Pilot-Scale Anaerobic Co-Digestion of the OFMSW: Improving Biogas Production and Startup

Constantin Stan 1,*, Gerardo Collaguazo 2, Constantin Streche 1, Tiberiu Apostol 1,* and Diana Mariana Cocarta 1

1 Department of Energy Production and Use, Faculty of Power Engineering, University POLITEHNICA of Bucharest, Bucharest 060042, Romania; constantin_streche@yahoo.com (C.S.); dmcocarta@gmail.com (D.M.C.)
2 Faculty of Engineering in Applied Sciences, Technical University of the North, Ibarra 100150, Ecuador; gicollaguazo@hotmail.es
* Correspondence: stan.constantin@yahoo.com (C.S.); tiberiuapostol80@gmail.com (T.A.);
Tel.: +40-727-030-863 (C.S.); +40-744-306-966 (T.A.)

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Abstract: This paper presents experimental results regarding anaerobic co-digestion of the organic fraction of municipal solid waste and fruit and vegetable waste in order to establish the efficiency of a 2 m$^3$ volume pilot plant in terms of biogas and methane yield and stability of the process. The research study presents the feasibility of developing anaerobic digestion as an effective method for municipal solid waste management. The experiments were conducted in mesophilic conditions (35 °C). Domestic waste water was used as inoculum. The results showed that the inoculum presence, temperature, and pH control, were essential in order to improve biogas production and its composition. Using liquid inoculum, the CH$_4$ percentage in the biogas oscillated between 44% and 51%, and the biogas production from 0.504 and 0.6 m$^3$/day. Compared to domestic waste water, animal manure increased the CH$_4$ concentration in biogas (up to 63%), while the daily biogas production increased by 26% and varied from 0.693 to 0.786 m$^3$. The cumulative biogas production at the end of the experiments were 11.7 m$^3$ and 15.89 m$^3$, respectively. Using inoculum and co-digestion, the plant startup time was significantly reduced, the total solids content decreased from 22.7% to 19.8%, while the volatile solids decreased from 37.6% to 31.2%.

Keywords: biogas; OFMSW; anaerobic digestion; co-digestion; pilot-scale biogas plant

1. Introduction

Global warming and climate change represent one of the major environmental problems facing society today, mainly because of the dependency on the fossil fuel energy (88.4% out of the total energy consumed in 2015) [1,2]. The world urban population has grown approximately 5.6 times between 1950 and 2017 [3], having as a consequence the increase in quantities of waste generated. In 2017, it is estimated that 1.3 billion tons of municipal solid waste (MSW) are generated at the global level [4,5], contributing to the current environmental problems. Therefore, this requires both the diversification of renewable energy resources and proper management of MSW so as to reduce their negative impact on the environment. This is the answer to the current policies and regulations regarding the targets for greenhouse gas emissions reduction, alternative solutions for conventional energy, waste generation reduction, and energy recovery from the municipal waste [6–9].

Improper MSW management contributes to negative effects on the environment and human health [10], and hence policies and stringent national and international regulations are provided [8,10,11] that require organic fraction MSW (OFMSW) reduction in landfilling and energy recovery [9].
Due to the heterogeneous and different sizes of MSW, it is very difficult to do/deal with the physico-chemical characterization [4,5].

Generally, the biodegradable fraction of the MSW (i.e., food and biomass residues, paper, and cardboard) varies between 30% and 65% [4,5,11]. In this respect, different research [12–19] shows that MSWs are a potential primary energy source, especially the organic fraction of the municipal solid waste (i.e., OFMSW) by the amount of biogas that can be produced through anaerobic digestion (AD). Therefore, AD is seen as a proper waste management solution in terms of solving the problem of waste generation and renewable energy production [20–22].

Anaerobic digestion is a microbiological process of organic matter decomposition in the absence of air. The key process phases of AD are as follows: (1) hydrolysis, in which the complex organic molecules are converted into simple sugars, fatty acids, amino acids, and water; (2) acidogenesis, where the molecules from the previous stage are broken into organic acids, ammonia, hydrogen sulfide, and carbon dioxide; (3) acetogenesis, in which hydrogen and carbon dioxide are converted into acetic acid; and (4) methanogenesis, the final stage of AD, when the intermediate products of the preceding stages are converted into methane, carbon dioxide, and water [23]. Different research studies [24–30] showed that both anaerobic digestion and biogas production are influenced by some physical and chemical parameters such as the total solids content (TS), the volatile solids content (VS), the carbon and nitrogen ratio (C/N), the particle size, the temperature and pH, the inoculum type, and the co-digestion with other organic wastes (i.e., animal manure, sewage sludge, waste from food industry).

