly characterize this substrate for possible future co-digestion
17 with urban wastewater using AnMBR technology, to make a preliminary assessment on
18 the fate of the OFMSW within the treatment scheme based on the characterization, and
19 finally to estimate biogas production of the OFMSW through anaerobic co-digestion with
20 wastewater.

21 22 **2. Materials and Methods**

23 **2.1 Source of substrates**

24 The OFMSW used in this study were leftovers from a number of restaurants on the
25 campus of the Universitat Politècnica de València. The restaurants provided the OFMSW
26 source separated from other waste. The substrate was weighted and stored in bags at 4°C
27 the day prior to experimental use. The study was carried out during the academic year,
28 from October 2012 to May 2013. The occurrence of the different food waste components
29 was: rice (which appeared in 88% of the samples), fruit remains and peel (80%), potatoes
30 (fried, baked, in omelettes) (68%), bread (64%), pasta (56%), seafood (52%), cooked
31 vegetables (44%), chicken (32%), salads (20%), fish (16%), pork chops (8%) and beef
32 steak (8%).

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2.2 Sample pre-treatment

An experimental device was constructed to simulate a household OFMSW grinder and consisted of a structure with a kitchen sink fitted with a commercial food waste disposer (InSinkErator Evolution 100). This was installed in the Carraixet WWTP (Alboraya, Valencia) next to the existing AnMBR pilot plant.

The OFMSW was manually screened to remove materials (e.g. shells cutlery and other foreign objects present in the leftovers) that could negatively affect the disposer operation. Since the wastewater influent of the existing AnMBR pilot plant is pre-filtered through a 0.5 mm space screen to protect the membranes, the OFMSW was also pre-treated in the same way (i.e. with a 0.5 mm space screen sieve after the grinding process). Ground OFMSW samples were previously pre-treated through a 5 mm space screen sieve to simulate typical WWTP fine screening. Fats and oils were removed by 30-minute aeration and surface scraping.

2.3 Analytical procedures

pH was measured by a portable pH meter (WTW pH315i). Settleable, total (TS), dissolved (DS) and volatile (VS) solids were analysed according to the Standard Methods: 2540-F, B, C, E (APHA, 2012) respectively. Total chemical oxygen demand (COD_t) was measured according to Standard Methods: 5220-B, using a Metrohm 702 SM Titration. Ammonium (NH_4^+-N), nitrite ($NO_2^- -N$), nitrate ($NO_3^- -N$), phosphate ($PO_4^{-3} -P$) and sulphate ($SO_4^{2-} -S$) were determined according to Standard Methods (APHA, 2012) (4500-NH3-G, 4500-NO2-B, 4500-NO3-H, 4500-P-F and 4500-SO4-E, respectively) in a Smartchem 200 automatic analyzer (Westco Scientific Instruments, Westco). Carbonate alkalinity and VFA concentration were determined according to the method proposed by WRC (1992). Total Nitrogen was measured using standard kits (Merck, Darmstadt, Germany, ISO 11905-1) and total phosphorous according to the acid peroxodisulphate digestion method (4500-P-B), which can be found in Standard Methods (APHA, 2012). Biochemical methane potential tests (BMP) were carried out by the Automatic Methane Potential Test System (AMPTS) [Bioprocess Control, Sweden]. Particle size distribution was measured by a laser diffraction technique on a Mastersizer 2000E [Malvern Instruments].

1 Due to the heterogeneity of the OFMSW samples in the first stage of the characterization,
2 some practical issues were considered to improve the representativeness of the results:
3 (1) The presence of some relatively large particles after grinding hampered the collection
4 of representative samples, due to the small volume required to determine total parameters
5 (COD_T , N_T and P_T). To ensure that the parameters were determined from homogeneous
6 samples, the samples were ground again in a kitchen blender in the laboratory. (2) To
7 speed up the determination of the soluble fraction, prior to 0.45 μm filtration, samples
8 were centrifuged at 9600 rpm for 8 minutes, sieved through a 0.5 mm and filtered under
9 vacuum through 1.2 μm (3) Suspended solids were determined using two different
10 approaches to verify the consistency of the results: the APHA(2012) protocol and as the
11 difference between total and dissolved solids.

12

13 **2.4 Biochemical methane potential tests**

14 To determine the Biochemical Methane Potential (BMP) of OFMSW in an anaerobic
15 treatment system, bench-scale experiments were carried out by the Automatic Methane
16 Potential Test System (AMPTS) [Bioprocess Control, Sweden]. These experiments were
17 performed in duplicate for each sample and blank in batch reactors of 500 mL capacity
18 each with a working liquid volume of 400 mL and 100 mL of head space, hermetically
19 sealed to simulate the anaerobic degradation of the OFMSW at a constant temperature of
20 35°C. No nutrient solution was added in these experiments. The pH was measured in all
21 batch reactors before the test started and at the end of the test to confirm that the reactors
22 were not acidified. When preparing a sample, a blank was also prepared to determine the
23 methane production from the inoculum. This methane production is subtracted from the
24 total methane production of the sample to determine net biogas production.

