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Valorization of Food Waste Leachates through Anaerobic Digestion †

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Abstract: According to the European Union data, on average 173 kg per person of total food waste (organic waste) is produced annually, of which 92 kg per person comes from households (organic waste). Food waste is defined as the waste from households, restaurants, canteens, and food industries, as well as markets. The importance of food waste stretches from the environmental pressures to the economic and social impacts. An environmental technology for the biodegradation of food waste is anaerobic digestion. It is a very attractive technique and combines waste treatment and renewable energy recovery. This study investigates the characteristics of food waste leachates from composting buckets and their valorization as a substrate for the anaerobic digestion process. A complete characterization of different food waste leachates was conducted (pH, COD, VFAs, heavy metals, etc.). The food waste leachates proved to be an ideal feedstock for anaerobic digestion. In this direction, batch tests were performed to evaluate the methane yield of the food waste leachates under different operating conditions. Three different SIR ratios were tested (0.5, 1.0, and 1.5). An SIR equal to 0.5 proved to be the best as the higher methane yield was achieved. The removal of the COD under all the operating conditions was higher than 70%, with the higher removal (85.18%) for an SIR equal to 1.5.

Keywords: food waste leachates; anaerobic digestion; SIR; VFA; heavy metals

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1. Introduction

Anaerobic digestion is an environmentally favorable technology and the most widespread good practice for the biodegradation of household waste. Anaerobic digestion is a complex biochemical process in which organic material is decomposed by several groups of microorganisms in the absence of oxygen while renewable energy such as biogas is generated.

This technique is very attractive and combines waste treatment and renewable energy recovery. In addition to these two benefits, anaerobic digestion also reduces the odor of the waste material, while the digestate is rich in nutrients that can be used as fertilizer after the process of fermentation.

It is important to mention that, for this technology, the monitoring of significant factors [1] is essential; these include: pH, total solids (TS), volatile solids (VS), ammonium (NH_4^+), chemical oxygen demand (COD), alkalinity, volatile fatty acids (VFAs), total organic carbon (TOC), and total nitrogen (TN).

2. Materials and Methods

2.1. Sampling and Pretreatment

Compost leachates were collected during the period from May 2020 to May 2021. They mainly consist of fruits and vegetables, and their sampling procedures were from different stages of the composting process.

Specifically, the samples were collected before their composting for analysis as raw materials and as leachate of the compost samples in a pre-compost phase. The samples were collected from specific sampling spots. The composting containers were placed in public markets for the collection of the food waste. In the period from October 2020 to May 2021, the samples were leachates from mechanical composting plants and waste transfer stations. They came from different cities of the region of Western Macedonia, in Greece. The sample codes, their origins and their dates of sampling, are presented in the following Table 1. For each sample, 1.5 L of compost leachates was collected and delivered to the laboratory of CERTH in Ptolemais within 24 h. Depending on the test method, the samples were filtered through a membrane filter (glass fiber, 0.45 syringe filter, etc.), acidified, and centrifuged (HPLC analysis). A portion of the filtrated liquid was freeze-dried, and the remaining was stored at 4 °C before further analysis.

Table 1. Descriptions of the samples

Date of Sampling	Sample Name	Description of Sample
1 June 2020	S1	Raw material from bucket in public market
12 June 2020	S2	Raw material from bucket in public market
22 June 2020	S3	Raw material from bucket in public market
22 June 2020	S4	Raw material from bucket in public market
3 November 2020	S5	Leachate from mechanical composting plants
22 December 2020	S6	Leachate from mechanical composting plants
17 March 2021	S7	Leachate from waste transfer station
17 March 2021	S8	Leachate from waste transfer station
28 April 2021	S9	Leachate from waste transfer station
28 May 2021	S10	Leachate from waste transfer station

2.2. Analytical Methods

The measurements of the TS, VS, COD, NH_4^+, alkalinity, TOC, and TN were carried out according to the APHA Standard Methods [2]. The pH was measured using a digital pH meter (Hanna, HI2260). The quantification of the major and trace metals was carried out according to the APHA Standard Methods and ISO 15586, and a graphite furnace atomic absorption spectrophotometer was used (Shimadzu, Tokyo, Japan, GFA-EX7i AA-6300). For the quantifications of the TOC and TN, a TOC analyzer (Shimadzu, TOC-L) was used.

