Feasibility Study of Anaerobic Codigestion of Municipal Organic Waste in Moderately Pressurized Digesters: A Case for the Russian Federation

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Featured Application: The results obtained can be used to optimize the process of anaerobic bioconversion of organic waste in the Russian Federation.

Abstract: Anaerobic digestion (AD) is a promising option to obtain renewable energy in the form of biogas and reduce the anthropogenic impact on the environment. In recent years there has been increasing interest in using pressurized digesters to improve the quality of biogas. However, maintaining high overpressure increases the requirements for the explosion safety of digesters. Consequently, there are natural limitations in the available technologies and facilities suitable for full-scale operation. In this work, we aimed to evaluate the possibility of using overpressure in the digester to improve the efficiency of codigestion of common municipal organic waste–sewage sludge and the organic fraction of municipal solid waste. Three levels of moderate excess pressure (100, 150 and 200 kPa) were used to meet requirements of existing block-modular anaerobic bioreactors based on railway tanks, which are widely utilized for AD in the Russian Federation. There was no significant change in methane content in biogas (65% ± 3%) at different values of overpressure, hydraulic retention time (HRT) and organic loading rate (OLR). The maximum methane and energy production rates (2.365 L/(L·day) and 94.27 kJ/(L·day), respectively) were obtained at an overpressure of 200 kPa, HRT of 5 days and OLR of 14 kg VS/(m³·day). However, the maximum methane yield (202.44 mL/g VS), energy yield (8.07 kJ/g VS) and volatile solids (VS) removal (63.21%) were recorded at an overpressure of 150 kPa, HRT of 7 days and OLR of 10.4 kg VS/(m³·day). The pressured conditions showed better performance in terms of AD stability at high OLRs.

Keywords: anaerobic codigestion; biogas; pressurized reactor; semipilot block-modular plant; high OLR

1. Introduction

The negative impact of human activity on the environment is associated not only with the increasing consumption of natural resources but also, to a greater extent, with the for-
mation of huge amounts of organic waste from agricultural and processing industries [1–3]. The problem of disposal of sewage sludge (SS) generated in municipal wastewater treatment plants (WWTP) is becoming increasingly important and amounts to up to 50% of the current operating costs of WWTP [4]. SS is formed as a byproduct of the physical, chemical and biological processes used in wastewater treatment. The currently accepted daily rate of SS formation per population equivalent (p.e.) varies from 60 to 90 g of total solids (TS), which for the EU is about 10 million tons of TS per year [4]. In the USA, Canada and Japan, about 8, 0.4 and 2.2 million tons of TS are generated annually, respectively [5]. In Russia, according to various estimates, from 2 to 4.5 million tons of TS are formed annually in municipal WWTPs [6–10]. To reduce the volume, improve the characteristics and reduce the potential hazard to public health, primary and secondary SS should be treated to ensure the possibility of their disposal or storage.

Annually, 60 million tons of municipal solid waste (MSW) are produced in Russia as well. Organic components constitute up to 50%–60% of MSW, about half of the organic fraction is biodegradable and consists mainly of paper and food waste [11]. About 97% of the MSW produced in Russia is disposed of at landfills [11].

Among all the possible methods for the treatment and disposal of SS, such as aerobic stabilization, composting, incineration, gasification, pyrolysis, etc., the method of anaerobic digestion (AD) is one of the most preferred [12]. AD of organic fraction of MSW (OF-MSW) is a promising option to reduce the anthropogenic impact on the environment. AD process is associated with the production of biogas of higher quality than landfill gas [4], as well as biofertilizers. Anaerobic codigestion of SS and OFMSW has several benefits, including increased economic scale through increased waste, as well as increased microbial community diversity through feeding on more diverse organic waste and diluting inhibitory compounds, which helps to stabilize the digester ecosystem [13–15].