Anaerobic digestion has been generally used for the anaerobic treatment of one type of substrate, but in recent years, researchers have demonstrated that AD becomes more stable when using multiple types of organic waste [15,31–39]. This has been presented in several studies on the anaerobic co-digestion of the OFMSW mixed with other wastes, for example OFMSW with sewage sludge; OFMSW with agricultural residues; and OFMSW with animal manure [12–15,25,37,40–45]. An important aspect to take into account concerning the anaerobic co-digestion is the total C/N ratio from the substrate composition. Anaerobic digestion is developing under favorable conditions when C/N is between 20:1–30:1 [18,20,37]. Fruits and vegetable wastes (FVWs) represent an important residue because they are produced in very big amounts in agricultural activities and supermarkets. Their generation increases the final operational costs of markets due to sales losses and disposal costs [46]. The co-digestion of the OFMSW and FVW with animal manure represents an option for controlling the AD stability process and maximizing biogas production [46,47]. Both residues can be used as an energy source meeting the specification of European Parliament legislation (Directive 2008/98/CE), which establishes that disposal in ecological landfills must be the direction only for the residues which cannot be recycled, reused or recovered [48].

This paper’s main objective is to evaluate the performance of an anaerobic digestion pilot plant for biogas production, based on the OFMSW and FVW in co-digestion with other organic substances as an activator of methanogenesis processes. In this study, the following characteristics were assessed: the reactor start-up time, the optimization of the biogas production, and composition and the OFMSW biodegradability based on the TS and VS reduction, since the VS is the organic part of the TS that is transformed into biogas [21]. Biogas production is mainly influenced by the distribution of the TS and VS [22–24]. The experimental setup is placed in the Laboratory of Environmental Monitoring and Certification of the Energy Potential of Waste from the Faculty of Power Engineering, from the University POLITEHNICA of Bucharest.

Additionally, the paper presents statistical data regarding the waste management in Romania and Ecuador in order to estimate the potential of biogas production and to establish the feasibility of developing AD as an effective method for municipal solid waste management in these countries and also other countries that have similar waste composition. Thus, one preliminary test and two different experiments were conducted: mono and co-digestion of OFMSW and FVW with other organic waste and animal manure. The process was assessed with and without inoculum as domestic wastewater.
2. Overview of Municipal Solid Waste Management in Romania and Ecuador

An efficient integrated waste management solution involves different steps such as separate collection, transport, recycling, and landfilling with monitoring of the landfill sites after closure. The waste management responsibility goes to local public administrations. Even though there are differences in the social and economic climate between Romania and Ecuador, they present a similarity in terms of MSW generation, composition, and management [11,13,14,37], with some exceptions corresponding to the Romanian economic crisis between 2007 and 2010 (Figure 1).

In terms of MSW production, Romanian statistics show that each person generated, on average, 0.9 kg/day of waste in urban areas and 0.5 kg/day of waste in rural areas. In 2015, in Romania, the MSW annual generation rate was 4.38 million tons of waste. About 73.5% from the total waste generation was landfilled, 13.11% was recycled, and 13.39% was recovered and treated by other methods [11,12]. In Ecuador, 4.06 million tons of municipal solid waste was generated in 2015, with a waste generation rate of 0.7 kg/per capita/day. From the total waste generation, 68% was landfilled, 18% was composed, 12% was recycled, and 2% was submitted to other treatments [37]. Accordingly, in both countries the most used treatment solution for MSW remains landfilling, which is no longer an actual and sustainable solution. Figure 2 presents the similarities between the MSW medium composition in Romania and Ecuador [11,12,37].

The difference between the organic fraction of the MSW from Romania and Ecuador is almost 10%, which can be explained by the different lifestyles of the populations from these countries. The high
content of the biodegradable fraction contributes to a good potential for biogas production. Knowing that biogas production is between 80–160 m$^3$ biogas/t of waste [21], the anaerobic digestion of the OFMSW as a method of MSW treatment is an attractive choice for both countries.

3. Materials and Methods

3.1. Substrates and Inoculum

The quantity and quality of the substrate is the main factor influencing the production and biogas composition [20,33].