Finally, the quantification of the VFAs was carried out by HPLC. A portion of 100 mL of filtered sample was acidified with 30 μL of H_3PO_4 (HPLC grade) and centrifuged at 10.000 rpm for ten minutes Moreover, the VFAs from the anaerobic digestion process were determined according to the Kapp method [3].

2.3. High Performance Liquid Chromatography

The used separation module (Ecom, Chrastany, Checz Republic, ECB2000) was equipped with a pump and degasser (Ecom, ECP2000), an oven (Ecom, ECO2000), and a diode array detector (Ecom, ECDA) and was coupled with a RP-C18 column (Fortis Technologies, Cheshire, UK, 250 × 4.6 mm, 5 μm). Spectra were obtained between 200 and 230 nm. The isocratic elution procedure applied to the mobile phase (0.02 mol/l KH_2PO_4/methanol) was stable at 98:2 for 50 min. The mobile phase was acidified with H_3PO_4 to reach pH 2.88. The flow rate was 0.6 mL/min at 35 °C and a 20 μL injection volume [4]. The standard stock solution containing 100 mg/L of the organic acids (acetic, propionic, and butyric acids) was prepared in ultrapure water.

2.4. Inoculum and Substrate

Anaerobic sludge was used as an inoculum (microbial culture) for the biomethane potential test arrays and was obtained from a commercial mesophilic anaerobic digester plant in the area of Eordea (Western Macedonia).

The substrate for the digestion process was the sample S9 (Table 1) because of the C/N ratio (Table 2). Three ratios of SIR (substrate to inoculum ratio) were monitored for biomethane potential at 1.5, 1.0, and 0.5. The calculation for the SIR ratios was determined according to the VS of the substrate and the inoculum. During start-up, flushing with N_2 took place, and all the samples were incubated in mesophilic conditions (35 +/− 2 °C) throughout the experimental process. All the batch tests were performed in triplicate.

Table 2. Complete characterization of compost leachate. VFAs represent the cumulated concentration of acetate, propionic, and butyric acid.

Parameter	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10
pH	4.29	4.65	4.52	3.84	4.02	4.82	4.35	4.91	4.48	4.05
TS (g/L)	118	24	69	114	36	18	33	26	41	60
VS (g/L)	98	14	45	100	28	14	25	17	31	48
COD (mg/L)	47,202	49,500	51,800	38,850	53,924	16,620	40,154	25,102	25,601	31,794
NH_4^+ (mg/L)	470	956	985	212	134	138	104	303	484	117
TOC (mg/L)	15,580	14,150	14,140	34,430	13,930	7368	9990	19,885	16,660	3692
TN (mg/L)	2558	3435	3588	3973	579	267	314	511	771	273
VFAs (g/L)	30,508	18,349	16,041	37,046	41,780	8847	12,768	14,874	21,613	24,946
C/N ratio	6.1	4.1	3.9	8.7	24.1	27.6	31.8	38.9	21.6	13.5

2.5. Biomethane Potential Test

BMP tests are a technique to determine the methane potential and the biodegradability of any type of waste [5]. Batch experiments were carried out using the Automated Methane Potential Test System II (AMPTS II). Each of the AMPTS' bioreactors had 500 mL bottles with 400 mL of working volume and 100 mL of headspace and was equipped with an individual mechanical stirrer and operated as a bench scale anaerobic glass bioreactor. The produced biogas from each glass bioreactor passed through a 3 M NaOH solution, which retained the CO_2 and H_2S. The upgraded biogas passed through a flow cell (one for each glass bioreactor), which measured gas productivity through water displacement. The digital impulse was registered by a computer [6,7]. The results of the BMP test experiments are expressed as normalized mL.

