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Section: Water Engineering

# Anaerobic co-digestion of sewage sludge with fish farming waste

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# Abstract

Costly management of wastes from Estonia fish processing plants usually sends their sludge from the sludge filter press process to wastewater treatment plant or composting. To reduce the cost, the potential use of this waste for the production of biogas through the anaerobic process was investigated. Anaerobic digestion has long proven to be an efficient way for the production of a renewable fuel, biogas, which can be used as a source of energy to produce electricity or heat. This renewable energy resource can be used to reduce processing costs of plants. As a result, fish waste becomes a valuable resource instead of a waste which has tipping fee. In this study, both batch and continuous flow anaerobic digestion experiments were performed at mesophilic ( $38 \pm 1^{\circ}$ C) condition. The inoculum used was from an anaerobic mesophilic digester from municipal wastewater treatment plant (WWTP). Primary substrate was sewage sludge from WWTP and secondary substrate was sludge from Saaremaa salmon breeding farm. The mesophilic anaerobic treatment of concentrated sludge from an Saaremaa salmon breeding farm pools with total solids (TS) from 3.2 to 7.0% was investigated in a one-stage periodically stirred tank reactor at 38 °C and 20–25 days hydraulic retention time (HRT). Organic loading rate (OLR) ranged from 1.08 up to 1.22 kg volatile solids (VS)/(m<sup>3</sup>\*d). Methane yields between 223.13–370.19 m<sup>3</sup> CH<sub>4</sub>/ton VS and 4.34–8.65 m<sup>3</sup> CH<sub>4</sub>/ton were achieved. The pH-value was hold at 6.92–7.45 during the whole operation. The fertilizing value of the treated sludge was estimated to be 0.87–1.12 kg N and 0.42–0.99 kg P per ton. The biogas analysis shows that CH<sub>4</sub> content varied from 63.3 to 74.6% and CO<sub>2</sub> content ranged from 11.2 to 29.0%.

Keywords: Anaerobic treatment; fish farming sludge; energy production; biogas; sludge treatment.

# 1. Introduction

Typical sewage sludge consists of primary sludge separated from wastewater during pre-settling, and biological excess sludge from the activated sludge system. Anaerobic digestion is an appropriate technique for the degradation and stabilisation of sludge before their final disposal. In recent years, much attention has been focused on the improvement of digester biogas production, in order to upgrade their role in stabilizing the sludge and also to produce a feasible bio-energy power plant. One option for improving methane yields is co-digestion. This process is well known and resulting in much higher methane yields when glycerol, food waste and similar types of organic waste were combined with sewage sludge, cow and pig slurries at biogas plants [1], [2]. In recent years there have been many successful efforts in the co-digestion of sewage sludge with several other substrates, such as the source-sorted organic fraction of municipal solid waste [3–5], glycerol from the biodiesel industry [6], cattle manure [7], pig manure [8] and solid slaughterhouse wastes [9].

Literature on biogas plants indicates that high biogas production is positively correlated with the addition of high concentrate organic by-products. Fish farms produce large quantities of organic waste. This material can accumulate on the pool, as well as be suspended in the water column. Its composition is determined according to several parameters, such as the non-consumed scraps of feeding stuffs and excrements, or other organic droppings from fish. [10]. Sludge from fish farms has three origins: fish faeces, drum filters and biofilters. In recirculated fish farms, significant sludge is produced and has a high content of fat and volatile suspended solids. Dewatering and managing the sludge is a challenging task, as it is very unstable. Fish farming sludge is an organic, readily digestible substance which cannot be easily stored over a long period. Requirements for the storage and disposal of wastes in an environmentally safe manner have to be considered in waste management.

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An average to large land-based fish farm (1000 tons feed/year) can produce up to 15 tons of sludge (dry matter) each month equivalent to 150 m<sup>3</sup> wet sludge (10% TS in wet sludge) with approximately 200 g of suspended solids (SS) per kilogram of fish feed [11]. This sludge needs to be managed and discarded properly. Besides suspended solids, however, the sludge also contains high amounts of chemical oxygen demand (COD) and nutrients. Therefore, instead of considering sludge as a pure waste, it can also be used as a source of carbon needed for denitrification. Nitrate commonly accumulates in the production water due to the intense nitrification that has to occur in the biofilters by changing ammonia into nitrate. The micro-organisms reducing the nitrate (denitrifiers) require carbon from the sludge as an energy source to carry out the reaction.