25

26 Six experiments were performed (see Table 2 in Results and Discussion) at low S/I ratio
27 to avoid possible inhibitory effects such as the accumulation of volatile fatty acids,,
28 varying two parameters: *Inoculum and OFMSW particle size*. Three different sludges
29 were used as inoculum: from a WWTP mesophilic anaerobic digester, from an AnMBR
30 pilot plant treating WW at 28°C, and sludge adapted to the OFMSW: from an AnMBR
31 pilot plant treating WW+OFMSW. The OFMSW particle sizes tested were: OFMSW
32 sieved by a 5mm space screen sieve, OFMSW sieved by 0.5 mm and finally the soluble
33 fraction.

34

1 To obtain the biodegradability of a sample, the experimental methane production is
2 compared with the methane expected, calculated theoretically from its COD
3 concentration (using the methane oxidation reaction and the ideal gas law) which gave
4 350 ml of methane from 1 gram of COD (at 0°C and 1 atm).

5 6 **3. Results and discussion**

7 **3.1 Particle size distribution**

8 Since the AnMBR pilot plant is equipped with a 0.5 mm screen-size rotofilter to protect
9 the membranes, it is important to analyse OFMSW particle size of the sample after
10 grinding to determine the quantity of particles that will be removed during pre-treatment
11 and thus will not be valorised in the anaerobic reactor. Bolzonella *et al.* (2003) made a
12 study of the particle fraction under 0.84 mm (considered as fine particles by Battistoni
13 (1993)), and found that 95% of particles in WW and 50 % of those in OFMSW are fine.
14 However, Bernstad *et al.* (2013) found that OFMSW particles under 1 mm composed
15 over 80% of the total size distribution.

16
17 Figure 1a shows the cumulative frequency plot of particle size distribution of the OFMSW
18 samples. These ten samples were collected in different days covering the experimental
19 period from October 2012 to May 2013. As can be seen, particle size distribution varies
20 appreciably from sample to sample and the average results of a total of ten samples can
21 be summarized as 0.01 ± 0.003 mm for the d_{10} percentile, 0.1057 ± 0.039 mm for d_{50} or
22 median diameter and 0.447 ± 0.148 mm for d_{90} percentile. From these experimental
23 measurements, it could be said that on average only 13% of the particles will be removed
24 in the pre-treatment of a wastewater treatment plant equipped with a 0.5 mm fine
25 screening membrane protector. Therefore, the OFMSW particle size is small enough to
26 ensure that most of the organic matter will pass through the sieving process and will be
27 fed into the anaerobic digester for valorisation.

28
29 **[Figure 1 Near Here]**

30
31 It is also important to know the size of the particles after grinding the OFMSW, as very
32 large particles could increase undesirable sedimentation in the sewage network and
33 settled particles will obviously not reach the WWTP. For comparison purposes, particle
34 size distribution was also analysed in samples from the sewage pipeline which conveys

1 wastewater to the Carraixet WWTP. The samples were collected 100 metres upstream the
2 WWTP intake when flow rate was low (05:00 – 06:00 AM), i.e. in a situation close to the
3 minimum sewage solid particle-carrying capacity. The cumulative frequency plot of these
4 samples is shown in Figure 1b. To enable a direct visual comparison of representative
5 sizes, box-whisker plots of median and percentile 90 in both WW and OFMSW are shown
6 in Figure 2. As can be seen in Figures 1 and 2, the WW particle size is smaller and inter-
7 sample variability is lower than in OFMSW samples. It should be noted that particles
8 collected from the sewage pipeline were being transported by the flow (i.e. they did not
9 settle in the pipeline) and their average measured density was $1965.58 \text{ kg m}^{-3}$.

10
11 **[Figure 2 Near Here]**

12
13 Considering a WW sewer pipeline particle sample size of 0.0398 mm (average of the d_{50}
14 from the collected samples), the terminal settling velocity according to Stokes' law gives
15 $0.732 \cdot 10^{-3} \text{ m/s}$ (at 15°C). Under these conditions, the flow regime surrounding the particle
16 is laminar (Reynolds number 0.025). The average d_{50} of all the OFMSW samples is
17 0.1057 mm (i.e. more than 2.6 times larger than the sewage particles) with a measured
18 density of $1116.65 \text{ kg m}^{-3}$. The terminal settling velocity of such a particle according to
19 the Stokes' law is $0.628 \cdot 10^{-3} \text{ m/s}$ (at 15°C), the flow regime around the particle also being
20 laminar (Reynolds number 0.058). Therefore, despite the larger size of the OFMSW
21 particles, the settling velocity is similar or even lower than the WW particles which reach
22 the WWTP at the minimum flow rate. This indicates that the OFMSW particles from
23 households could also be conveyed within the wastewater flow and reach the WWTP.
24 However, this is just a preliminary assessment and as particle transport is quite a complex
25 issue, in which many factors play a role, such as hydrodynamic conditions, density,
26 viscosity, shape of particles, etc., a thorough assessment of particle behaviour in the
27 sewage pipelines is relevant and requires further experimental work.