3. Results

The results of the complete characterization of samples S1 to S10 are summarized below in Tables 2 and 3. The significant variation of the leachate characteristics could be attributed to the impact of the factors that affect quality and quantity, including waste composition, age of the waste, and the composting technology used [8]. The leachate obtained was brown with an unpleasant odor that could be attributed to the organic acids and volatile fatty acids produced from the composting food waste. Other volatile nitrogen and sulfur compounds could also have contributed to this odor [9].

The accumulated biomethane yields and the production flows of the three SIRs are shown in Figure 1. Tables 4 and 5 shown the main characteristics of the inoculum and feedstock. Figure 1 illustrates the accumulated Nml CH_4/g VS (a) and the flow rate (Nml/day) of the three SIR ratios. Table 4 summarizes the composition of the SIR feedstock and the inoculum used in the batch test. Finally, Table 5 depicts the main characteristics of each bench scale bioreactor during the start-up phase and after the end of the batch experiment.

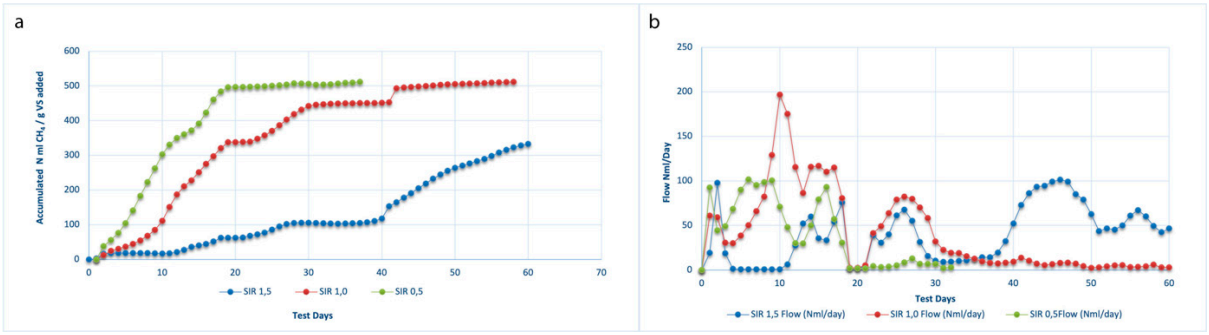


Figure 1. (**a**) Accumulated Nml CH4/g VS added of SIR 0.5, 1.0, and 1.5. (**b**) Flow (Nml/day) of SIR 0.5, 1.0, and 1.5.

Table 3. Major and minor trace elements of compost leachate.

Parameter (mg/L)	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10
Na	6208	7210	7506	7005	3980	2075	233	577	552	1251
K	874	869	891	1329	145	280	1011	1320	1905	2779
Mg	337	227	226	532	370	166	90	316	168	895
Zn	1.4	2.1	2.1	4.3	4.0	1.4	12	27	18	16
Fe	203	335	340	164	281	357	1.4	0.5	31	32
Cu	0.3	0.3	0.5	0.6	0.6	0.2	0.6	0.3	1.1	1.6
Pb	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Ni	nd	nd	nd	nd	nd	nd	1.5	1.0	1.2	2.5
Cr	nd	nd	nd	nd	nd	nd	1.2	3.4	3.3	2.6
Cd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Mn	nd	nd	nd	nd	nd	nd	1.2	1.2	3.8	3.4

Table 4. Composition of the SIR feedstocks and inoculum used in the batch experiments in bench scale bioreactors.

Parameter	Inoculum	FW (0.5)	FW (1.0)	FW (1.5)
Alkalinity (mg/L $CaCO_3$)	15,000	3225	4150	5075
VS (mg/L)	24,000	2400	4800	7200
COD (mg/L)	22,820	8173	16,047	23,921
NH_4^+ (mg/L)	972	67	83	103
TOC (mg/L)	6623	952	1940	2930

Table 5. Main characteristics of each bench scale bioreactor during the start-up phase and after the end of the batch experiment. VFAs represent the cumulated concentration of acetate, propionic, and butyric acid.

