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Codigestion of Untreated and Treated Sewage Sludge with the Organic Fraction of Municipal Solid Wastes

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Abstract: Disposal of biodegradable waste has become a stringent waste management and environmental issue. As a result, anaerobic digestion has become one of the best alternative technology to treat the organic fraction of municipal solid wastes and can be an important source of bioenergy. This study focuses on the evaluation of biogas and methane yields from the digestion and co-digestion of mixtures of waste untreated sludge and the organic fraction of municipal solid wastes. These are compared with the results obtained from the digestion and codigestion of mixtures containing waste active sludge and the organic fraction of municipal solid wastes. The two types of substrates were used to perform biomethanation potential tests, in mesophilic conditions (35 °C) at lab scale. It was observed a maximum biogas yield for 100% of untreated sewage sludge, corresponding to 0.644 Nm^3/kg VS and 0.499 Nm^3/kg VS of biogas and methane production respectively. The study also demonstrates the possibility of increasing biogas production up to 36% and methane content up to 94% using waste untreated sludge substrate in both digestion and codigestion, compared to waste active sludge substrate.

Keywords: co-digestion; sewage sludge; methane production; BMP; municipal solid waste

1. Introduction

The huge amount of sewage sludge and of Organic Fraction of Municipal Solid Waste (OFMSW), which are disposed of daily through incineration or land filling constitutes a huge environmental challenge. The European Union regulations demand that biodegradable municipal waste to landfill sites must be reduced by 25% with respect to 1995 levels by 2010 with a further reduction of 65% by 2016, see [1,2]. According to recent estimates of the European Commission, about 88 Mt of bio-waste extracted from municipal solid waste [3] and 10 Mt of Waste of Active Sludge (WAS) dry matter [4,5] are produced annually in the EU-27. However, given the organic content and chemical composition of WAS and OFMSW, they are easily biodegradable in anaerobic conditions that favor decomposition and mineralization producing biogas and residues, that can be used as nutrient soil replacement. Organic waste management through Anaerobic Digestion (AD) represents a useful solution to decrease the environmental impact caused by landfill disposal.

Waste sludge considers treated sludge in three forms: primary sludge, secondary sludge (which is called WAS) and mixture of primary and secondary sludge (thickened sludge). Primary sludge, according to [1], is more easily degradable in anaerobic conditions than WAS. A typical aerobic treatment for sewage is usually performed in a wastewater treatment plant (see Figure 1), with various scales of aerobic duration and sedimentation, in order to reduce the Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand (COD) of the waste prior to its landfilling or conveying to surface water. However, primary and secondary treatment process releases a significant amount of methane, which is lost to the atmosphere, increasing the environmental impacts and losing potential energy of the sludge [6]. An AD process integrated with the aerobic treatment, would recover a significant amount of biogas for energy production treatment. Moreover, the possibility of treating together OFMSW and sewage sludge to produce biogas in a system eventually integrated with the aerobic treatment has an interesting potential. However the best mixture between OFMSW and sewage, in terms of biogas production, is still under analysis. Also whether to use WAS (secondary sludge) or Primary Sludge (PS) as even untreated sludge is not yet ascertained. In order to evaluate the quality of biowaste to serve as a substrate in anaerobic digestion several methods are used, such as; Anaerobic Biomethanation Potential (ABP), pilot plant and full scale plant test (see Table 1). Various studies are available on anaerobic co-digestion of treated sludge WAS, Primary Sludge (PS), thickened sludge with OFMSW or biowastes. Various studies are available on anaerobic co-digestion of treated sludge WAS, Primary Sludge (PS), thickened sludge with OFMSW or biowastes, see Kolbl [7,8]; however, no one addresses the co-digestion with Untreated Sewage Sludge (USS—fresh sludge without primary and aerobic treatment). Lab scale results show that the co-digestion of WAS-OFMSW could be the most effective way to improve digester performance, according to [2,9–12]. Murto et al. 2004 [2] observed that co-digestion with the high buffered system leaded to imbalance the process; Cabbai et al. [9] indicated that the high acid load of co-digestion substrates leads to the inhibition of AD process; Cavinato et al. [11] reported that thermophilic conditions perform better in a co-digestion process for sludge/biowaste in the terms of biogas production. Kim et al. [13] investigated the effect of different variables (temperature and mixing ratio), reporting that the addition of food waste to WAS digestion increases methane yield. Gomez et al. [14] observed that co-digestion (WAS-OFMSW) with pH and mixing ratio control achieved high methane production. Sosnowski et al. [15] compared the effects of different mixing ratios and determined that high organic load improves biogas yield. Cabbai et al. [9] concluded that certain types of organic waste source (household and supermarkets wastes) in co-digestion with WAS increase methane yield by 47% with respect WAS digestion alone. From all the above reported studies co-digestion of sewage sludge and organic solid wastes appears to be an important strategy the management of urban wastes.