28 29 **3.2 Chemical characterization of the OFMSW**

30 Table 1 shows the results of the chemical characterization of the OFMSW and the WW
31 performed in this study. Due to the observed variability in the water volumes used during
32 the OFMSW grinding process, these results were normalized by Eq.(1) to the average
33 water volume (4 L kg^{-1}) to enable direct comparison of all the samples. Characterization

of the wastewater fed into the AnMBR pilot plant, which received only WW as influent (Giménez *et al.*, 2014), is also included in Table 1 to allow direct comparison with the OFMSW characterization results.

$$C_{normalized} = \frac{V_{used} * C_{obtained}}{V_{normalized}} \text{ (Eq. 1)}$$

where:

$C_{normalized}$ is the normalized concentration; V_{used} is the volume used in the grinding process in each case; $C_{obtained}$ is the concentration obtained experimentally; and $V_{normalized}$ is the normalized volume, which corresponds to 4 L per kg of ground OFMSW.

The water volume in the grinding process varied from 2.8 to 5.8 L per kg of ground OFMSW. These values are relatively low in comparison with recent studies, which fall in the range 7.2 L kg⁻¹ (Käppalaförbundet and SÖRAB, 2009) to 15.6 L kg⁻¹ (Marashlian and El-Fadel, 2005), with an average value rounding 12 L kg⁻¹ (Bernstad *et al.*, 2013). The lower water usage in the present study is possibly a consequence of the experimental procedure in which all the daily OFMSW was ground in one go, instead of several times each day, as would be the case in a typical household. The average water volume obtained in this research can be considered as a reference for the minimum water consumption. According to the Spanish Integral Plan of Solid Waste (2007-2015), the average OFMSW production in Spain is 0.63 kg per inhabitant equivalent (IE) and day. Therefore, considering the average water volume 4 L kg⁻¹ used in the grinding process, the extra water consumption results in 2.52 L d⁻¹ IE⁻¹, clearly insignificant in comparison with the average water supply, which can be in the 200 – 300 L d⁻¹ IE⁻¹ range.

Table 1. OFMSW chemical characterization results obtained in the present study and wastewater characterization results obtained by Giménez *et al.* (2014)

Parameter	units	OFMSW			WW		
		n	average	SD	n	average	SD
COD (5 mm)	mg COD·L ⁻¹	39	63600	13400			
COD (0,5 mm)	mg COD·L ⁻¹	7	59400	14570	137	585	253
Soluble COD	mg COD·L ⁻¹	34	18100	4200	110	80	20
pH		40	6.3	0.72	471	7.7	0.2
Cond	mS·cm ⁻¹	45	6.08	1.23	489	0.195	0.017
Alkalinity	mg CaCO ₃ ·L ⁻¹	42	161.4	65	515	332	58
VFA	mg HAc ·L ⁻¹	43	757	233.5	516	7.9	5.5
Total Nitrogen	mg N·L ⁻¹	33	91.6	19.4	78	55	12.8

Total Soluble Nitrogen	mg N·L ⁻¹	33	49.5	11.2			
% Soluble Nitrogen		31	55	14	45	67.8	11.3
N-NH ₄	mg N·L ⁻¹	35	23.5	6.4	376	32.2	8.9
N-NO ₂	mg N·L ⁻¹	16	0.1	0.1			
N-NO ₃	mg N·L ⁻¹	16	0.8	0.5			
Total Phosphorous	mg P·L ⁻¹	31	114.5	39.1	52	10.3	3.6
Total Soluble Phosphorous	mg P·L ⁻¹	27	89.3	33.7			
% Soluble Phosphorous		27	80	14	52	47.7	9.9
P-PO ₄	mg P·L ⁻¹	28	81.9	26.7	368	4	1.6
S-SO ₄ ^(a)	mg S·L ⁻¹	26	132.7	35.5	211	105	13
S-SO ₄ grinding water ^(a)	mg S·L ⁻¹	20	133.8	21			
Suspended Solids ^(c)	mg SS·L ⁻¹	16	26800	15000	459	323	176
Volatile Suspended Solids ^(c)	mg VSS·L ⁻¹	16	25000	13900			
% VSS		16	94	7	43.3	80.4	7.9
Suspended Solids ^(d)	g SS·L ⁻¹	20	29.9	8.8			
Volatile Suspended Solids ^(d)	g VSS·L ⁻¹	15	29.3	7.8			
% VSS		15	98	4			
Total Solids	g TS·L ⁻¹	23	47.6	12.5			
Volatile Total Solids	g VTS·L ⁻¹	19	42.2	14.5			