AD is a microorganism-driven process that occurs in the absence of oxygen. The AD process includes four main stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis; products formed at one stage are used as substrates at the next stage [16]. The AD process is usually carried out in one reactor, where all metabolic reactions take place [17]. According to Monnet [18], typical pressures in digesters range from 4 to 16 inches of water column (101.6–406.4 mm of water column; 0.001–0.004 MPa).

Most microorganisms in the digester are insensitive or tolerant to pressure, which means that their growth rate is practically not inhibited at pressures up to 200 kPa [19]. The pressure sensitivity of bacteria participating in the process of anaerobic fermentation and the effect of increased pressure on methanogenic activity have not been practically studied. Nevertheless, methanogens are known from deep-water basins, where the pressure can reach 100,000 kPa [20].

After the technical feasibility of anaerobic bioconversion of organic waste in digesters operating at elevated pressure was proved, further research was aimed at increasing the efficiency of the new AD systems, namely, through increasing the organic load under elevated pressure [19]. It should be noted that when compressed gas is supplied to the digester, the concentration of dissolved carbon dioxide increases. Dissolved carbon dioxide is an electron acceptor from hydrogen in the process of hydrogenotrophic methanogenesis (the main route of methanogenesis in thermophilic fermentation). Thus, dissolved carbon dioxide reduces the partial pressure of hydrogen and thus improves the interaction between syntrophic bacteria and methanogenic archaea. As a result, the yield of methane increases [20,21]. Therefore, increasing CO₂ concentration in bulk sludge may enhance AD productivity. An increase in CO₂ concentration can be achieved by introducing flue gases, as well as increasing the pressure in the digester [20,21].

Organic loading rate (OLR) is an important operating parameter that represents the bioconversion capacity of an AD system [22]. Typically, AD of waste is carried out at an OLR of 3 to 5 kg of volatile solids (VS) per 1 m³ of the reactor per day (kg VS/m³·day) [23–27]. At low OLR values, the digester works ineffectively, and at high values, there is a risk of system failure due to overload [23–27]. It should be noted that the optimal OLR value mostly
depends on the type of substrate and the TS concentration [24,26,28–30]. Even in modern anaerobic processes, the OLR significantly affects the performance of the digester [31–34].

In [35], it was shown that the maintenance of the minimum limiting ratio of the total amount of dissolved carbon dioxide and OLR (1:40) is necessary to ensure a stable and efficient AD process. To secure this, it is necessary to reduce the outflow of gaseous carbon dioxide and increase the amount of dissolved carbon dioxide in one of the above ways. By raising the pressure in the digester to 0.15 MPa, it was possible to ensure good thermophilic AD performance at 2–3 times higher OLRs [35].

However, there are few studies on the effect of increased pressure on the performance of digesters [36]. Modern research is mainly focused on the use of high pressure in biogas technology to increase the calorific value of biogas and its further use in gas distribution networks [19,36–38].

In [19,33], it was compared the efficiency of a digester (anaerobic filter) operating at two different pressures of 9 bar (900 kPa) and 1.5 bar (150 kPa) with a stepwise increase in the OLR from 5 to 17.5 kg COD/(m³ · day). It was found that pressure does not directly affect the stability of the AD process. However, the low pH value in bulk liquid, caused by the high partial pressure of CO₂, limited the performance of the digester at high OLRs. In terms of gas quality, biogas collected in the digester at 9 bar contained a higher percentage of methane. The authors noted the need to improve the stability of the digester operating under high pressure and consider it promising to use the technology of anaerobic digestion under pressure in a large-scale plant [19,33]. However, maintaining such high overpressure values undoubtedly increases the requirements for the explosion safety of such digesters, and there are also limitations in the available technologies and facilities suitable for full-scale operation.

In this work, we aimed to evaluate the possibility of using overpressure in the digester to improve the efficiency of codigestion of SS and OF-MSW using block-modular anaerobic bioreactors based on railway tanks (60–100 m³), which are widely used as digesters in the Russian Federation (Figure 1) [39]. The investigated range of overpressure in the digester was 100–200 kPa due to the maximum operating pressure in the railway tank of 250 kPa and the nominal pressure of valves and pipeline elements of 600 kPa.