Researchers have also focused on co-digestion technology based on synergisms/antagonisms between substrates [16], showing that a higher concentration of micronutrients in sludge compensates the shortage of OFMSW in a pH environment suitable for AD bacteria. It was shown that aerobic digestion became more stable with the C/N ratio of co-substrates remaining within the desired range of 22–30, see [17]. The co-digestion of WAS-OFMSW at different mixture ratios, was successfully experimented by [9,11,16]; when Solids Retention Time (SRT) is longer than Hydraulic Retention Time (HRT), Volatile Solids (VS) and microbial biomass should be retained for a higher biogas production, hence mixing can be reduced [18]; when co-digesting proteins-rich substrate WAS can provide the required buffering capacity [19]. Improvement in methane yield through co-digestion was achieved with increasing amount of organic waste in wastewater sludge digestion [9,15]. Nevertheless, to improve methane yield there is a limit to the addition of organic matter depending on the digestion conditions and stability (see Table 1). During the AD of a co-substrate microorganisms utilize carbon from 25 to 30 times faster than nitrogen [20] and the nitrogen content in WAS compensates a possible lack of nutrients in OFMSW while their content of lipids increases biogas yield [21]. On the other hand a lipid-rich substrate leads to an increase in Long Chain Fatty Acids (LCFAs) which may form a hydrophobic layer that destabilizes the digestion process [22], affecting bacteria transport and reducing contact between the substrate and the encapsulated bacteria. LCFAs entrapment causes the flotation and inhibition of methanogenic bacteria leading to cellular membrane damage [23]. Hence, the major benefit of WAS and OFMSW co-digestion is to reduce the toxicity within the media [2,15], and to provide other nutrients which are not present at sufficient levels in OFMSW. However, other authors showed negative results in their research, probably attributable to specific characteristics of the digested substrates [2,24]. This research activity provides information on biogas and methane yield of Untreated Sludge (USL) in different conditions and mixtures with OFMSW, given the lack of experimental data in the Literature, and compares its performance with WAS behavior. Although many studies have analyzed the co-digestion of WAS or thickened sludge with OFMSW still no data is available on the co-digestion of WUS and OFMSW. This works contributes by determining the BMP of different mixture in different condition of WUS-OFMSW and WAS-OFMSW in batch reactors, the sludge feedstock was taken from a sewage treatment plant in central Italy, while OFMSW consisted of household organic waste, collected in Perugia municipality area.

Table 1. Biogas and methane yields in similar studies.

Composition	SPG	CH_4	Method	References
(100% WAS)	0.390 (m^3/kg VS)	64%	BMP	[9]
(41.5% WAS-58.5% OFMSW)	0.620 (m^3/kg VS)	n.r.	BMP	[9]
(50% WAS-50% OFMSW)	0.34 (m^3/kg VS)	60%	Pilot scale	[11]
(100% WAS)	0.15 (m^3/kgVS)	61.8%	Pilot scale	[11]
(50% WAS-50% OFMSW)	0.35 (m^3/kg VS)	60%	Full Scale	[11]
(75% WAS-25% OFMSW)	0.45 (m^3/kg VS)	53.8%	Pilot scale	[9]
(41% WAS-59% OFMSW)	0.43 (m^3/kg VS)	64%	Full scale	[25]
(77% TAS-23% (KW & FWP))	0.38 (Nm^3/kg VS)	n.r.	Pilot scale	[26]
((60% PS & 40% WAS)-OFMSW)	0.6 (m^3/kg VS)	n.r.	Full scale	[27]
(100% biological sludge)	0.27 (m^3/kg VS)	60%	BMP	[12]
(80% OFMSW-20% biological sludge)	0.22 (m^3/kg VS)	n.r.	BMP	[27]

2. Materials and Methods

2.1. *Sewage Sludge and Organic Fraction of Municipal Solid Wastes Samples*

Both WUS and WAS were obtained from a wastewater treatment plant which is part of network that serves a 150,000 citizens of the town of Perugia in central Italy; the plant collects the households effluents (a population equivalent of 30,000) and the industrial wastewater of the area. With reference to Figure 1, the plant layout consists of a series of treatment units (primary clarifier and secondary clarifier) and a final drying section for the sludge generated to be used in composting.

