

STABILIZATION OF ANIMAL BY-PRODUCTS IN ANAEROBIC CONDITIONS COUPLED WITH DIGESTATE MEMBRANE POST-TREATMENT

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Abstract

The aim of the study was to establish the most appropriate technological parameters for anaerobic digestion of non-straw-bedded pig manure. The influence of anaerobic digestion on the susceptibility of digested manure to separation into liquid and solid phase was also taken into consideration. It was established that anaerobic digestion of the manure in conditions of high ammonia concentration ($5790 \div 5940 \text{ mg NH}_4^+/\text{dm}^3$) turned out to be the most effective for the HRT value of 30 days, which corresponds to the OLR at the level of $1.57 \text{ kg VS}/(\text{m}^3 \cdot \text{d})$. In this condition, the highest biogas production ($0.86 \text{ m}^3/\text{m}^3 \cdot \text{d}$) as well as the most appropriate indices of organic matter reduction (VS: 45%; COD: 57%) were achieved. Since the digestion effluent exhibited a high content of biogenic and organic substances, the digested manure after centrifuging was treated with membrane processes combined with nanofiltration (NF) and reverse osmosis (RO) – in separate runs. Application of reverse osmosis ensured a high removal of pollutants. However, the permeate exhibited too high concentration of ammonia-nitrogen, which made it impossible to release into natural water reservoir without infringing the law. It was suggested that manure leachates should be pre-treated in a process geared towards the removal of ammonia. The precipitation of ammonium magnesium phosphate ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$), known as struvite, seems to be an interesting solution to the problem, as it enables to a concurrent binding of both nitrogen and phosphorus. The other process used, i.e. nonofiltration (NF) turned out to be ineffective as regards the degrees of contaminants removal.

Streszczenie

Celem prezentowanych badań było ustalenie najkorzystniejszych parametrów technologicznych fermentacji metanowej gnojowicy świńskiej pochodzącej z hodowli trzody chlewnej. Dodatkowo określono wpływ fermentacji beztlenowej na podatność materiału przefermentowanego na frakcjonowanie na frakcję stałą i płynną. Za najkorzystniejszy hydrauliczny czas zatrzymania przedmiotowej gnojowicy w bioreaktorze uznano wartość 30 dni, która odpowiadała obciążeniu komory bioreaktora ładunkiem s.m.o. na poziomie $1.57 \text{ kg}/(\text{m}^3 \cdot \text{d})$. Dla tych warunków prowadzenia procesu odnotowano najwyższą dobową produkcję biogazu ($0.86 \text{ m}^3/\text{m}^3 \cdot \text{d}$) oraz najwyższe stopnie biokonwersji materii organicznej (s.m.o.: 45%; ChZT: 57%). W związku z tym, że wody odciekowe po procesie frakcjonowania gnojowicy przefermentowanej charakteryzowały się znacznym obciążeniem biogenami oraz związkami organicznymi, podjęto próby ich membranowego oczyszczania z zastosowaniem procesów nanofiltracji i odwróconej osmozy. Oczyszczanie wód pofermentacyjnych w procesie odwróconej osmozy zapewniło wysoki stopień usunięcia ładunku zanieczyszczeń. Oczyszczone wody pofermentacyjne charakteryzowały się jednak zbyt wysokim stężeniem azotu amonowego, co ostatecznie uniemożliwiło ich bezpośrednie odprowadzenie do odbiornika naturalnego. Wobec powyższego zasugerowano wstępne podczyszczanie wód odciekowych przed procesem odwróconej osmozy mające na celu obniżenie stężenia azotu amonowego. Interesującym rozwiązaniem umożliwiającym jednocześnie związanie azotu oraz fosforu wydaje się być proces strącania fosforanu amonowo-magnezowego o potocznej nazwie struvit. Oczyszczanie wód pofermentacyjnych z zastosowaniem procesu nanofiltracji okazało się nieefektywne.

Keywords: Anaerobic digestion; Biogas; Membrane processes; Nanofiltration; Reverse osmosis; Pig manure.