Figure 1. Typical for the Russian Federation block-modular anaerobic bioreactors based on railway tanks (with the permission of SelkhozBioGaz LLC) [39].
2. Materials and Methods

2.1. Inoculum and Substrate

Thermophilic anaerobic sludge (TS = 1.30%, VS/TS = 64.4%, pH 7.7) obtained from a Lyubertsy WWTP (Moscow, Russia) was used as an inoculum.

Sewage sludge (secondary SS) was obtained from a Lyuberetsky WWTP (Moscow, Russia). Fresh SS was collected weekly and stored in a refrigerator at 4 °C until use.

OF-MSW was simulated using feedstuff for pigs to maintain a constant composition of OF-MSW throughout the experiment, as in the previous work [40]. The feedstuff consisted mainly of cereal byproducts, sunflower meal and rape cake. Table 1 summarizes the cosubstrates characteristics, which are typical for Russian cities (information from personal communication with managers and operators of digesters of WWTPs (Lyuberetsky WWTP (Moscow, Russia), Kuryanovsky WWTP (Moscow, Russia), Nizhny Novgorod WWTP (Nizhny Novgorod, Russia))).

Table 1. Characteristics of cosubstrates.

| Parameter               | Sewage Sludge | Feedstuff for Pigs |
|-------------------------|---------------|--------------------|
| pH                      | 6.7           | -                  |
| TS, %                   | 5.29          | 91.0               |
| VS/TS, %                | 65.34         | 89.5               |
| Crude fat, %TS          | 2.3           | 2.2                |
| Crude protein, %TS      | 20.7          | 13                 |
| Carbohydrates, %TS      | 32.0          | 40.2               |
| Fiber, %TS              | 8.05          | 8                  |
| N, %TS                  | 4.3           | 2.76               |
| C, %TS                  | 34            | 43                 |
| H, %TS                  | 5.1           | 6.3                |
| C/N                     | 7.9           | 15.6               |

The substrate mixture was prepared daily for each stage as follows:
- for the startup stage—for every 10 L of SS, 70 g of feedstuff for pigs was added;
- for S-I, S-II and S-III stages—for every 10 L of SS, 450 g of feedstuff for pigs was added.

No additional chemicals were added for pH adjustment throughout the experiment.

2.2. Analytical Methods

Total solids (TS) and volatile solids (VS) content were determined by standard methods [40]. A bench-top pH meter FE20 (Mettler Toledo, Greifensee, Switzerland) was used to measure pH. The content of fats, carbohydrates, proteins and fibers, as well as the elemental composition (C, H, N), was determined as described previously [40].

The biogas composition was measured using gas probes and a gas meter (Figures 2 and 3). An additional gas chromatography (GC) analysis was performed to check the accuracy of the sensors every 2–3 days. To do this, biogas samples were taken using a 50 mL syringe from the sampling ports of the digesters and injected manually into a GC (Crystal 5000.2, Chromatek, Russia). CH₄ and H₂ were separated on Zeolite NaX 60/80 mesh and CO₂ on Hayesep Q 80/100 mesh. Helium and argon were carrier gases through steel columns (length—3 m, internal diameter—2 mm) with NaX and Hayesep Q at flow rates of 20 and 25 mL/min, respectively. The temperatures of the injector, oven and thermal conductivity detector were set to 45 °C, 60 °C and 200 °C, respectively. The gases were calibrated using an external standard method based on a standard curve of detector response to known gas concentrations [41].
Figure 2. Schematic diagram of the experimental semipilot AD plant.

Figure 3. General view of the experimental semipilot AD plant.

2.3. Digesters Configuration

Experiments on continuous AD with moderate overpressure in the digester were carried out in an experimental semipilot AD plant consisting of four identical digesters, as shown in Figures 2 and 3.