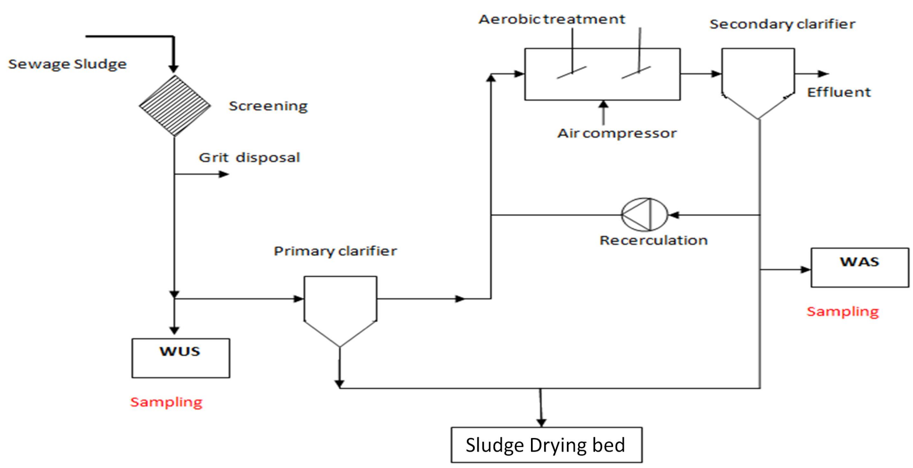

Figure 1. WWTP of the Umbrian Water management company.

As received fresh sewage was collected on site before the primary and secondary treatment unit during a normal working day with constant time steps (5 L sludge/10 min) summing up to 100 L. WUS was stored for 24 h. While WAS was collected from the exhaust of the secondary unit. The samples used in the BMP tests were prepared according to UNI 5667-13/2000. OFMSW was assembled from households waste with the following concentration in weight: 45% vegetables and fruit waste, 35% residuals of bread and pasta, 10% rice, 5% paper and 5% coffee; the materials were eventually homogenized with an electric mixer. The samples of OFMSW were obtained according to CENT/TS 14778-1, CENT/TS 14779. The inoculum was collected from a nearby anaerobic digestion plant.

2.2. Samples Analysis

The chemical and physical analyses were performed according to standard methods by means of a thermobalance TGA 701 LECO and a Truspec CHN LECO [28]. Moisture, ash and volatile solids content were obtained according to CEN/TS 14774; CEN/TS 14775 and CEN/TS 15148. To perform proximate analysis the samples were heated according to CEN/TS14780 and the ultimate analysis was carried out according to CEN/TS 15104. pH of substrates was measured continuously throughout the tests with a probe (Hanna Instruments HI 9124, double junction electrode, resolution 0.01). WUS, WAS, OFMSW and inoculum used in the test had the characteristics shown in Table 2.

Table 2. Characteristics of the substrates.

	Moisture (%)	Total Solids (%)	Volatile Solids (%)	Ash (%wb)	Fixed Carbon (%wb)	pH	C/N
WUS	93.97	6.03	4.33	1.7	0	7.01	12
WAS	95	5.0	3.12	1.88	0	7.3	8.6
OFMSW	76.22	23.78	19	1.9	2.88	6.25	35.8
Inoculum	97.0	3	2.06	0.94	0	7.78	13

2.3. Experimental Setup

Biomethane Potential (BMP) tests were carried by means of in house designed vessels, with a global capacity of about 1 L realized in Boro-silicate glass and equipped with a major neck connected to a pressure sensor UNIK 5000 GE Measurement & Control. In addition, minor necks with plugs to guarantee sealage during the test are used to get biogas samples and to measure pH. Biogas production is continuously derived from pressure (UNIK 5000, accuracy to 0.04% and stability typically 0.05%), and recorded for post processing. Biogas was sampled and analyzed by a gaschromatograph (490 micro GC, Agilent Technologies, Santa Clara, CA, USA), Helium and Argon were used as a carrier gas with a flow rate of 10 mL/min. Temperature of detector injector and columns were 180 °C, 100 °C, and 80 °C respectively. Biogas in excess was continuously vented to avoid pressurized conditions and explosion risks. For a throughout description of the laboratory equipment see [28–31].