In the framework of the experimental study, OFMSW and FVW were used as substrates. The OFMSW was collected from a local food market. It was basically composed of raw food waste: cabbage, potatoes, tomatoes, and cucumber. Fruit and vegetable waste was collected from the fresh product department of a supermarket. The mixture of fruits that was used is composed of apples (25%), pears (25%), bananas (10%), plums (15%), apricots (15%), and peaches (10%). Additionally, dry grass and green corn stalks were used. After the collection process, the OFMSW and FVW were ground in order to reduce particles size.

The considered liquid inoculum is made up of domestic wastewater, animal manure, and organic waste. The used inoculum had a high pH indicating high buffering capacity. The buffering contributed to maintaining pH in an adequate range, refraining the accumulation of volatile fatty acids, which occurs often during the digestion of OFMSW that contains high amounts of food waste [49]. The characteristics of substrate and inoculum are presented in Table 1.

<table>
<thead>
<tr>
<th>Waste</th>
<th>Weight W [kg]</th>
<th>Percentage [%]</th>
<th>Moisture H [%]</th>
<th>TS [%]</th>
<th>VS [%] (VS/TS)</th>
<th>pH</th>
<th>C/N</th>
</tr>
</thead>
<tbody>
<tr>
<td>OFMSW</td>
<td>102</td>
<td>35.8</td>
<td>82.4</td>
<td>17.6</td>
<td>82.7</td>
<td>6.3</td>
<td>19</td>
</tr>
<tr>
<td>Fruits</td>
<td>40</td>
<td>13.7</td>
<td>92.1</td>
<td>7.9</td>
<td>87.1</td>
<td>6.6</td>
<td>37</td>
</tr>
<tr>
<td>Potatoes</td>
<td>30</td>
<td>10.2</td>
<td>84.1</td>
<td>15.9</td>
<td>85.4</td>
<td>5.9</td>
<td>34</td>
</tr>
<tr>
<td>Dry grass</td>
<td>6</td>
<td>2.0</td>
<td>24.6</td>
<td>76.4</td>
<td>81.6</td>
<td>7.2</td>
<td>12</td>
</tr>
<tr>
<td>Green corn stalks</td>
<td>35</td>
<td>11.9</td>
<td>77.3</td>
<td>12.7</td>
<td>75.3</td>
<td>6.1</td>
<td>26</td>
</tr>
<tr>
<td>Beef manure</td>
<td>40</td>
<td>13.7</td>
<td>72.6</td>
<td>27.4</td>
<td>73.8</td>
<td>6.3</td>
<td>18</td>
</tr>
<tr>
<td>Pig manure</td>
<td>40</td>
<td>13.7</td>
<td>73.2</td>
<td>26.8</td>
<td>74.3</td>
<td>7.3</td>
<td>12</td>
</tr>
<tr>
<td>Total</td>
<td>293</td>
<td>100</td>
<td>79.51 *</td>
<td>20.49 *</td>
<td>79.17 *</td>
<td>6.49 *</td>
<td>22.78 *</td>
</tr>
</tbody>
</table>

* weighted average values. OFMSW = organic fraction of municipal solid waste; TS = total solids; VS = volatile solids; C/N = carbon and nitrogen ratio.

Because of the high moisture content (83.2%), no added water was necessary for the AD process. As inoculum for the AD process, an optimized amount of 30 liters of domestic wastewater from a septic tank were used.

The presented research approach was taken as different studies [35–38] have shown that anaerobic co-digestion with different organic substrates (e.g., municipal wastewater treatment sludge and fat, oil, and grease; food waste with tall fescue; pig slurry with maize cob, etc.) has improved the AD process in terms of biogas production and composition.

3.2. Analytical Methods

The characteristics of the substrate and inoculum contribute to the determination of parameters such as the humidity, TS content, VS content, pH, and C/N ratio. To identify the humidity, TS, and VS, the thermogravimetric method was used. The pH was measured using a digital measuring device with combine electrode, type IQ SENSOR NET System 2020 XT. The humidity was measured with specific equipment, an electric heated oven, type Nitech SLW53ECO. TS and VS were measured with a calcination oven Nabertherm L9/11/SW type. By an elemental analyzer, Euro EA3000 type, the C/N ratio was measured.
The biogas composition was determined by means of a Dräger X-am 5600 gas analyzer type, which uses infrared technology to determine CH₄ and CO₂ and electrochemical sensors to determine O₂ and H₂S.