^(a): Lab determination; ^(b): Calculated by balance; ^(c): Direct method; ^(d): Calculated by difference

1

2 **Chemical Oxygen Demand (COD) and Sulphate concentration**

3 Total and soluble COD were determined in the samples. Table 1 shows COD_T sieved by
4 5 and 0.5 mm and soluble COD (COD_S). As expected, the COD concentration (63600 ±
5 13400 mg·L⁻¹ sample sieved by 5 mm and 59400 ± 14570 mg·L⁻¹ sample sieved by 0.5
6 mm) is much higher than the average concentration of the AnMBR pilot plant influent
7 (585 ± 253 mg·L⁻¹). It was therefore expected that this high organic load would
8 significantly increase biogas production. It can also be observed that the difference
9 between the COD concentrations in samples sieved by 5 and 0.5 mm is less than 5%.
10 This, combined with the fact that 90% of the particle size is under 0.5 mm, confirms that,
11 despite pre-treatment, a large proportion of the OFMSW will be treated in the AnMBR
12 pilot plant. The soluble fraction followed a steady trend and was approximately 30% of
13 the total COD. The COD concentration of around 63600 mg·L⁻¹ is similar to that obtained
14 by Kujawa-Roeleveld *et al.* (2003).

15

16 As can be seen in Table 1, the sulphates determined in the samples were mainly from the
17 grinding water. The soil characteristics of the Mediterranean coastal area, which contains
18 high concentrations of sulphate, contributes to the presence of this compound in the water

1 supply. Sulphate concentration determines the competition between sulphate-reducing
2 bacteria (SRB) and methanogenic archaea (MA) for the available substrate (COD) in
3 anaerobic processes. This competition depends on the COD/SO₄-S ratio. SRB need 2 g
4 COD·g⁻¹ S-SO₄ for sulphate reduction, so that when the ratio is higher than two, there is
5 enough COD for the growth of both populations. As the OFMSW COD is higher than
6 that measured in the WW and sulphate concentration remains almost the same in both
7 samples, the COD/SO₄-S ratio will be higher when OFMSW is included in the WW
8 treatment (COD/SO₄-S ratio of 5.6 in the WW and 448 in the OFMSW). This means that
9 there will be much more substrate available for MA, which will promote methanogenic
10 activity in the process, which will increase biogas production significantly.

11

12 **pH and Conductivity**

13 OFMSW pH and conductivity values remained quite stable, with a pH range between 5.5-
14 7, depending on storage period, and higher conductivity (around 6 mS·cm⁻¹) than WW
15 (190.5 μS·cm⁻¹).

16

17

18 **Volatile fatty acids (VFA) and Alkalinity**

19 As can be seen in Table 1, the recorded VFA and alkalinity present high variability (from
20 493 to 1234 mg HAC·L⁻¹, and from 47 to 248 mg CaCO₃·L⁻¹, respectively). This is
21 probably due to the different stages of OFSMW fermentation and also to the variability
22 in the sample composition. Conversely, the concentration of these compounds in WW is
23 relatively stable, exhibiting a very low VFA concentration and an average of 332 mg
24 CaCO₃·L⁻¹ alkalinity.

25

26 VFA and pH were monitored in two degradation experiments, in which the samples were
27 maintained at ambient temperature for 10 and 7 days (see Figure 3). These experiments
28 were started on different dates: the experiment on the first sample began at the beginning
29 of February and the second in mid-February. As can be seen, both samples exhibited
30 exactly the same time course evolution, and both the VFA and pH were reasonably well
31 modelled with first and second order kinetics, respectively. Biological degradation was
32 fast and began immediately, exhibiting noticeably higher VFA concentrations than those
33 reported by Bernstad *et al.* (2013) (around 800 mg VFA L⁻¹ after 30 days), which could
34 be due to the lower particle size and the higher temperature used in the present research

1 (average temperature 14°C). This factor should be taken into account in the storage time
2 of OFMSW in tanks, as it is not advisable to store it for prolonged periods due to the
3 intense acidification and negative effects on any subsequent anaerobic treatment.

4
5 **[Figure 3 Near Here]**
6

7 8 **Nutrients: Nitrogen and phosphorous**

9 Table 1 shows the average concentration of the different forms of nitrogen and
10 phosphorous. As can be seen in this table, OFMSW nitrogen concentration is almost two
11 times higher than in the WW ($91.6 \text{ mg N} \cdot \text{L}^{-1}$ vs $55 \text{ mg N} \cdot \text{L}^{-1}$). However, taking into
12 account the relative flow rate contribution from both sources OFMSW $2.52 \text{ L d}^{-1} \text{ IE}^{-1}$ and
13 wastewater $200 - 300 \text{ L d}^{-1} \text{ IE}^{-1}$, the higher concentration of nitrogen in the OFMSW will
14 not affect the nitrogen influent concentration of the OFMSW and WW mix. A similar
15 conclusion can be drawn for the OFMSW phosphorus concentration, whose values are 10
16 and 20 times higher for total phosphorous and orthophosphate, respectively, in
17 comparison to wastewater.