These advantages make fish farming sludge an ideal co-substrate for the anaerobic digestion process. Recent experiments with co-digestion, applying fish farming sludge, glycerol, brewery yeast, whey, municipal solid waste, pig manure and kitchen waste to mixtures of sewage sludge, have shown a significant increase in the methane yield. The main objective of this work was to evaluate the use of fish farming sludge as a co-substrate, in order to boost biogas production during the anaerobic treatment of sewage sludge. The effect of fish farming sludge supplementation on methane yield was examined in continuous experiments, and the fish farming sludge limiting concentration in the feed for a stable digestion process was estimated (the risk of organic overloading).

# 2. Materials and methods

#### 2.1. Feedstock

Sewage sludge was sludge originating from the municipal sewage treatment plant of the city of Tallinn (population 420,000), Estonia. The sludge was stored fridge at +4°C until use. The characteristics of the sludge are summarized in Table 1. The inoculum (Inoc) was taken from the city of Tallinn WWTP biogas plant anaerobic digester what is operating at +38°C with sewage sludge.

	TS %	VS %	COD gO <sub>2</sub> /l	Ptot gP/l	N <sub>tot</sub> , gN/l	NH <sub>4</sub> -N gN/l	pН
Inoc	2.30	55.85	29.2	0.77	480	0.87	7.13
SS	2.67	69.27	36.2	0.63	400	0.42	6.49
Inoc	2.44	52.95	32.2	0.81	496	0.96	7.12
SS	3.36	64.97	38.9	0.58	422	0.54	6.03

Table 1 Main characteristics of sewage sludge used in the experiments

Fish farming sludge (FS) was obtained from a fish farming pool WWTP in Saaremaa Estonia (Table 5). These sediments that were formed by fish stool and settled fish feed. Under the study of Mizanur *et al.* tank sediment is enriched with organic matter, nitrogen, phosphorus and macro and micro nutrients as well, and hence it can be a potential fertilizer [12]. This description shows that fish tank residue is appropriate for to using it as substrate for biogas producing. In the fish pond residue there are two main nutrient sources, fish feed and fertilizers. Addition of manure and feed provides organic N and P, while inorganic form comes from chemical fertilizers. The organic form of the sediment constitutes about 35–40% of the total P [12]. Fish farming takes place inside the premises. Pools are made of concrete and plastic. Fish breeding capacity is 100 tons per year. Water temperature is 15 °C and aeration air is hold on 16.0–16.5 °C for to avoiding from steaming. Farming fish was Trout while sampling of sludge in the pond. Breeding period is 12–14 months and approximately g weight up to 1.5 Kg per fish. 1–2% of feeding material falls in sediment sludge. Contaminated water treated by Drum filter system and effluent compensated with well fresh water and some organic effluent goes back to the pools. The farm produces 50 tons sludge in a year.

Table 2 Main characteristics	of fish farming sludge
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	TS %	VS %	COD gO <sub>2</sub> /l	Ptot gP/1	N <sub>tot</sub> , gN/l	NH <sub>4</sub> -N gN/l	pН
FS	7.06	82.90	82.4	1.83	616	O.10	5.70
FS	3.27	72.23	88.2	2.43	648	0.23	5.15

#### 2.2. Experimental procedure

#### 2.2.1. Continuous experiments

Two series of continuous experiments were carried out in order to investigate: (a) the limiting concentration of fish farming sludge in the feed, (b) the methane production of the fish farming sludge-supplemented sludge during anaerobic digestion and (c) heavy metals content and sludge suitability for agriculture. First, three digesters with a working volume of 4.5 l were constructed using fiberglass. The digesters supply pipes on the top of digesters were sealed with rubber stoppers containing an influent to allow injection of wastes. Effluent port on the bottom was sealed with hose clamp to allow sludge outlet. A

water heater was used to maintain the temperature of the digesters at +38oC. The digesters were connected to gas clocks. Biogas was collected by displacement of water. The reactors were operated in a draw-and-fill mode (on a daily basis) with a hydraulic retention time (HRT) of 20 days. Initially, the reactors were inoculated with anaerobic sludge originating from the municipal biogas plant of the city of Tallinn. The feed in the reactors was sewage sludge: as sole substrate (R1), supplemented with 50% (w/w) fish farming sludge (R2), and supplemented with 100% (w/w) fish farming sludge (R3). The digesters were operated using this feed for 147 days. The reactor was fed once a day (every 24 h) with a total feeding volume of 225 ml/d, resulting in a hydraulic retention time of 20–23 d. Organic loading rate was in range 1.08 up to 1.22 kg VS/(m3\*d). The mixed liquid from the reactor was stirred periodically for 15 min, once an hour. The temperature was maintained at 38oC via water heater through water jackets surrounding the reactors. The initial feed was sewage sludge and the bioreactor was operated using this feed for 20 days. Fish farming sludge was then added to the feed so that the reactor was fed continuously with sewage sludge containing 50% fish farming sludge.