The cosubstrate mixture was fed into a 50 L cylindrical steel feeding tank. From this tank, using a screw pump (ONVM, Moscow, Russia) through a loading valve (Belimo HR230-3, Wetzikon, Switzerland), the substrate was fed into the pressure pipeline for
mixing the digester content. The digester was a sealed cylindrical steel (Steel 20) tank with a volume of 250 L and a flat bottom, operating under an overpressure of up to 400 kPa (Figure 2). The required temperature mode (thermophilic, 55.1 ± 0.5 °C) was maintained in the digester using a heater. Mixing of bulk sludge was carried out hydraulically using a screw pump (ONVM, Moscow, Russia) as follows: bulk sludge from the middle part of the digester was supplied through a pressure pipeline to the lower and upper parts of the digester to wash out the sediment and destroy the floating crust, respectively. The effluent was discharged through the discharge valve (Belimo HR230-3, Wetzikon, Switzerland) into the effluent tank, which was a cylindrical plastic (HDPE) tank with a volume of 150 L. The generated biogas was fed through a gas shutoff valve (CEME, Carugate, Italy) to a receiver, which was a gas-tight bag with a maximum volume of 50 L. The required pressure in the digester was maintained with the help of a biogas compressor (UK26-1.6M, Moscow, Russia), which supplies part of the gas from the receiver to the pressure pipeline for mixing the digester. Excess biogas from the receiver was fed to the drum gas meter (Ritter TG10, Bochum, Germany) to account for its quantity. The bench-top pH meter and OPTIMA 7 (MRU GMBH, Neckarsulm-Obereisesheim, Germany) gas analyzer were used to analyze the pH of the effluent and the composition of the biogas leaving the gas meter, respectively.

2.4. Operational Conditions

Once a day for 83 days, a daily dose of the cosubstrate mixture was manually loaded into a feed tank (Figure 2), from where it was fed into the digester by a screw pump. Daily dose of the cosubstrate mixture was 20, 30 or 40 L depending on HRT shown in Table 2. Each digester was operated at HRT shown in Table 2. Mixing in R1, R2 and R3 was turned off when a new portion of the substrate mixture was fed into the digester for the prevention of premature removal of the fresh cosubstrate mixture through hydraulic short-circuiting. Biogas samples were taken from the digesters five times a week (on weekdays) from the samplers to determine the composition of biogas.

Table 2. Experimental plant operating modes.

| Parameter          | Dimension | Value                |
|--------------------|-----------|----------------------|
| Stage              | -         | Startup S-I S-II S-III |
| Time               | d         | 1–31 32–60 61–90 91–114 |
| HRT                | d         | 10 10 7 5            |
| Mixing mode        | (minutes mixing/minutes idle) | 10/110 |
| Average influent vs. content | g/L | 35.8 73 72.6 70.2 |
| Average OLR        | kg VS/(m³·day) | 3.6 7.3 10.4 14 |
| Overpressure in digesters | kPa | 0 0 100 150 200 |

Figure 4 presents the change in OLR throughout the experiment.
3. Results

During the experiment, the average methane production rate (MPR) in the digester was the highest at 200 kPa overpressure in the range of the applied hydraulic retention times (HRT). At the 5-day HRT, the MPR at an overpressure of 200 kPa was higher than the MPR at an overpressure of 150 kPa by 3.5% (Figure 5b). During the stepwise increase in OLR in control, there was observed a deterioration in the process stability. During the stepwise increase in OLR in a digester with an overpressure of 100 kPa, an increase in the MPR was observed at the 7-day HRT, and a deterioration in the process stability at 5-day HRT (Figure 5a). A sharp increase in the MPR was observed with a decrease in the HRT to 7 days (64% in ratio to 10-day HRT) and 5 days (79% in ratio to 10-day HRT) in a digester with an overpressure of 150 kPa.

At the same time, the average methane content (MC) in biogas remained at the level of 65% ± 3% throughout the experiment in all digesters.
Methane yield (MY) in all digesters at 10 days HRT was practically the same (±3%), except for digester without overpressure (Figure 6a). However, a stepwise decrease in the HRT to 5 days led to a decrease in MY by 60%, 7% and 6% for digesters with an overpressure of 100, 150, and 200 kPa, respectively (Figure 6b). At the same time, with an HRT of 7 days, an increase in MY was observed at an overpressure of 150 and 200 kPa by 16 and 7%, respectively, compared with HRT of 10 days.