For the experimental study, a quantity of 80 kg of inoculum (i.e., animal livestock manure from pigs and cows) was used. The ratio of inoculum TS/substrate was 36.1%, which is in accordance with data from the literature specifying 30–50% [14,39]. The OFMSW/other organic wastes ratio is 1:1. In this way, we were able to evaluate the influence of the different parameters on the pilot plant performance. The total load of the reactor was 293 kg.

As shown in Table 1, the organic substrate had a high content of VS (79.17% of total solids), and consequently it had a high biogas production potential. Additionally, the C/N ratio was about 23 and a high moisture content of 79.5% increases the AD process efficiency.

On the other hand, the pretreatment of the OFMSW in co-digestion with other organic wastes consisted in particle size reduction by shredding at 4–12 mm, so that the degradation of the organic matter could be conducted under proper conditions [14,21,25].

### 3.3. Pilot-Scale Startup and Operation

The experiments were conducted using an anaerobic digestion pilot plant that was built according to a proper design. The vertical pilot plant was a dry anaerobic digester in one stage with a volume of 2 m³ with mixing and recirculation of the leachate (Figure 3). The dry process was used based on the literature data regarding the advantages of the wet AD, such as simple systems, which are easier to maintain because they do not require complex equipment (e.g., pumps, stirring systems, substrate feed in systems) [18–20].

![Figure 3. Isometric view of the pilot plant used for the neutralization/stabilization of OFMSW.](image)

The pilot plant is flexible in use in terms of the process temperature control, and it can operate at mesophilic (35 °C) or thermophilic (55 °C) conditions due to the thermal insulation with mineral wood and the electrical heating thermostatic system arranged on the entire surface of the digester for operation in cold seasons.

The reactor was operated under mesophilic conditions at 35 °C ± 2 °C. The process temperature can be automatically controlled, and the inside temperature is continuously monitored by means of temperature sensors connected to a Thermocouple Data Logger with LCD Display and USB Interface.

The main components of the pilot plant were: (1) mechanical resistance system of the plant (pilot plant support); (2) AD reactor; (3) mobile biogas capture and measuring system; (4) guidance
structure of biogas capture and measurement system; (5) feeding system; (6) temperature sensors; (7) leachate collection and recirculation system; (8) biogas sampling; (9) digestate discharge system (crank driving a screw conveyor); and (10) discharge flange.

The pilot plant could be loaded with size particles in the range of 30–50 mm, and on the bottom of the plant was the leachate collection and recycling system. Due to this system, the proper conditions for the homogeneous distribution of the microorganisms responsible for the decomposition of organic matter were maintained. The biogas composition and quantity were periodically monitored (3 to 5 times/day).

The daily biogas volume, in m$^3$, was determined by measuring the vertical movement of the reactor top (Figure 4) based on the biogas pressure inside the reactor.

![Figure 4. Float cover assembly.](image)

The biogas production was estimated using Equation (1):

$$V_{BG} = \pi r^2 h$$

(1)

3.4. Experimental Conditions

3.4.1. Experiment No. 1

This preliminary experiment was aimed at highlighting the importance of the inoculum in biogas production and to test, according to the time parameter, and whether the absence of inoculum may lead to biogas formation mainly due to the biodegradability of the substrate used (high content of VS). Therefore, the first material used in this experiment was 150 kg of OFMSW composed of raw food waste: cabbage, potatoes, tomatoes, and cucumbers. The batch experiments were conducted for a hydraulic retention time (HRT) of 21 days, and the temperature varied between 26.5 °C and 39.5 °C; the average temperature was of 34 °C to maintain the mesophilic conditions [13,21,33].

3.4.2. Experiment No. 2

In this experimental set-up inoculum was used, and an extra 48 kg of OFMSW sorted at the source was added to the reactor filled with the previous substrate experimental test. For inoculum, we used 30 liters of domestic wastewater from a septic tank. The batch experiment was conducted for an HRT of 30 days at mesophilic temperature with continuous parameter monitoring to observe the influence of the temperature and pH on biogas production [15,17,20,21,40,45,46,50–53]. The reactor operated under mesophilic conditions with leachate recirculation. The biogas production was measured from 3 to 5 times per day based on the quantity of the resulting biogas.