#### 2.2.2. Batch experiments

Methane production potential (MPP) tests were done with Automatic Methane Potential Test System II (AMPTS). The AMPTS II follows the same measuring principles as conventional methane potential tests which make the analysis results fully comparable with standard methods. Sample material was mixed in to 500 ml serum bottle reactors, in 400 ml amounts. Each reactor contained the individual materials, nutrient medium, and inoculum. Zheng et al. (2013) suggested that an inoculum-to-substrate ratio (ISR) of  $\geq$ 2 has never been reporter as inhibitory [13]. In these experiments we used substrate-to-inoculum ratio of 0.2 and 0.5. The serum bottles were sealed with tube clamps immediately after blow out with nitrogen (2 min). Bottles was put into incubation unit (+38 ± 0.2°C) and mixed by a slow rotating agitator. Produced biogas in each reactor goes through an individual vial containing 3 M alkali solution (NaOH). Gases such as CO<sub>2</sub> and H<sub>2</sub>S are removed by chemical reactions and CH<sub>4</sub> is the only gas that passes through unchanged. All the tests were run in duplicate. With the AMPTS II both the gas volume measurements and data logging are fully automatic during the long incubation period and experimental data is calculated and generated into a standard data sheet.

# 2.3. Analytical methods

The pH was measured by an electrode (Denver Instrument, UP-5), while total (TS) and volatile (VS) solids, total and soluble chemical oxygen demand (COD), total nitrogen (TN), ammonium (NH<sub>4</sub>-N) and total phosphorus (TP) were determined according to standard methods [14]. Gas samples from continuous experiments were took by biogas analyser (Gas Data GFM416 Biogas Analyser).

#### 3. Results and discussion

### 3.1. Continuous experiments

The methane yield of an anaerobic process depends on the amount of organics (represented by VS content) and the biochemical characteristics of the organics [13]. Therefore, it is necessary to distinguish the biochemical characteristics of the organics. Table 3 and 4 shows overviews of the VS values. As the Fig.1 shows the maximum methane production in terms of VS added took place at 100% fish farming sludge. Although due to the risk of crust formation it is not the most recommended concentration. The crust formation forms as a result of decrease of the pH value. To avoid crust formation the feeding took place every second day. Concentrations 50% and 100% are mostly influenced by the fish farming sludge. Concentrations 10% and 35.6% are mostly influenced by the raw sludge and therefore less stable. Methane production of CH<sub>4</sub> produce about 70% (fluctuates between 65 and 75).

#### Table 3. Experimental Results

Mix	Retention time,	VS in,	Per added VS,	ORL.RT	VS out, %	TS out, %
	Day		m <sup>3</sup> /tonVS	kg VS/(m3*day)		
10% FS + 90%SS	35	4.40 <b>-</b> 5.06 (4.59)	108.52-561.32 (298.01)	0.98-1.13 (1.02)	50.31-52.12 (51.21)	1.19-1.55 (1.37)
35,6% FS +	36	6.46-6.89	34.57-623.80	1.44-1.53 (1.49)	45.57-47.62	2.36-2.37 (2.37)
64,4%SS		(6.71)	(252.46)	(252.46)		
50 % FS +50%SS	72	4.82-8.07 (5.94)	56.41-537.32 (269.18)	1.07-1.79 (1.32)	50.00-49.39 (49.69)	2.29-2.38 (2.34)
100 % FS	49	4.92 <b>-</b> 5.48 (5.22)	105.03-601.34 (413.11)	1.09-1.22 (1.16)	43.38-69.01 (51.03)	1.59-2.31 (2.02)

Mix	Retention time,	VS in,	Per added VS,	ORL.RT	VS out, %	TS out, %
	Day		m <sup>3</sup> /tonVS	kg VS/(m3*day)		
10% FS + 90%SS	35	4.40-5.06 (4.59)	108.52-561.32 (298.01)	0.98-1.13 (1.02)	50.31-52.12 (51.21)	1.19-1.55 (1.37)
35,6% FS +	36	6.46-6.89	34.57-623.80	1.44-1.53 (1.49)	45.57-47.62	2.36-2.37 (2.37)
64,4%SS		(6.71)	(252.46)	52.46)		
50 % FS +50%SS	72	4.82-8.07 (5.94)	56.41-537.32 (269.18)	1.07-1.79 (1.32)	50.00-49.39 (49.69)	2.29-2.38 (2.34)
100 % FS	49	4.92-5.48 (5.22)	105.03-601.34 (413.11)	1.09-1.22 (1.16)	43.38-69.01 (51.03)	1.59-2.31 (2.02)