Figure 5. Dynamics of methane production rate (MPR) (a) and its average value (b) depending on the overpressure and HRT in digesters.

Figure 6. Cont.
Throughout the experiment, the pH in the digesters was in the range of 6.4–7.3 (Figure 7a), except for control and digester having an overpressure of 100 kPa (Figure 7a). At an HRT of 7 days for digester without overpressure there was a constant decrease in pH from 6.3 to 4.2. At an HRT of 5 days for digester with an overpressure of 100 kPa there was a constant decrease in pH from 6.9 to 5.2. The highest degree of vs. removal (63.21%) was observed at an overpressure of 150 kPa and HRT of 7 days (Figure 7b).
Figure 7. Dynamics of pH values (a) and average vs. removal (b) depending on the overpressure and HRT in the digesters.

The highest energy production rate (EPR) (94.269 kJ/(L·day)) and the highest energy yield (EY) (8.069 kJ/g VS) were observed at an overpressure in the digester of 150 and 200 kPa and the HRT of 5 and 7 days (Figure 8a,b).

Figure 8. Cont.
Figure 8. Average values of energy production rate (EPR) (a) and energy yield (EY) (b) depending on the overpressure and HRT in the digesters.

4. Discussion

Literature analysis shows that most studies on the effect of overpressure on various parameters of the AD process were carried out in digesters with a small volume: from 0.6 L [20] to 50 L [42]. Table 3 summarizes the results of such studies. The most similar substrate (simulated food waste) was subjected to anaerobic digestion in 3-L digesters by Kim et al. [43]. At OLR of 2.67 kg COD/(m$^3$·day) and overpressure from 1 to 3 bars, the MPR and MY values were obtained in the range of 0.74–0.75 L/(L·day) and 280 mL/g COD, respectively; pH had near-neutral values (7.4–7.5). Interestingly, the methane content increased from 52.4% to 67% with an increase in excess pressure from 1 to 3 bar. The difference in AD performances can be explained by the extremely high OLR (5.49–16.38 kg VS/(m$^3$·day) in the current work compared to Kim et al. [43].

Table 3. Comparison of AD performance using pressurized digesters.

| Feedstock       | Maize Silage | SFW | G&MS | Maize Silage and G&MS | G&MS | SFW&SS |
|-----------------|--------------|-----|------|-----------------------|------|--------|
| Digester volume, L | 50           | 3.0 | 21   | 50                    | 50   | 200    |
| Excess pressure, bar | 1–9          | 1–7 | 10–50| 1–9                   | 10–50| 0–2    |
| OLR, kg VS/(m$^3$·day) | 5.1          | 2.67| 4.49 | 5                     | 4.42–4.19| 3.6–14|
| HRT, days        | -            | -   | 1.52 | -                     | 4.07–4.19| 10–5   |
| pH              | 7.2–6.5      | 7.5–6.7| >6.5 | 7.2–6.5              | 6.65–6.5| 6.5–6.9|
| MC, %           | 67–76        | 52.4–77.4| Up to 93 | 65–75 and 70.5–77.3 | 79–90.5| 65 ± 3 |
| MY, mL/g VS     | 330–310      | 280–230| 330–260| 333–313 and 303–258 | 330–260| 56–202 |
| MPR, L/(L·day)  | -            | 750–600| (1.485–1.17) * | (1.665–1.565) * and (1.515–1.425) * | (1.459–1.09) * | 0.586–2.365 |
| AD process improvement/deterioration | increase in MC/decrease in MY | increase in MC/decrease in MY | increase in MC/decrease in MY | increase in MC/decrease in MY | increase in MPR/low MY, no change in MC |
| Reference       | [42]         | [43] | [44] | [45]                   | [46] | This study |

*–calculated; MC–methane content, %; SFW–synthetic food waste; G&MS–mixture of grass and maize silage; SFW&SS–mixture of synthetic food waste and sewage sludge.