2.4. Experimental Procedure

The vessels were filled up to 20% of their volume with different mixtures of WUS-OFMSW and WAS-OFMSW prepared similarly to the WAS-OFMSW mixture tested in [9,11,12,25–27].

The ratio concentrations of WUS to OFMSW and WAS to OFMSW by weight were: 50:50, 70:30 and 100% in weight respectively. The vessels were then tightly closed and flushed with N_2 to vent the air and remove O_2.Then sensors are applied and the vessels are sealed and immersed in a thermostatic bath (see Figure 2) in mesophilic conditions (approximately 35 ± 0.5 °C).

All the vessels were shaken manually two times a day (for 1 min) during the initial two weeks of the test period as recommended by Reference [18,32]. In order to avoid formation of the buffer layer within the substrate, and to insure that substrate molecules and bacterial can join, see [32]. Table 3 shows the concentration of the vessels.

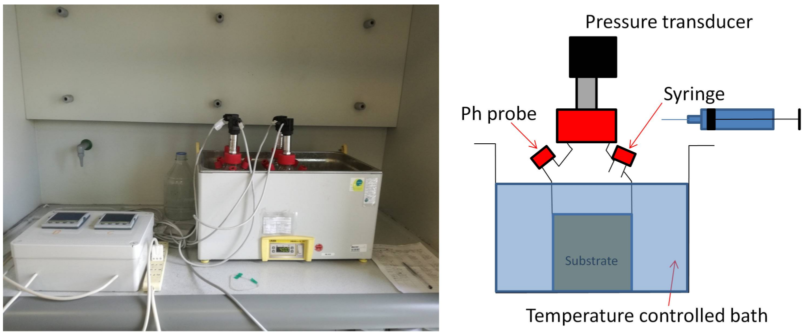

Figure 2. Laboratory equipment used in BMP tests.

Table 3. Substrates characteristics and compositions of all the vessels.

Vessels	Substrate	Moisture (%)	VS/TS	C/N	pH
1 & 2 (70%-30% weight) WUS:OFMSW	107 g WUS 46 g OFMSW 47 g Inoculum	90.6	83.3	17.67	6.9 (vessel 1) 7.0 (vessel 2)
3 & 4 (50%-50% weight) WUS:OFMSW	59 g WUS 59 g OFMSW 82 g Inoculum	90.0	85.6	19.37	6.4 (vessel 3) 6.1 (vessel 4)
5 (100% weight)	200 g Inoculum	97	68.7	13	7.8
6 (100% weight)	200 g WUS	93.97	71.8	12	7.0
1* & 2* (70%-30%) WAS:OFMSW	107 g WAS 46 g OFMSW 47 g Inoculum	91.15	81.2	15.85	6.9 (vessel 1) 7.0 (vessel 2)
3* & 4* (50%-50% weight) WAS:OFMSW	59 g WAS 59 g OFMSW 82 g Inoculum	90.28	84.56	18.37	6.9 (vessel 1) 7.0 (vessel 2)
6* (100% weight)	200 g WAS	95	62.4	8.6	7.3

Symbol "*" denotes the vessels containing WAS.

Measurements of pH were performed with a probe on the substrates every day during the initial four weeks, then performed once a week due to the relative stability pH value. During the initial phase pH value was corrected every three days by adding 1.0 mL of KOH to vessels 1 & 2, 1.3 mL of KOH to vessels 3 & 4. While the vessels which contain WAS substrate (vessels: 1*, 2*, 3*, 4* & 6*) and mono-substrate of WUS (vessel 6) did not need correction.

3. Results and Discussion

Daily and cumulative production of biogas of all vessels are presented respectively in Figure 3 and the specific of biogas and methane production in Figure 4. The duration of the test was around a hundred days. During start-up vessels 1, 2, 3 & 4 (co-mixture of WUS-OFMSW) pH decreased significantly reaching high acid values; pH control increased the production rate of the vessels 3 & 4, while production remained low for vessel 1 & 2. Total and volatile solids were determined, using a syringe and maintaining anaerobic conditions, every 20 days to track organic matter decomposition. Decomposition rates vary among WUS and WAS substrates of both co-digestion and mono-digestion. The highest rates of VS and TS were observed with WUS substrates in both types of digestion as shown in Figure 5.