3.4.3. Experiment No. 3

For this batch experimental test, the digester was fed with fresh 293 kg of organic substrate, comprised of the following: OFMSW and FVW, dry grass, green corn stalks, and animal manure. The composition and physico-chemical characterization is presented in Table 1.
The retention time was of 31 days, and the reactor operated under mesophilic conditions and leachate recirculation.

4. Results and Discussion

4.1. Biogas Production

In the first experiment test, due to the absence of inoculum, the pH value varied around 6.3 and required daily value correction by adding leachate calcium hydroxide (lime). The pH was stabilized at 6.7 after a period of 16 days, indicating the possibility of the beginning of the methanogenesis phase [13,34].

After seven days from the plant startup, a gaseous emission was measured, being composed of CO$_2$, H$_2$S, and O$_2$. On day 14, the CO$_2$ was 92%, while the concentration of O$_2$ was 8%, which was inhibitive for microorganism formation. On the 18th day, the CH$_4$ measured in the gas composition was only 8%. After this, the gas production mainly consisted of CO$_2$ and rapidly decreased in time.

Therefore, in the first experimental test of the AD of the OFMSW without inoculum, the production of biogas was very low. Only gas containing CO$_2$ and other traces (i.e., H$_2$S and O$_2$) was obtained with low CH$_4$ content and noncombustible. The methanogenesis phase did not develop in appropriate conditions, due to the absence of the inoculum, and a low pH (up to 6.7) was obtained. This generated an acid environment that inhibited the formation and growth of methanogenic bacteria. This fact has also been noticed in other scientific research [17,36,38,39,41–44,54]. Under these conditions, on day 21 we decided to stop the experiment.

Using the substrate accumulated in the first experiment, liquid inoculum was added to the installation. The temperature variation throughout the second experimental test varied from 29.5 °C to 37 °C, with a medium temperature of 33.7 °C (Figure 5), while the pH varied from 6.1 to 7.4 (Figure 6).

![Figure 5. Experiment No. 2: temperature variation with time.](image1)

![Figure 6. Experiment No. 2: pH variation with time.](image2)
In the first four days of the test, the pH encountered a decrease from 6.9 to 6.1, indicating that the process was under acid phase. The methanogenesis phase began after five days from the reactor loading. The pH in this phase was constant, around the value of 7.04. For the process stabilization, the pH value was maintained at 7.7 by adding 3–5 g of calcium hydroxide (lime) per liter of leachate. To maintain the conditions necessary for a homogeneous distribution of the micro-organisms responsible for organic matter decomposition, the leachate was recirculated periodically: every three hours from the top of the plant by spraying.

The quality of the biogas was quantified in terms of CH₄ percentage. The cumulative biogas and CH₄ production, in m³, was determined by summing the daily values. The obtained results are illustrated in Figure 7.

Across the experimental activities it was observed that the daily production of biogas increased rapidly in the first 10 days of the digester loading, reaching a value of 0.58 m³. Between days 7–19, the biogas production varied between 0.5 and 0.6 m³/day. The average biogas production during this period was of 0.58 m³/day (0.261 m³ of CH₄ per day). The maximum production of biogas (0.6 m³/day) was recorded on day 14. After day 19, until the end of the experiment, the biogas production started to drop, reaching a minimum of 0.087 m³/day on day 30.

The cumulative production of CH₄ after the 30 days of the experimental study was of approximately 5.02 m³ CH₄. The quantity of biogas that can be produced per ton of waste is equivalent to about 59.1 m³ biogas (25.35 m³ CH₄). The specific CH₄ production in relation to the VS, considering the information from Table 1, is about 0.172 m³ CH₄/kg VS (0.40 m³ biogas/kg VS). The experimental results are comparable to those reported in the scientific literature. Thus, Pavi S. et al. [21], K. D. Monson et al. [26], J. Rapport et al. [27] reported that at an industrial level the specific biogas production is between 80–120 m³/tons of waste.

Other studies such as Mao et al. [55] and Velmurugan et al. [56] showed that the specific production of methane at a mesophilic and retention time (HRT) of 28 to 60 days, is between 0.2 and 0.477 m³ CH₄/kg VS for the OFMSW sorted at the source. On the other hand, Li et al. [46] and Velmurugan and Ramanujan [56] demonstrated that the specific production of biogas from vegetable waste is 0.387 m³ biogas/kg VS at mesophilic temperature and an HRT of 30 days. The differences can be explained by the fact that the experiments with the pilot plant took place outside. Consequently, there are high temperature variations, in particular during the night. Additionally, the data reported by the scientific research mentioned above refers to longer periods of study (30–60 days).