Parameter	FW (0.5)		FW (1.0)		FW (1.5)	
	Initial Concentration	Final Concentration	Initial Concentration	Final Concentration	Initial Concentration	Final Concentration
pH	6.41	7.95	6.03	7.79	5.80	7.88
VS (g/L)	19.54	2.65	31.6	4.24	47.38	4.71
VFAs (mg/L HACeq)	4371	1678	5175	1076	9366	948
NmL CH_4/g VS	512.00		511.76		333.02	
COD (mg/L)	7138	1204	12,451	3349	26,880	3983
NH_4^+ (mg/L)	552	1053	818	1553	669	1355
Test Days	32		60		60	

4. Discussion

The pH values of the samples ranged between 3.84 and 4.91. According to the literature [10], in the process of the degradation of organic material carbon dioxide and a low

amount of ammonia are produced, and these two products further result in the formation of ammonium ions and carbonic acid. The carbonic acid dissociates to produce hydrogen and bicarbonate ions, which influence the pH level.

The solids (TS, VS) are influenced by the total amount of dissolved organic and inorganic material. According to the literature, a typical leachate ranges from 0.589 to 196 g/L [8]. The present values in this study remain within this range, and the range of volatile solids is between 13.5 g/L and 98 g/L.

The range of TOC values is between 3.500 mg/L and 35.000 mg/L. The TOC content decreases during composting due to the microorganism activity [11] and the further degradation of the organic substances necessary for their metabolism.

Nitrogen is oxidized mainly to ammonium and to nitrite and subsequently to nitrates when nitrification is achieved [10]. The values of the ammonium range between 985 mg/L and 134 mg/L, and the total nitrogen ranges between 3.973 mg/L and 267 mg/L.

The COD values include the oxygen demand created by biodegradable as well as non-biodegradable substances. The COD is highly variable, and this is due to the food waste composition and the climate characteristics [8], with reported values varying between 16.620 and 53.924 mg/L.

Finally, the values of the VFAs were present in high concentrations; this means that the composting process is in an initial stage, or it is a raw compost material and characterized as immature [12].

The batch experiments lasted 32 days for the SIR 0.5 and 60 days for the SIR 1.0 and 1.5, respectively, until the minimum or no biogas production was observed. Almost 2400 NmL and 2456 NmL methane were produced from SIR 1.5 and 1.0, respectively, and 1326 NmL methane was produced from SIR 0.5. According to Figure 1a, the values of Nml methane correspond to the methane yields 333.02 NmL CH_4/g VS added; 511.76 NmL CH_4/g VS added; and 512 NmL CH_4/g VS added, for SIR 1.5, 1.0, and 0.5, respectively. SIR 0.5 led to a higher methane yield in 32 days than the other two SIRs in 60 days. The degradation of the VS, VFAs, and COD for each SIR are shown in Table 5.

As illustrated in Figure 1b, a high biogas flow rate was observed from the 1st day of the three SIR batch experiments and continued until the 19th day. Furthermore, the flow reduction for SIR 0.5 ceased at the 32nd day. SIR 1.0 displayed a high flow rate until the 30th day. After the 30th day, a continuous reduction in the biogas flow rate was observed until it stopped at the 60th day. Finally, SIR 1.5, after the 30th day, displayed a continuous increase in the biogas flow rate until the cease of the batch experiments test at the 60th day.

5. Conclusions

In this study, the changes in food waste compost leachate were monitored with regard to the seasonality, the composting time, and the biomethane yield in three different SIRs. The compost leachate showed a high organic load, which means it is an ideal substrate for composting or anaerobic digestion, but simultaneously, it showed high values of volatile fatty acids, which means the compost is in the initial stages of the composting process and is considered as immature. Regarding the values of the COD and total nitrogen, in this stage of the process they are in high concentrations, and thus, it is recommended to use the leachate in low application rates or after dilution.

Finally, food waste compost leachates could be characterized as an ideal substrate for anaerobic digestion. The three different food waste SIR ratios in the bench scale experiment, produced biomethane in a sufficient quantity (expressed as Nml CH_4/g VS added); however, the SIR 0.5 produced a higher biomethane yield in half the days of the procedure. The degradation of the COD rates of the SIR 0.5, 1.0, and 1.5 were 83.13%, 73.11%, and 85.18%, respectively.

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Conflicts of Interest: The authors declare no conflict of interest.

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