Daily production of vessels 1 & 2 (70/30 of WUS-OFMSW) and 1* & 2* (70/30 of WAS-OFMSW) show similar trends. An initial phase with a low production rate is followed by a high production rate phase until a plateau is reached and rustained in the final decaying phase. However, the performance and biodegradation of substrate of 70/30 of WUS-OFMSW (vess. 1 & 2) is higher than the one of the 70/30 of WAS-OFMSW mixture of (vess. 1* & 2*). According to the biodegradation behavior of vessels of 1 & 2 (Figure 5) a slower acidogenic phase is present with respect to vessels 1* & 2*, a similar behavior was described by [25]. This affected methane production, which for vessels of 1 & 2 was measured by μGC in the range of 40–44% volume of biogas. Overall, biogas and methane production of vessels 1 & 2 are significantly higher than those produced by vess. 1* & 2* (see Figure 4 and Table 4). This test was carried out with a high buffering capacity and a balanced process for vessels l* & 2* in agreement with [33].

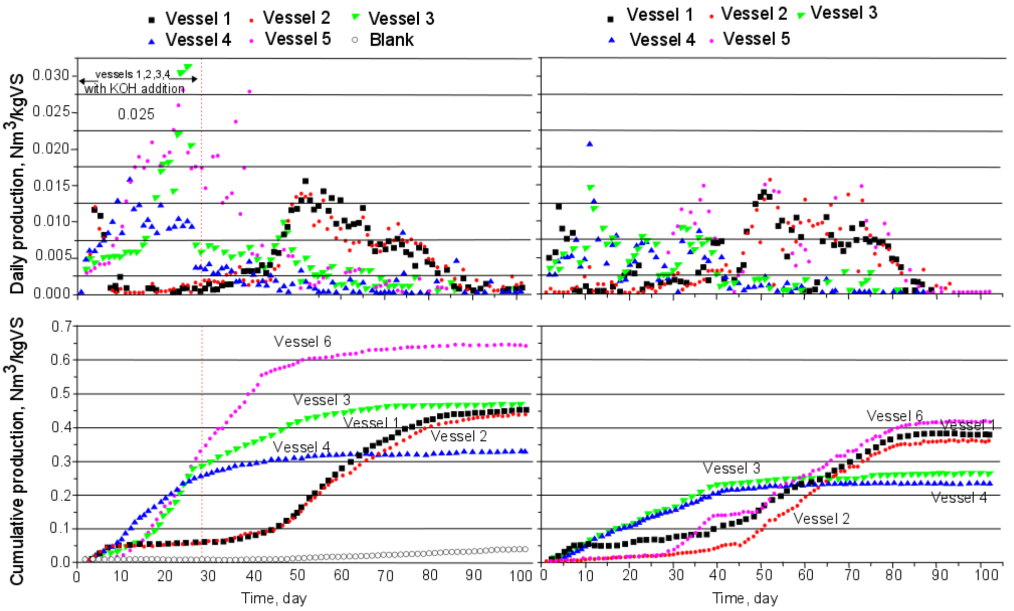

Figure 3. Daily and cumulative performance of biogas production of WAS and WUS digestion in both co-digestion and mono-digestion.

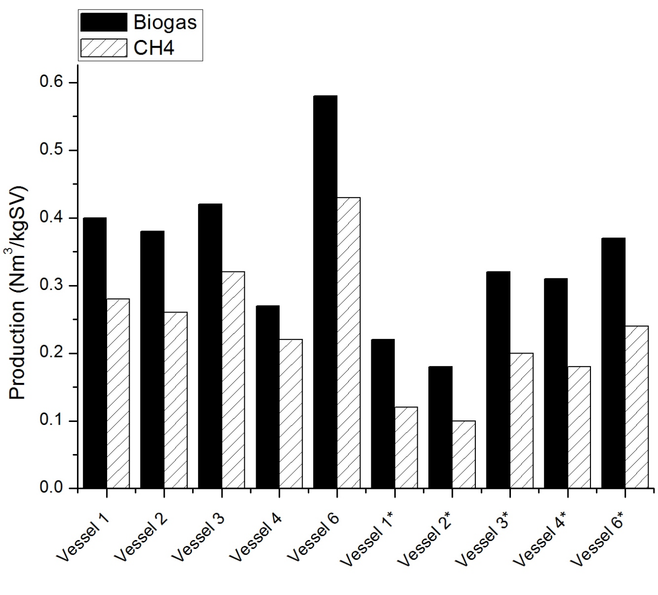

Figure 4. Specific biogas and methane production of all vessels.