The digestate resulting from the test was analyzed in terms of TS and VS. The obtained results showed a decrease of 19.8% and 31.2%, respectively, compared to the preliminary experiment.
when inoculum was not used. Other studies, such as Forster-Carneiro et al. [57], showed that on thermophilic temperature using OFMSW sorted at the source, the TS and VS was reduced by 34.7% and 44.2%, respectively, for an HRT of 60 days. Also, Soavcool et al. [44] and Li et al. [54] reported a decrease of 41.8% of the VS at 30 °C mesophilic conditions and an HRT of 60 days. The difference that exists between the degradability is due to the temperatures and the higher HRT used in the studies mentioned.

From the experiments, we observed that the use of inoculum improved the AD process by reducing the starting time for the acidogenesis phase and that biogas production was accelerated.

In the third experiment, the temperature varied from 28.5 °C to 38 °C with an average temperature of 33.8 °C (Figure 8), while the pH varied between 5.9 and 6.9, after correction by adding calcium hydroxide (lime) (Figure 9).

We can appreciate that the acid phase lasted eight days after the loading of the installation. Once the pH was 7.1, the biogas production increased because of the methanogenic phase (Figure 10). The pH was stabilized at around 7.4 after 10 days.

![Figure 8. Experiment No. 3: temperature variation with time.](image)

![Figure 9. Experiment No. 3: pH variation with time.](image)

![Figure 10. Experiment No. 3: daily and cumulative biogas and CH₄ production.](image)
In the first 12 days, the CH₄ and biogas production reached 0.36 m³ per day and 0.693 m³ per day, respectively. The highest rate of CH₄ and biogas production was achieved between days 12–25, oscillating from 0.693 to 0.786 m³ per day. In this period of time, the average daily production of CH₄ and biogas was 0.405 m³ and 0.732 m³, respectively. The maximum CH₄ production of 0.463 m³ was reached by day 20. From day 24 until the end of the experiment period, the CH₄ production began to decline gradually. This can be explained by the fact that the vegetable and fruit waste from the substrate had a high degree of degradability and the animal manure contributed to the degradation of the OFMSW.

The cumulative production of CH₄ by day 31 of the experiment was of about 8.21 m³ CH₄. The specific biogas production related to the VS, based on Table 1, was of about 0.334 m³ biogas/kg_VS (0.173 m³ CH₄/kg_VS).

These values are comparable to the data from different past scientific research. Thus, the studies of Pavi et al. [21] showed that the specific production of biogas from the anaerobic co-digestion of the OFMSW with vegetables and fruit wastes was between 0.20–0.42 m³/kg_VS at mesophilic conditions. Also, Gashaw [19] demonstrated that the specific biogas production of the OFMSW in co-digestion with animal manure was 0.34 m³/kg_VS at a ratio of OFMSW: animal manure of 50% at 55 °C and HRT of six weeks.

We can observe that the use of the animal manure contributed to a more stable methanogenesis phase with a lower variation of the pH value. Also, the degradability degree of the substrate was substantially improved.

In Figure 11, the average cumulative biogas yields measured from mono-digestion of OFMSW with inoculum and co-digestion of OFMSW with FVW and animal manure are displayed.

![Average cumulative biogas yields of mono and co-digestion of OFMSW.](image)

The cumulative biogas yield by day 31 of the experiment was of about 15.89 m³ biogas in the case of co-digestion, while for mono-digestion of OFMSW it was 11.7 m³ biogas, which is an increase of 26%. In the case of co-digestion, the biogas cumulative production was higher, which had been expected seeing that the hydrolysis and the alcoholic fermentation of fruits and vegetables takes place at a much higher speed than the other organic substrates [30,34,35]. The mono-digestion resulted in an average cumulative biogas production lower than the co-digestion, but in every condition, the biogas production started relatively fast after feeding the reactor. This is due to the high concentration of simple carbohydrates in fruits and vegetables, which are rapidly biodegraded under anaerobic conditions [19,30,31].
The digestate characterization showed that TS and VS decreased by 22.7% and 37.6%, respectively, compared to initial ones.