1. INTRODUCTION

Animal by-products, straw and after-harvest leftovers are considered to be natural fertilizers meant first and foremost for the application in agriculture. Liquid manures require only a limited pre-treatment before they are agriculturally applied. The process consists of chemical composition homogenisation and removing larger solid particles [1]. According to the law [2-3], animal by-products should be applied in the way which does not negatively affect the environment. It is necessary to comply with permissible doses (max. 45 m³/ha), application periods (1st March-30th November), agro-technical requirements as well as storage conditions. Moreover, liquid manures should be stored in leak-proof containers with the capacity sufficient to keep the manure generated over a period of at least 4 months. During the period, the manure undergoes natural decomposition which leads to the release of gases into the atmosphere, mainly CO₂, CH₄, NO_x, NH₃ [4-5]. Especially, ammonia released during excretion and storage is associated with agricultural activity. In areas with high – intensity animal production, about 70-80% of ammonia emission is generated by farming-related sources [4, 8]. Apart from causing atmospheric pollution, manure leakages may cause soil as well as surface and ground waters degradation [4, 6-7].

Farms rearing animals on a massive scale (> 2000 animal standings for pigs weighted more than 30 kg or 750 standings for sows) are obliged to use at least 70% of the manure generated as natural fertilizers in their own land. The remaining part can be donated or sold on condition that it will be also used as natural fertilizers. Significant concentration of stock-raising farms as well as their dense distribution, make it difficult to utilize the generated manure for their own purposes. Besides, transportation of liquid manures to the areas suffering from their deficiency, is very expensive [1,8].

The manure which is not directly used as natural fertilizers is considered as wastes and has to be treated with appropriate methods to reduce the negative influence of the raw manure on the environment [1-2]. Manure may, for example, be treated in the process of controlled anaerobic digestion. The process allows to generate renewable energy – in the form of biogas as well as stabilized digested biomass. Besides, the process eliminates odorous nuisance and has a positive impact on separation of the digested manure into solid and liquid phase. The solid part can be more efficiently transported into areas suffer-

ing from natural fertilizer deficiency at a lower cost while the liquid fraction can be used as organic fertilizer within the farm generating it. Such liquid fractions are rich in ammonia, which is easily absorbed by plants. Whereas, excessive amounts of liquid phase can be treated additionally. Depending on the treatment efficiency, it can be used as technological water within the farm or released into a natural water reservoir as long as its composition does not violate the norms specified in legal regulations [11].

The aim of the study was to establish the most appropriate technological parameters for anaerobic digestion of non-straw-bedded pig manure. The mesophilic digestion for biogas production was conducted at the hydraulic retention times (HRT) ranging from 20 to 40 days. The criteria taken into account while assessing the applied HRT values included: biogas production indices, degrees of organic matter decomposition and indices of process stability. Moreover, the influence of the anaerobic digestion on the separation properties of the digested manure was evaluated. Since the digestion effluent exhibited a high content of biogenic and organic substances, the digested manure after centrifuging was treated with membrane processes, i.e. nanofiltration (NF) and reverse osmosis (RO) – in a separate run.

2. MATERIALS AND METHODS

In this experiment, pig manure from a non-straw bedded rearing farm was used as a digestion feedstock. The samples were collected at the outlet of the pipe connecting animal standings with manure storage tanks. Table 1 presents the physical and chemical properties of the raw manure.

The digestion process was conducted in a bioreactor with a working volume of 3 dm³. The digester was maintained at a constant temperature of 36°C (±0.5). The process was carried out at the following hydraulic retention times (HRT): 20, 25, 30, 35 and 40 days. The applied range of HRT corresponds to the organic loading rate (OLR) value of between 1.18 and 2.35 kg VS/(m³·d). The digesters contents were mixed periodically – 5 minutes in every 3 hours.