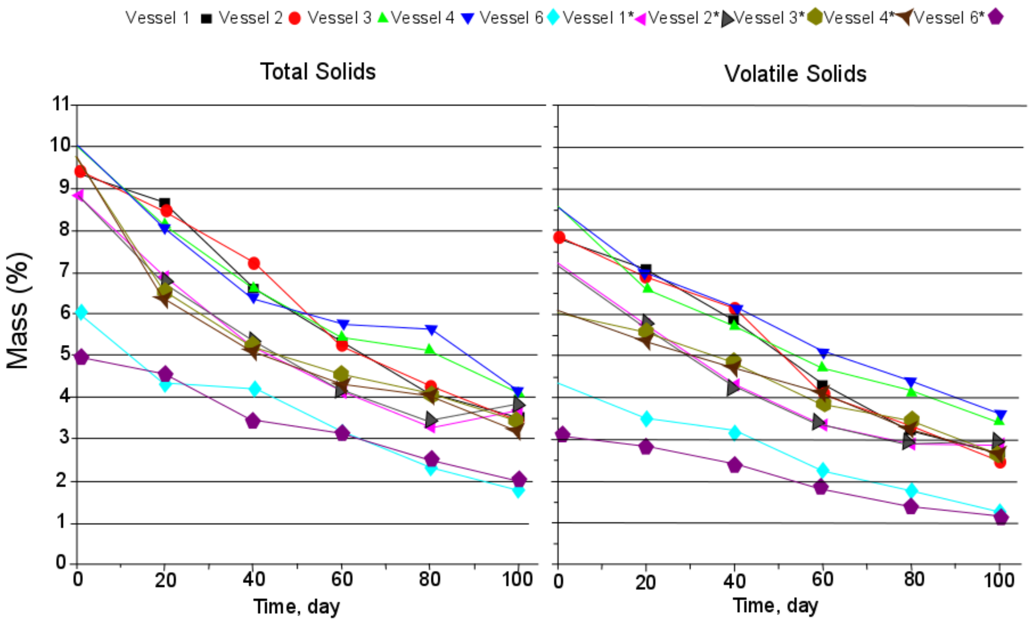

Figure 5. Total and volatile solids decomposition determined every 20 days.

Vessels 3* & 4* were characterized by slower degradable substrate compared to vessels 3 & 4 (even their 1st degradation phase started on the first day of the test). Moreover, the daily yield of vessel 4 indicated that the biogas production rate increased sharply at the beginning and then gradually decreased with constant and low rate for the last day of the test. While daily production of vessel 3 was starting to climb steeply and then drop sharply after that it became progressively stable (after day 25 till 70). Daily production of vessel 3* & 4* it shows a repeated pattern of degradation that occurs every 15 days during the test till day 45. However the vessels 3* & 4* have lower methane content than Vessels 3 & 4 (see Figure 4). In the case of vessel 4, it has the lowest yield of biogas and by tracing the degradation behavior of it, that seemed to descend back down at day 50 (see Figure 3). Moreover, in the initial phase of the test all the conditions of the process were normal, especially the high concentration of OFMSW (high acidic load) which required a correction of pH with 1.3 mL KOH and the 2nd phase was carried out with exhaustion of the buffering capacity (the carbonate system at pH value closed to 8) during the last period, a similar trend was reported by [34]. The test confirms that a neutralized substrate can be controlled (vess. 3 & 4), and that despite the acidic environment of the substrate with higher content of organic load (mixtures WUS-OFMSW) but they released production significantly. Anyway, the average yields of biogas and methane of co-mixture 50/50 of both WAS and WUS to OFMSW are lower than yields of all other mixtures and mono-digestion, due to the solid retention time which increased under the high solid concentration, that as reported by Bolzonella et al. [25], the SGP decreased from 0.18 to 0.07 m^3/kg VS fed when increasing the solid retention time in AD process. Both mono digestion process of WAS and WUS (vessels 6 and 6*) were stable and balanced with high biogas production. In fact, both substrates are characterized by lipids, so that both required a long retention time due to slow biodegradation, a similar behavior (sludge digestion) was described by [9,35]. Vessel 6* required sufficient time to reach the phase of biogas generation, vessel 6 produced more because of a higher organic load than vessel 6*. Vessel 6 achieved the highest peak of methane contents (0.035 Nm^3/kg VS) in day 40 and was more than 76.6% of CH_4 (see Figure 3). The test confirmed that significant differences of biogas yield and methane content from WUS compared to yields of WAS. However the statistical analysis of specific production of biogas and methane confirmed that the WUS and co-digestion mixture (WUS-OFMSW) yielded higher than those produced by pure sewage sludge and by the co-digestion mixture (WAS-OFMSW), as shown in Table 4. these data should be further scaled up to pilot pants scale and to industrial scale based also on the influence of Organic Loading Rate (OLR) and Hydraulic Retention Time (HRT).