Table 4. Biogas Production



# Fig. 1. Methane production per added VS



Fig. 2. Production of CH<sub>4</sub>, in percentage



Fig 3. ORL during the experiments, kg VS/(m3\*day)



Fig. 4. Biogas production, cm3/day

#### Table 5. Nutrients and Minerals in Tests

	Test no. 1 Inoculum	Test no. 2 Inoc+F0.2	Test no. 3 Inoc+F0.5	Test no 4 Inoc+F0.2F35%	Test no. 8 Inoc+F0.2F50%	Test no. 11 inoc+F0.2F75%	Test no. 12 inoc+F0.2F90%	Fish farm pool sludge	Sewage sludge	Inoculum	Limit values in Estonia [16]
Dry solids %	2.39	2.31	2.22	2.35	2.36	2.44	2.31	2.92	3.2	2.41	_
Phosphorus – P %	0.044	0.067	0.057	0.045	0.055	0.052	0.053	0.042	0.099	0.027	—
Potassium –K %	0.039	0.037	0.023	0.034	0.04	0.039	0.036	0.024	0.043	0.036	-
Sulfur – S %	-	-	_	-	-	-	_	0.008	0.048	0.011	-
Zink – Zn mg/kg	11.3	15.8	14	10.2	13.6	13	13.1	7.25	25.9	5.8	2500.0
Copper – Cu mg/kg	6.57	7.04	5.35	5.23	7.49	6.62	6.45	1.73	12.3	5.02	1000.0
Mercury – Hg mg/kg	< 0.01	< 0.01	Non found	Non found	< 0.01	0.01	Non found	Non found	< 0.01	Non found	16.0
Cadmium – Cd mg/kg	0.066	0.05	0.057	0.061	0.093	0.08	0.074	0.016	0.176	0.027	20.0
Chromium – Cr mg/kg	0.671	0.812	0.617	0.522	0.741	0.694	0.747	0.064	1.6	0.353	1000.0
Nickel – Ni mg/kg	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	300.0
Lead – Pb mg/kg	0.359	0.341	0.297	0.298	0.383	0.299	0.345	0.03	0.516	0.175	750.0
N %	2.33	3.02	3.02	2.98	3.08	3.08	3.10	3.84	3.77	2.59	_
С %	24.69	_	_	-	_	_	_	40.17	32.09	26.29	_
Н%	3.49	_	_	-	_	_	_	5.59	4.50	3.66	_
Crude protein %	-	_	_	-	_	_	_	24.00	23.56	16.19	_
Crude fat %	-	-	_	-	-	-	_	11.78	6.09	2.38	-

High concentration of light metals such as calcium, sodium, potassium and magnesium are known to be inhibitory to methanogens [17]. The heavy-metal content of the processed sludge meets requirements set by the Estonian law [16]. The contents of N, P and K are in line with, or higher than those of e.g. swine or cattle manure, which should make this sludge attractive to use as a bio-fertilizer, similar results were found in study conducted in Department of Biotechnology, Lund University [17].

#### 3.2.1 Batch experiment results

Two identical batch experiments were conducted. In the first experiment the AMPTS II was operated for 42 days and in the second 21 days. The experiments operation time was reduced because the main process occurs during the first 7 days. Main characteristics of sewage sludge used in the experiments are presented in Table 1 and main characteristics of fish farming sludge in Table 2. The theoretical value for the production of methane by VS was calculated to be 393.91 m3CH4/tVS and the production from wet weight 9.29 m3/m3. The calculations were made using 100% TS, crude protein, crude fat and carbohydrates [18]. Tests results are expected to be lower than the theoretical calculations. It is due to the instability of the anaerobic digestion process and the degradability of the organic matter. All organic matter is not easily decomposable and may need thermal pre-treatment.