Nanofiltration (NF) and reverse osmosis (RO) were conducted in the device type GH-100-400, produced by the US-based company Osmonics. Its capacity amounted to 400 cm³. The device worked in the dead-end mode, on flat membranes with the active volume of 36.3 cm². In case of NF process, cellulose membrane type SF10 was used while for RO – a polyamide one type ADF was applied. The processes

Table 1.
Characteristics of the raw manure

Indicator	Unit	Raw manure	
		Range of value	Average value
pH	-	6.6 ÷ 7.3	6.9
TS	g/dm ³	57.2 ÷ 62.5	59.0
VS	g/dm ³	45.8 ÷ 49.2	47.2
BOD ₅	mgO ₂ /dm ³	23550 ÷ 26070	24450
COD	mgO ₂ /dm ³	26650 ÷ 37090	30765
BOD ₅ /COD	-	0.70 ÷ 0.88	0.79
Ammonia nitrogen	mgNH ₄ ⁺ /dm ³	4565 ÷ 5726	5414
Total nitrogen (Kjeldahl)	mgN/dm ³	4615 ÷ 5820	5485
Phosphates	mgPO ₄ ³⁻ /dm ³	1321 ÷ 1576	1495
Total phosphorus	mgP/dm ³	1369 ÷ 1625	1555

Table 2.
Characteristics of the digested manure

Indicator	Unit	HRT [days]				
		20	25	30	35	40
pH	-	7.6	7.8	7.9	7.9	7.9
TS	g/dm ³	41.2	39.4	36.0	36.9	37.0
VS	g/dm ³	31.4	28.8	25.8	27.2	27.1
COD	mgO ₂ /dm ³	24420	14350	13080	12950	14370
Ammonia	mgNH ₄ ⁺ /dm ³	5940	5790	5810	5925	5895
Phosphates	mgPO ₄ ³⁻ /dm ³	1093	993	855	853	838
VFA	mgCH ₃ COOH/dm ³	7250	3830	3155	3475	3792
Alkalinity	mgCaCO ₃ /dm ³	12350	12255	13735	13655	14365

were conducted at the trans-membrane pressure of 2 MPa. The rotary velocity of the stirrer for both processes was maintained at the level of 200 rpm/min. The scope of the analyses conducted included: pH value measurement and determinations of total solids (TS), volatile solids (VS), chemical oxygen demand (COD), biochemical oxygen demand (BOD₅), total Kjeldahl nitrogen (TKN), ammonia nitrogen (NH₄⁺), phosphates (PO₄³⁻), total phosphorus (P_{tot}), sulphates (SO₄²⁻), total volatile fatty acids (VFA) and total alkalinity (TA). The susceptibility of the digested manure to separation into solid and liquid phase was based on the capillarity suction time

(CST) measurement. Both quantitative and qualitative analyses of the biogas produced were carried out during the experiment. The biogas was stored in a plexus tube containing 5% NaOH solution. The recorded amounts of biogas were adjusted to the volume at standard temperature (0°C) and pressure (1 atm). The biogas was periodically analysed for CH₄ content (% vol.) [12-14].

3. RESULTS AND DISCUSSION

The process of methane fermentation was conducted under high ammonia concentration ($5790 \div 5940 \text{ mgNH}_4^+/\text{dm}^3$), successfully increasing the hydraulic retention time (HRT) of the manure in the bioreactor from 20 to 40 days. In the paper, the influence of particular HRT values on the degree of organic matter reduction, biogas production and stability of the process was discussed. Physical and chemical characteristics of the digested manure is presented in Table 2.

Methane fermentation is one of biological methods of waste utilization. Its greatest strength is the capacity to generate renewable energy in the form of biogas. The organic content is the key factor behind wastes susceptibility to biodegradation. It is assumed that wastes characterized by organic matter exceeding 30% can be treated with the process of anaerobic digestion. What is more, the ratio of BOD/COD is also crucial. The value of the latter indicator exceeding 0.6-0.7 may signify high biodegradability [15]. Taking into account the value of the indicators mentioned above, it may be acknowledged that the analysed manure exhibits a high degree of biodegradability (VS/TS – 80%; BOD₅/COD – 0.8).