Table 4. Biogas and methane yield and VS removal percentage of co-mixtures and mono-substrates, (with variance 0.00311 for biogas and 0.00273 for methane).

	Biogas (Nm^3/kg VS)	Methane (Nm^3/kg VS)	VS Removed
(WUS:OFMSW)			
(70%-30% weight)	0.444	0.331	66
(50%-50% weight)	0.399	0.315	60
(100% WUS)	0.644	0.499	72
(WAS:OFMSW)			
(70%-30% weight)	0.370	0.243	58
(50%-50% weight)	0.245	0.162	58
(100% WUS)	0.410	0.283	65

Biogas and methane SPGs of the blank (inoculum) are 0.083 Nm^3/kg VS and 0.024 Nm^3/kg VS respectively. These were subtracted from the production of the mixtures, based on the different masses of inoculum which were present. Figure 6 illustrates the linear fit of biogas and methane production of WUS-OFMSW and WAS-OFMSW.

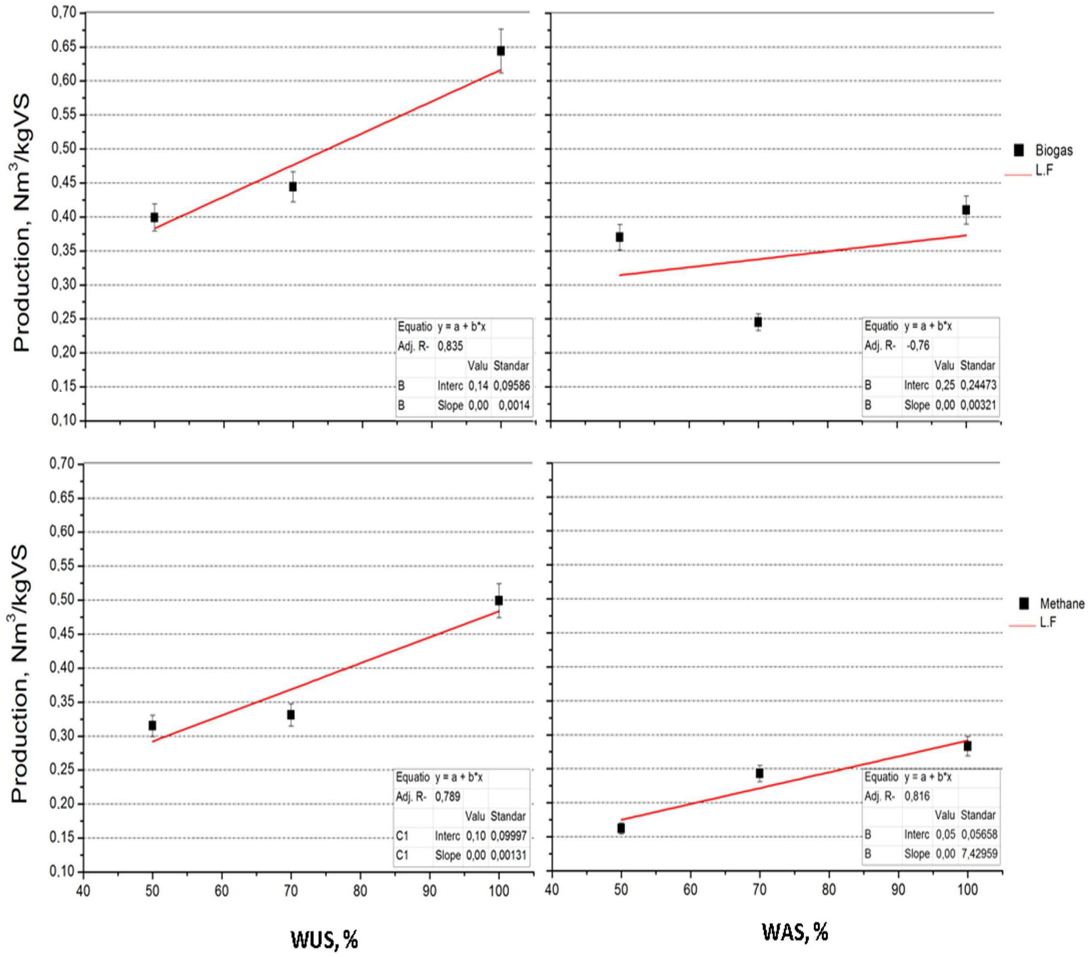

Figure 6. The relationship of WAS and WUS concentration vs biogas and methane production.