18 19 **Suspended solids (SS)**

20 Suspended solids were calculated following the APHA (2012) protocol and as the
21 difference between total (TS) and dissolved solids (DS). As can be seen in Table 1,
22 although the mean value obtained was very similar for both methods, the results of the
23 latter method had a lower standard deviation ($26.8 \pm 15 \text{ g} \cdot \text{L}^{-1}$ vs $29.9 \pm 8 \text{ g} \cdot \text{L}^{-1}$,
24 respectively). In both cases almost 100% of the SS is in the form of volatile solids. These
25 values are similar to those obtained by Kujawa-Roeleveld *et al.* (2003) but significantly
26 lower than those reported by Luostarinen and Rintala (2006), Nayono *et al.* (2009) and
27 Rajagopal *et al.* (2013), which ranged from 255 to 295 $\text{g}_{\text{TSS}} \text{ L}^{-1}$. The proportion of
28 suspended solids to total solids was also calculated and was found to be similar by both
29 methods (56% for APHA protocol and 63% for the calculation method). Total solids
30 concentration is approximately $50 \text{ g} \cdot \text{L}^{-1}$ with 90% of volatile solids.

31 32 **3.3 Biochemical Methane Potential assays of OFMSW**

1 Table 2 shows the main results of the six BMP experiments carried out. As can be seen,
 2 anaerobic biodegradability is quite similar in all the experiments (average
 3 biodegradability 73%), irrespective of inoculum and particle size of the OFMSW sample
 4 tested. The pH was measured at the beginning and at the end of each BMP test, being in
 5 the range 6.5-6.9 at beginning and 7.1-7.7 at the end. Therefore, no inhibition due to
 6 acidification was observed. The average of the first five BMP experiments (i.e. those that
 7 include total OFMSW) was 252 ± 11 ml $\text{CH}_4 \cdot \text{g}^{-1}$ COD and 421 ± 15 ml $\text{CH}_4 \cdot \text{g}^{-1}$ VS.
 8 Although the final results were similar, there was a noticeable difference in the time
 9 evolution of methane production (see Figure 4 a), which varied according to the origin of
 10 the inoculum. Samples inoculated with sludge from the AnMBR pilot plant when it
 11 treated WW only (E3 and E4) exhibit a period of low methane production (from days 5
 12 to 20 in Figure 4a) followed by a rapid increase in methane production, reaching similar
 13 biodegradability rates to the samples inoculated with sludge from the urban WWTP
 14 mesophilic AD (E2). This observed lag-phase is due to the hydrolysis step of the
 15 anaerobic digestion process and could be attributed either to fewer hydrolytic bacteria in
 16 the AnMBR pilot plant or the lower hydrolysis yield of the hydrolytic bacteria present.
 17 This was confirmed by comparing the evolution of methane production from total
 18 OFMSW and the soluble fraction of the OFMSW (see Figure 4b). As can be seen in
 19 Figure 4b, the lag-phase perfectly matches the soluble fraction degradation and as the
 20 particulate fraction is being hydrolysed the methane production of the total OFMSW
 21 sample increases again.

22

23 **Table 2.** Overview of the conditions and main results of the six BMP experiments performed.

	E1	E2	E3	E4	E5	E6
Inoculum	AD ⁽¹⁾	AD ⁽¹⁾	AnMBR ⁽²⁾	AnMBR ⁽²⁾	AnMBR ⁽³⁾	AnMBR ⁽³⁾
Sample size	5 mm	0.5 mm	5 mm	0.5 mm	0.5 mm	Soluble
Ratio S/I (g COD·g ⁻¹ VS)	1:1.5	1:3	1:2	1:3	1:2	1:6
% Biodegradability	68%	76%	72%	75%	70%	73%
BMP (ml CH ₄ ·g ⁻¹ COD)	236	265	250	250	260	255
BMP (ml CH ₄ ·g ⁻¹ VS)	401	443	418	418	426	447

24 (1) Sludge from urban WWTP mesophilic anaerobic digester; (2) Sludge from a AnMBR pilot plant treating WW; (3)
 25 Sludge from a AnMBR pilot plant adapted to treat WW jointly with OFMSW. Results shown in the table are the average
 26 values of the duplicates from each experiment.

27

28

29

[Figure 4 Near Here]

30

1
 2 Físgativa *et al.* (2016) in their review-paper reported a mean value of 460 mL CH₄ · g⁻¹
 3 VS added, Browne and Murphy (2014) obtained a range between 467-529 mL CH₄ · g⁻¹
 4 VS added in their study, Zhang *et al.* (2012) obtained 455 mL CH₄ · g⁻¹ VS added,
 5 Davidsson *et al.* (2007) obtained a range between 300-400 mL CH₄ · g⁻¹ VS added and
 6 Cecchi *et al.* (2003) obtained 401-489 mL CH₄ · g⁻¹ VS added. It can thus be seen that the
 7 range in the literature is similar to that found in the present study.