Anaerobic digestion of maize silage alone and mixed with grass silage in a two-stage high-pressure fermentation plant [43,47] led to the production of 288–333 L CH$_4$/kg vs.
at an overpressure of 1–3 bars. The pH was in the range of 7.35–6.8 with a tendency to decrease with increasing overpressure, and the COD removal was 89.95%–95.28% with a tendency to increase with increasing overpressure [45].

The results obtained in [48] showed that an increase in pressure had a significant negative effect on the removal of COD, although the process proceeded at favorable OLR values. At an OLR of 20 kg COD/(m³·day), the pressure increase led to a decrease in COD removal from 88% at atmospheric pressure to 62% at 4 bar.

In [48], despite the increase in methane content, MPR decreased with the increase in pressure. At the same time, MY also decreased: at an OLR of 20 kg COD/(m³·day), the MY was around 330 mL/g CODremoved at no overpressure and there was a consistent decrease in MY to 270 mL/g CODremoved at an overpressure of 1 bar. At the same time, pH was in the range of 7.14–7.98 with a tendency to decrease with increasing overpressure.

Lemmer et al. [45] observed a decrease in the MY in the AD of grass/maize silage from 303.8 mL/g CODadded to 258.0 mL/g CODadded at increasing pressure from 1 bar to 9 bar.

In current study, anaerobic codigestion of SS and OF-MSW at an overpressure in digesters of 150–200 kPa was possible at OLRs of 10.5–16.5 kg VS/(m³·day), which is significantly higher than the OLR used at WWTP in the Russian Federation (3–6 kg VS/(m³·day)) (information from personal communication with managers and operators of digesters of SelkhozBioGaz LLC and WWTPs (Lyuberetsky WWTP (Moscow, Russia), Kuryanovsky WWTP (Moscow, Russia), Nizhny Novgorod WWTP (Nizhny Novgorod, Russia))).

Thus, the current findings correlate well with data obtained by other authors using various substrates. High degrees of vs. removal in the current study were presumably associated with the use of pig feed containing a significant amount of easily biodegradable organic matter as a cosubstrate. The increase in the efficiency of the AD process at excess pressure in the digester is primarily associated with an increase in the concentration of dissolved carbon dioxide. Accepting electrons from hydrogen in hydrogenotrophic methanogenesis, it reduces its partial pressure and stimulates syntrophic interactions [16].

The second reason for AD process improvements can be in better buffering capacity due to higher bicarbonate ion concentration in the bulk liquid. Higher alkalinity helps to withstand shock loads of volatile fatty acids. Herein pressurized AD may well become an essential strategy to efficiently convert high-strength, easily degradable wastes such as OF-MSW or food waste into energy, thereby maximizing performance and process stability. Successful testing of semipilot overpressurized digesters makes it possible to further scale up the process in block-modular anaerobic bioreactors based on railway tanks, which are widely used as digesters in the Russian Federation and adapted to moderate pressurization. Thus, the possibility of using moderate overpressure without any major constructive changes makes it easy to re-equip existing digesters to operate at higher organic loads while maintaining process stability.

5. Conclusions

The article assessed the possibility of using excess pressure in digesters of block-modular design under the conditions of the Russian Federation (Figure 1). For the feasibility study on the thermophilic codigestion of SS and OF-MSW, the semipilot digesters (200 L volume) were moderately pressurized (100, 150 and 200 kPa) due to the maximum operating pressure in the railway tank of 250 kPa. The created semipilot plant is based on a block-modular principle. In this case, the results can be scaled in a ratio of 1/20 to a pilot plant, and after making minor corrections, to a full-scale plant with a reactor volume of 100 m³.

Additional long-term studies in existing full-scale block-modular railway tank digesters would be worthwhile to prove the feasibility of pressurized AD technology in the field.

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