4.2. Biogas Composition

In this study, the biogas composition in terms of CH₄, CO₂, and O₂ was analyzed. In Figure 11, the biogas composition variation (% in volume) over time is presented. After two days, CH₄ was identified in the gas composition, reaching up to 6%. The CH₄ concentration in the biogas increased rapidly over the next eight days up to 44%, while the pH increased (Figure 12), and the CO₂ concentrations decreased. The maximum CH₄ concentration of 51% was recorded by day 17. Between days 10 to 25, the CH₄ concentration oscillated between 44% and 51%. This indicated that the methanogenesis phase of the AD process was stable. In this period, the average concentration of the CH₄ in the biogas was 45.6%. At the end of the experiment (days 26–30), the CH₄ concentration began to decrease from 47% to 44%. The concentration of the CH₄ identified in the biogas composition was according to the values given in the specific literature (45–65%) [17,35,39,42].

![Figure 12. Experiment No. 2: Biogas composition variations over time.](image)

Throughout the experimental work, the biogas composition varied, and the H₂S was between 150–190 ppm (parts per million). During the entire methanogenesis phase, the O₂ concentration was on average of 0.25%.

In the experiment with co-digestion of the OFMSW with FVW and animal manure, we observed that the CH₄ concentration increased rapidly during the first 12 days reaching 52% (Figure 13). Afterwards, from day 13 to day 31, the CH₄ concentration varied from 44% to 63%. This shows that the methanogenesis phase was stable. During this period, the average CH₄ content was 55.73%.

![Figure 13. Experiment No. 3: Biogas composition variation over time.](image)
Also, the CO$_2$ concentration in the methanogenesis phase (days 13–31) varied from 35% to 54%, with an average of 42.3%. In the same period, the average O$_2$ concentration was 0.3%. Comparing the results with those from the previous test, we can observe that the use of manure led to an increased CH$_4$ concentration in the biogas composition of 10%, going from about 46% to 56%.

As presented in Figures 11 and 12, the average methane content of biogas produced in the pilot plant ranged between 51% and 63%. The co-digestion of OFMSW and FVW resulted in higher methane content, compared with mono-digestion. This is because of the balanced supply of nutrients that contributed to an optimal living environment for anaerobic microorganisms [46].

5. Conclusions

This experimental study demonstrated that using co-digestion of OFMSW and FVW increased the cumulative biogas yield and the cumulative methane production compared with mono-digestion. In every experimental condition, biogas production started relatively fast after feeding, 8–10 days. This is due to the high concentration of simple carbohydrates in fruits and vegetables, which are rapidly biodegraded under anaerobic conditions. On the other hand, the use of manure as well as inoculum allowed a better methanogenesis phase stability with a lower variation of the pH value which contributed to a higher CH$_4$ concentration in the biogas (from 45.6% to 55.73%). Also, biogas production increased by about 26% compared to the AD of the OFMSW with liquid inoculum (domestic wastewater).

The anaerobic co-digestion of the OFMSW with other types of organic substrates led to the AD process stability and improved the biogas composition by increasing the methane concentration and biogas generation. The average daily methane production during the methanogenesis phase was 0.405 m$^3$ compared to 0.261 m$^3$ in the case of the OFMSW AD. At the end of the experiments, the cumulative methane production was higher by about 39%, and in the co-digestion case reaching values of 8.21 m$^3$. In this way, we illustrated how the AD practice could be improved, enhancing at the same time the biogas productivities. Additionally, from the results of the digestate analysis, we pointed out that the degradability was enhanced. Thus, the TS decreased from 19.8% to 22.7% and VS from 31.2% to 37.6%.

The results obtained in this work could potentially increase the renewable energy sources in Romania and worldwide, by offering practical and appropriate options for a proper management of the OFMSW and FVW. Future developments will be focused on improving the anaerobic digestion process of OFMSW and the economic feasibility of a biogas plant. Therefore, future research will be extended in terms of: reducing HRT with environmental benefits and the optimization of the volume of the digesters; use of more efficient mechanical mixing processes; use of alkaline inoculants; improving the inculants concentrations and the environment development for methanogenic bacteria (development of biocatalysts); and AD reactor automated control systems improvement so that this can respond to changes in process parameters (pH, temperature, and others). Also, future works would study the possibility of establishing the market for AD-derived products and the decrease of AD projects in terms of costs (i.e., construction and operation of AD plants) based on regulation in force, public policies, and tax incentives.

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