9 **3.4 Preliminary assessment of the fate of OFMSW in a conventional WWTP**

10 As a preliminary assessment of the effect of incorporating OFMSW into a current
 11 conventional wastewater treatment system with primary sedimentation and further
 12 treatment of organic matter via an aerobic process, settleability tests were performed
 13 using a WW + OFMSW mixture. Considering the relative flow rate contribution from
 14 both sources OFMSW 2.52 L d⁻¹ IE⁻¹ and wastewater 225 L d⁻¹ IE⁻¹ (200 – 300 L d⁻¹ IE⁻¹
 15 ¹), the mixture was prepared with 11.20 mL of ground OFMSW per 1 L of WW. These
 16 settleability tests provide some insight into the proportion of solids that would be treated
 17 in the water line by aerobic oxidation without COD valorisation. Only the particulate
 18 matter settled in the primary clarifier would be valorised if the WWTP includes anaerobic
 19 digestion as part of its sludge treatment line. The results of the settleability experiments
 20 are summarized in Table 3.

21
 22 **Table 3.** Average results from the three settleable solids experiments performed.

	WW	OFMSW+WW
Settleable solids (ml L ⁻¹)	14.5 ± 4	28 ± 2
Supernant Total COD (mg·L ⁻¹)	305±78	793±133
Supernant Total nitrogen (mg N·L ⁻¹)	57.42±14.08	58.25±14.5
Supernant Total phosphorus (mg P·L ⁻¹)	1.82±0.24	2.44±0.05

23
 24 As can be seen in Table 3, both treatment lines of a conventional WWTP (water and
 25 sludge lines) would be clearly impacted by the presence of the OFMSW in the influent.
 26 The settleable solids fraction has significantly increased (almost double), and these are
 27 the solids that will reach the digestion stage of the WWTP. The COD concentration would
 28 be increased by around 2.6 times more than its concentration in urban WW, leading to a
 29 considerable escalation in the aeration costs of the WWTP. Therefore, to make the most
 30 of all the COD present in the influent (due to the WW plus the extra COD contributed by
 31 the OFMSW) it would be wise to treat it anaerobically, and AnMBR technology would

1 thus be an option. The effect of OFMSW incorporation on influent nutrient composition
 2 would be negligible.

3.5 Mass balance-based influent composition estimation due to co-digestion

5 As has been shown above, there are remarkable differences between OFMSW and WW,
 6 not only in pollutant concentrations but also in their flow rate contribution. If co-digestion
 7 of WW and OFMSW is implemented in the WWTP, the new influent composition can be
 8 estimated through the mass balance of both streams. The results are shown in Table 4.

11 **Table 4.** Estimation of co-digestion influent composition through mass-balance.

Parameter	units	OFMSW	WW	OFMSW+WW	Increase*
Flow rate	(L·IE ⁻¹ ·d ⁻¹)	2.52	225	227.5	1.011
Total COD	(mg COD·L ⁻¹)	59400±14570	585 ± 253	1236.4	2.114
Soluble COD	(mg COD·L ⁻¹)	18100±4200	80 ± 20	279.6	3.495
Total N	(mg N·L ⁻¹)	91.6 ± 19.4	55 ± 12.8	55.4	1.007
N-NH ₄	(mg N·L ⁻¹)	23.5 ± 6.4	32.2 ± 8.9	32.1	0.997
Total P	(mg P·L ⁻¹)	114.5 ± 39.1	10.3 ± 3.6	11.5	1.112
P-PO ₄	(mg P·L ⁻¹)	81.9 ± 26.7	4 ± 1.6	4.9	1.216
S-SO ₄	(mg S·L ⁻¹)	132.7 ± 35.5	105 ± 13	105.3	1.003
TSS	(mg SS·L ⁻¹)	29900± 8800	323 ± 176	650.6	2.014

12 * Increase with respect to WW, calculated as the ratio (OFMSW+WW)/WW

14 As can be seen in Table 4, the main effect of incorporating OFMSW into WWTP would
 15 be in COD and TSS (which would increase around 2 times), and the effect of the
 16 remaining parameters (flow-rate and nutrients) would be almost negligible. This means a
 17 considerable increase in the COD/SO₄-S ratio could be expected, affecting the
 18 competition between sulphate-reducing bacteria (SRB) and methanogenic archaea (MA)
 19 in anaerobic conditions. Since SRB use part of the organic matter to reduce sulphate to
 20 sulphide (2 grams COD per gram of S-SO₄), less organic load is available for MA, so that
 21 the higher the COD/SO₄-S ratio in the influent, the higher the methane production in the
 22 anaerobic digester.