The process of methane fermentation was initially adopted by the application of the shortest HRT value, i.e. 20 days, which was tantamount to $2.35 \text{ kg VS}/(\text{m}^3 \times \text{d})$ of bioreactor organic loading rate (OLR). Under those conditions, low organic matter reduction was recorded (VS = 33.5%; COD = 20.6%). Furthermore, as the HRT value was subsequently extended, reduction degrees of VS and COD increased (Fig. 1).

The maximum VS reduction, i.e. 45.3% was achieved when the HRT was extended to 30 days, which was associated with a decrease in OLR value to $1.57 \text{ kg}/(\text{m}^3 \times \text{d})$. Further increases in HRT value (35 ÷ 40 days) did not impact the parameter in a positive way. In case of COD reduction, it was shown that the most appropriate value was achieved for the HRT in the range of 30 ÷ 35 days. The application of the highest value of HRT (40 days) did not impact the organic matter bioconversion, expressed as COD reduction (Fig. 1).

During the experiment, the biogas production as well as methane content in the biogas generated was recorded. Figure 2 shows the amount of biogas produced for various HRT, expressed in terms of daily production and biogas yield. Conducting the process for the lowest value of HRT, i.e. 20 days, daily biogas production and biogas yield amounted to

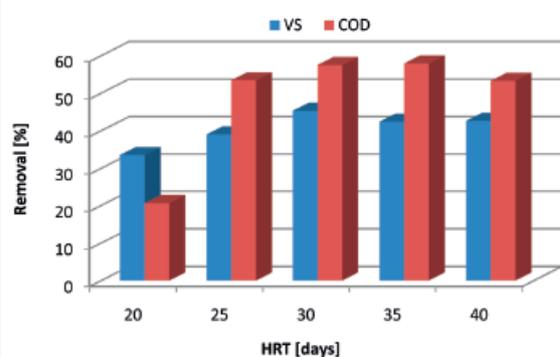


Figure 1.
The influence of various HRT values on organic matter biodegradation

$0.59 \text{ m}^3/\text{m}^3 \times \text{d}$ and $0.25 \text{ m}^3/\text{kg VS}$ respectively. The highest daily biogas production, i.e. $0.86 (\text{m}^3/\text{m}^3 \times \text{d})$ was recorded for the HRT of 30 days. When the HRT value was extended to 35, the daily biogas production decreased by about 10% ($0.78 \text{ m}^3/\text{m}^3 \times \text{d}$). Whilst the biogas yield achieved the highest value ($0.58 \text{ m}^3/\text{kg VS}$) in those conditions. Similarly to the organic matter decomposition, the application of the highest HRT (40 days) did not have a positive influence on both biogas production indices.

It was established that the HRT value did not impact the content of methane in biogas produced. The average CH₄ content amounted to between 74% vol. and 76% vol. A high content of CH₄ was ascribed to the digestion feedstock rich in proteins, which are believed to produce biogas of the highest CH₄ proportion [16-17].

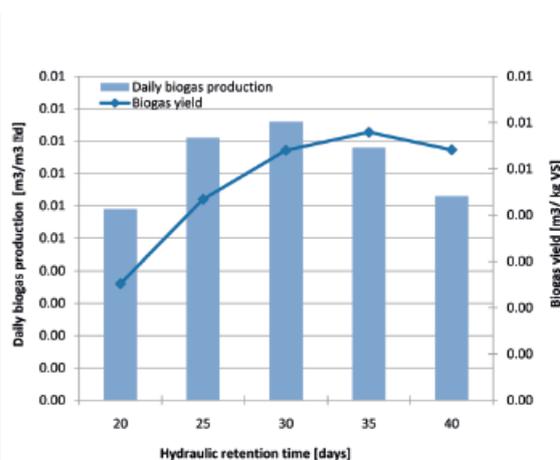


Figure 2.
The influence of various HRT values on biogas production indices

Table 3.
Factors influencing the stability of the methane fermentation

HRT [days]	pH [-]	NH ₄ ⁺ [mg/dm ³]	VFA [mg/dm ³]	Alkalinity [mgCaCO ₃ /dm ³]	VFA/TA [-]
20	7.6	5940	7250	11350	0.64
25	7.8	5790	3830	12255	0.31
30	7.9	5810	3155	13735	0.23
35	7.9	5925	3475	13655	0.25
40	7.9	5895	3792	14365	0.26