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Fig.5. Flow rate [Nml/day], Tests set no. 1



Fig.6. Accumulated gas volume [Nml], Tests set no. 1



Fig.7. Flow rate [Nml/day], Tests set no. 2



Fig.8. Accumulated gas volume [Nml], Tests set no. 2

At the first day the gas flow rate was the highest. After 10 days the average gas flow rate was 5.50 [Nml/day] and after 15 days the average gas flow rate went down to 2.29 [Nml/day]. (Using only inoculum was not taken into account in these calculations). From the figures 7 and 8 it can be seen that the main volume of accumulated gas is formed after 7 days. After

7 days the change in accumulated gas volume was minimal. This result is similar to a study conducted at Technical University of Lisbon where after 10 days, 81% of the total biogas was formed [19].

Table 6. The minimal and maximal gas flow and for each test mix combination used

Mix	Max [Nml/day]	Min [Nml/day]
(Inoculum) Flow [Nml/day]	61.07	1.40
Inoc+FS 0.2 Flow [Nml/day]	205.25	0.90
Inoc+FS 0.5 Flow [Nml/day]	310.85	1.40
Inoc+SS+FS 2,5% Flow [Nml/day]	188.20	0.90
Inoc+SS+FS 5% Flow [Nml/day]	193.05	0.50
Inoc+SS+FS 10% Flow [Nml/day]	190.65	1.00
Inoc+SS+FS 15% Flow [Nml/day]	177.60	0.95
Inoc+SS+FS 35% Flow [Nml/day]	174.55	2.60
Inoc+SS+FS 50% Flow [Nml/day]	191.55	2.80
Inoc+SS+FS 75% Flow [Nml/day]	156.80	3.00
Inoc+SS+FS 90% Flow [Nml/day]	197.55	1.70

The most effective gas production was when the substrate/inoculum rate was 0.5. The least effective was using only inoculum. The proportion of 0.2 was tested more thoroughly to find out what results if using fish and raw sludge give. The best gas flow came from Inoc+SS+FS 90% (197.55 Nml/day) and the lowest was Inoc+SS+FS 35% (147.55 Nml/day). The first and the second test sludge may be slight different due to the difference of the fish feed used. Also the TS and VS were different during the tests.

Table 7. Characteristics and outcomes from tests set no. 1

Tests	Dry compon	ents in pulps	[g]	Production of methane			
	Inoculum	FS	Sewage sludge	Substrate (VS)	Inoculum (VS)	Production by wet weight m <sup>3</sup> /m <sup>3</sup>	Production by VS m <sup>3</sup> CH <sub>4</sub> /tVS
Inoc	400			0.000	5.143		
Inoc+FS 0.2	383.16	16.84		0.985	4.926	14.23	243.31
Inoc+FS 0.5	360.39	39.61		2.317	4.634	21.66	370.19
Inoc+SS+FS2,5%	353.32	1.17	45.51	0.908	4.543	4.34	223.13
Inoc+SS+FS5%	355.34	2.23	355.34	0.914	4.569	5.05	246.91
Inoc+SS+FS10%	358.91	4.12	36.98	0.923	4.615	5.45	242.72
Inoc+SS+FS15%	361.95	5.71	32.34	0.931	4.654	5.69	232.45

#### Table 8. Characteristics and outcomes from tests set no. 2

Tests	Dry compon	ents in pulps	[g]			Production of methan	Production of methane		
	Inoculum	noculum FS		Total solids (TS)	Volatile solids (VS)	Production by wet weigh m <sup>3</sup> /m <sup>3</sup>	Production by VS m <sup>3</sup> CH <sub>4</sub> /tVS		
Inoc	400			0.000	5.166				
Inoc+FS0.2	360.52	39.48		0.931	4.656	8.44	357.94		
Inoc+FS0.5	314.02	85.98		2.028	4.056	8.65	366.88		
Inoc+SS+FS35%	358.70	14.46	26.85	0.927	4.633	5.57	248.14		
Inoc+SS+FS50%	359.14	20.43	20.43	0.928	4.638	6.71	295.47		
Inoc+SS+FS75%	359.84	30.12	10.04	0.929	4.648	4.21	181.94		
Inoc+SS+FS90%	360.25	35.78	3.98	0.930	4.653	7.40	316.03		

#### 4. Conclusions

The study showed that the potential use of this substrate for the production of biogas through the anaerobic process technology is promising. The co-digestion increased the methane yields, biogas production and also stabilized the process. The sludge would be attractive to use as a bio-fertilize in agriculture. Due to the risk foaming of crust, further tests are needed for using 100% fish farming sludge with residence time of 20 days. It should be investigated what is the reason for

the decrease of the pH value because under these conditions was noticed drop of pH and crust formation. The fish pool sludge should also be tested using different fish feeds, since the sludge properties are conditioned by the feed properties.

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