24 The increase in methane production in anaerobic digestion can be estimated by taking
 25 into account the anaerobic biodegradability of both COD sources: OFMSW, which was

1 73% (see Section 3.3) and the COD from the WW, which was assumed to be 43% in
2 Kassab *et al.* (2013). This means 255.15 mL of methane will be produced per 1 L of the
3 new influent (i.e. composed of both WW and OFMSW), while methane production is
4 88.04 mL per 1 L of WW only. Should co-digestion be implemented, methane production
5 would therefore increase by 2.9 times the production of WW only. Further research is
6 currently being carried out at the AnMBR pilot plant in Carraixet WWTP (Valencia,
7 Spain) to confirm these results experimentally.

10 **4. Conclusions**

11 The exhaustive characterization performed confirms that co-digesting OFMSW with WW
12 in an AnMBR plant is a feasible option for recovering energy from waste. OFMSW is
13 characterized by a high COD concentration ($63600 \pm 13400 \text{ mg COD}\cdot\text{L}^{-1}$) compared to
14 WW ($585\pm 253 \text{ mg COD}\cdot\text{L}^{-1}$), sulphate concentration is in the same range (around 110
15 $\text{mg S}\cdot\text{SO}_4\cdot\text{L}^{-1}$), therefore the COD/SO₄-S ratio will increase when the OFMSW is
16 incorporated into the influent of a WWTP, resulting in a higher organic load available for
17 MA. The average BMP of OFMSW is $421\pm 15 \text{ mL CH}_4\cdot\text{g}^{-1} \text{ VS}$, yielding an anaerobic
18 biodegradability of 73%. Adding OFMSW to the influent wastewater would increase
19 methane production 2.9-fold in comparison with wastewater only. Considering the
20 relative flow rate contribution from both OFMSW ($2.52 \text{ L d}^{-1} \text{ IE}^{-1}$) and wastewater (225
21 $\text{L d}^{-1} \text{ IE}^{-1}$) streams, the higher nitrogen and phosphorus concentrations present in the
22 OFMSW will become negligible when both streams are mixed. The settleable solids tests
23 and the particle size distribution analyses confirm that both treatment lines of a
24 conventional WWTP (water and sludge lines) would be clearly impacted by the
25 incorporation of OFMSW into the influent. Anaerobic processes are thus more suitable,
26 due to their ability to valorise the high COD content to produce biogas (a renewable
27 energy) instead of increasing the energetic cost associated with aeration in aerobic
28 processes.

30 **5. Acknowledgements**

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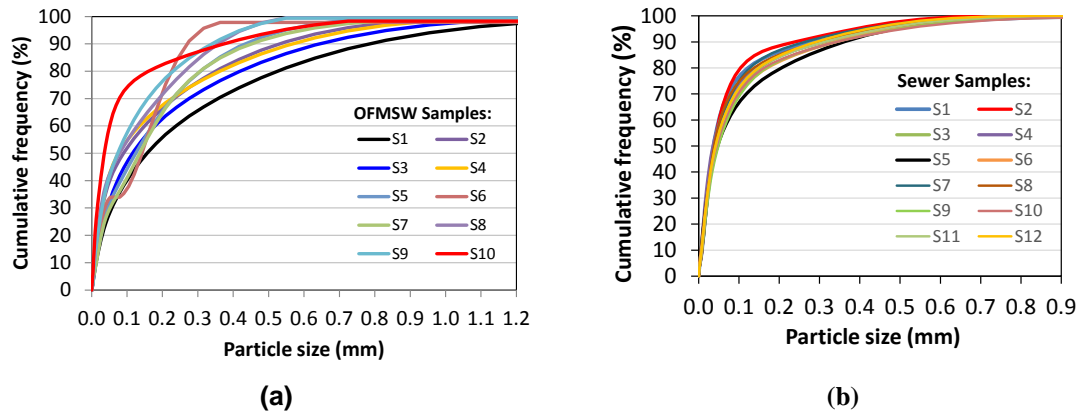
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27

28

1 **FIGURE 1**

2



3 **Figure 1.** Particle size distributions (a) in OFMSW samples (b) in sewer pipeline samples collected 100
4 metres upstream of the Carraixet WWTP inlet.