With the increase of the amount of sludge (WUS and WAS) it can be observed a significant growth in methane and biogas production, that's consistent with what mentioned in the Literature [9,15,25]. But in fact, the amount of the production growth from WUS digestion is a higher than that of WAS digestion, that explains the role of the WUS which have higher amount of VS than WAS and its role to improve the C/N ratio of the substrate. Causing ideal condition to increase the production of biogas and methane. Substrates of 50/50 have a higher hydraulic potential compared to substrates of 70/30, where the addition of OFMSW improve and accelerate the hydrolysis of WUS digestion (Figure 5), that which agrees to [36]. Vessels 1, 2, 3, 4 and 6 had a higher content of the initial organic load with respect to vessels 1*, 2*, 3*, 4* and 6* respectively, all the vessels contain WUS were characterized by a richer lipid than vessels contain WAS substrate that required a longer time for degradation. In the case of mono digestion and codigestion of WAS and OFMSW, pure WAS (vess. 6*) achieved a higher methane content than co-digestion, that agrees with [12]. The experimental results indicate that USS digestion improved the biogas yield up to 20%, 57% and 62% of 70/30 (WUS-OFMSW), 50/50 (WAS-OFMSW) and 100% of USS respect to the same ratio of WAS-OFMSW and 100% of WAS, CH_4 yield was increased up to 94% by (50/50) WUS-OFMSW. Moreover, our results which were obtained for WAS and its mixtures with OFMSW accord with results reported in Reference [9,11] in the terms of behavior and biogas/methane yield, and CH_4 yields were superior to those obtained in Reference [15,37–39]. The co-digestion between WUS and OFMSW is a suitable solution for waste management and an alternative renewable energy source from the conversion of wastes into biogas, these observation accords with [40]. However, the results indicated that the digestion of pure WUS (Table 4 and Figure 4) is the best substrate for anaerobic digestion, where it achieved the highest biogas and methane yield: 0.644 Nm^3/kg VS and 0.499 Nm^3/kg VS respectively. Additionally results indicated that a higher sewage sludge content could significantly increase biogas and methane production rate.

4. Conclusions

The experiment has shown that an 100% of WUS mono-digestion is the optimal substrate for biogas and methane production. As expected using WUS substrates, positively affects biogas and methane production, with a significant increases compared to the use of treated sewage sludge extracted after aerobic treatment, and increases biogas yield in the range of 20% to 62%, methane content in the range of 36% to 94%, and an increase in the range of 3 to 10% in removable VS of WUS-OFMSW co-digestion compared to WAS-OFMSW substrate. Biogas and methane cumulative production during co-digestion of WUS-OFMSW increases notably when increasing the WUS to OFMSW ratio. The influence of seasonal change in the characteristics of the sludge will be taken into account in future work.

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Author Contributions: The work was entirely developed and managed by Khalideh Al bkoor Alrawashdeh under her PhD project at the Biomass Research Centre, Perugia Italy. The superivors Francesco Fantozzi and Gianni Bidini have checked the work. Other colleagues have helped performing experimental campaign, such as Annarita Pugliese, Katarzyna Slopiecka, Valentina Pistolesi, Sara Massoli and Pietro Bartocci.

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

Abbreviations

The following abbreviations are used in this manuscript:

AD	Anaerobic Digestion
ASH	Ashes (%)
BMP	Bio-Methane Potential
BOD	Biochemical oxygen demand (mg/L)
C/N	Carbon to Nitrogen ratio
F.C.	Fixed Carbon (%)

FVW	Fruit and Vegetable Wastes
KW	Kitchen Waste
LCFA	Long chain fatty acids
M	Moisture (%)
n.r.	not reported
OFMSW	Organic Fraction Of Municipal Solid Waste
PS	Primary Sludge
SPG	Specific gas production (Nm_3/kg VS)
SRT	Solids retention time (days)
TAS	Thickened Activated Sludge
TS	The total content of solids (%)
VS	Volatile solids (%)
WAS	Waste of Activated Sludge
WUS	Waste Untreated Sludge

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