5

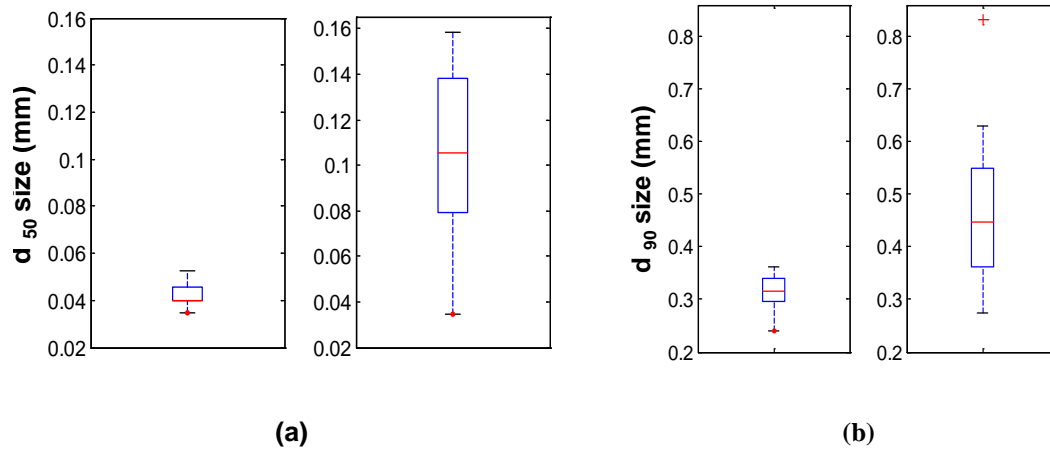
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7

1 **FIGURE 2**

2

3



4 **Figure 2.** Box-Whisker plots of representative sizes from sewer pipeline samples (100 metres upstream of
5 the WWTP inlet) and in OFMSW samples **(a)** d_{50} particle size **(b)** d_{90} particle size.

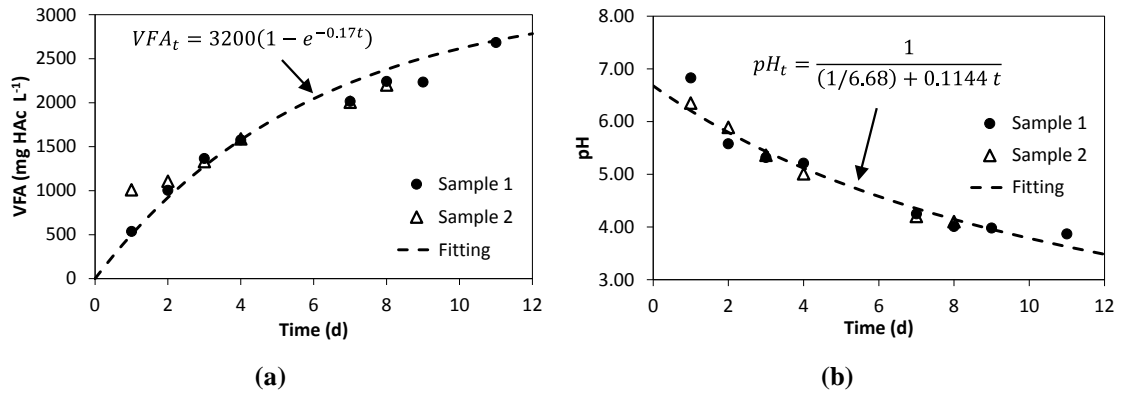
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1 **FIGURE 3**

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3



4 **Figure 3.** Time course evolution of VFA (a) and pH (b) in two OFMSW samples left at ambient
5 temperature (average temperature 14°C).

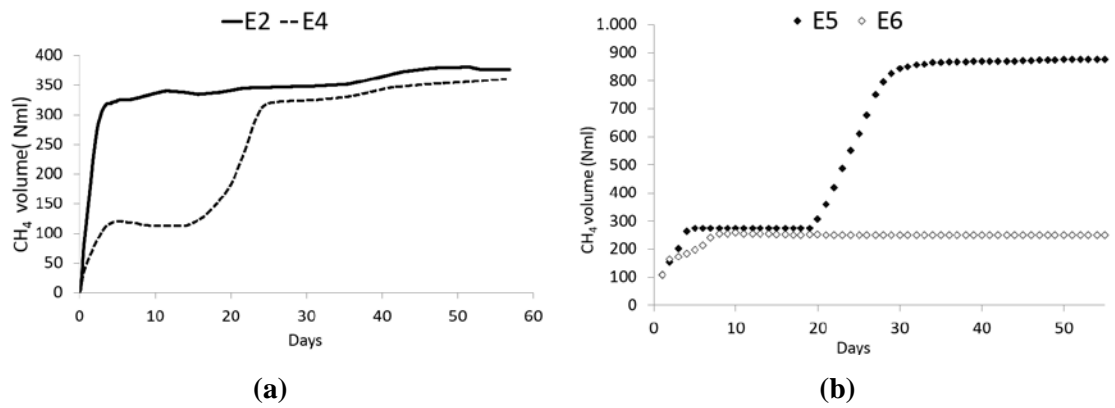
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1 **FIGURE 4**

2



3 **Figure 4.** Time course evolution of methane production in: (a) BMP experiments E2 (inoculated with
4 sludge from urban WWTP mesophilic digester) and E4 (inoculated with AnMBR pilot plant sludge)
5 (b) BMP experiments E5 (0.5 mm sieved OFMSW as substrate) and E6 (soluble fraction of OFMSW as
6 substrate), both inoculated with AnMBR pilot plant sludge.

7