Discussing the overall stability of the anaerobic digestion, a primary indicator which influences the biological conversion is pH value. The use of the pH as a process indicator is based on the fact that the pH drop is commonly related to the VFAs accumulation [18-20]. However, if anaerobic digestion is conducted at a high concentration of ammonia, which is mainly generated in proteins mineralization, the ammonia released counteracts the decrease of pH value to a certain extent. What is more, a significant increase in acidity of digested biomass does not take place until the process collapses and the acidic phase dominates. Taking into account the above facts, a more reliable stability indicator seems to be a volatile fatty acids to total alkalinity (VFA/TA) ratio. If the latter exceeds the threshold of 0.3÷0.4, it is believed to have an inhibitive effect on biogas production or can even lead to the collapse of the process [18, 21-22]. Taking into account the achieved values of the VFA/TA ratio, it can be pointed out that the process exhibited stable properties for the HRT value above 25 days – Table 3.

In areas with intense live-stock raising, the amount of manure generated often exceeds plant requirements for nutrients. It seems reasonable to separate the manure into liquid and solid fraction. The separation is justified on the following grounds [1,9]:

- separation of valuable solid fraction, which can be used directly, as an organic fertilizer or treated further, e.g. in the process of composting,
- reduction of odorous nuisance,
- improvement of hydraulic properties of the digestate and
- reducing transportation costs in case the manure is used outside the farm where it originated.

Considering the above, the research encompassed the analysis of the influence of methane fermentation

on the separation properties of the digested manure. Figure 3 shows results of CST test recorded for both raw and digested manure at various HRTs. As compared to the raw manure (524s), the digested manure exhibited a significantly lower values of the CST (274÷310 s). It allows to conclude that the anaerobic digestion affects the separation properties of the digested manure in a positive way.

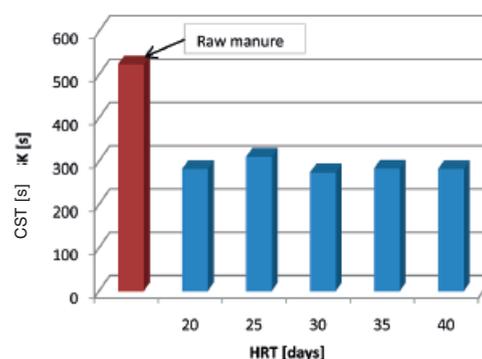


Figure 3.
Results of CST test recorded for raw and digested manure

After the separation of manure into solid fraction, which can be directly used as a natural fertilizer or treated further, we are left with liquid fraction, i.e. manure leachate. Due to its significant contents of biogenic and organic substances, the leachate was treated by means of membrane techniques, i.e. nanofiltration (NF) and reverse osmosis (RO) – in a separate run. The pH value was adjusted to the level of about 7 before the leachate underwent the membrane processes. As it is commonly known, at the pH value of the digested manure amounting to about 8, more than 5% of the nitrogen constitutes its unionized form, which is not detained by osmotic membranes. Whilst for the pH value of one unit lower (pH = 7), less than 1% nitrogen is unionized [23].

Table 4.
Characteristics of the manure leachate after treatment with the application of membrane processes

Indicator	Manure leachate after digestion (HRT = 30 days) and pH value adjustment (H ₂ SO ₄)	Permeate				Maximum permissible limits [11]
		NF		RO		
		Value	Degree of reduction [%]	Value	Degree of reduction [%]	
pH [-]	7.0	7.5	-	7.7	-	6.5÷9.0
Total phosphorus [mg P/dm ³]	182.6	6.4	96.5	0.73	99.6	1
Ammonia nitrogen [mg NH ₄ ⁺ /dm ³]	5670	2138	62.3	794	86.0	10
Total nitrogen [mg N/dm ³]	5980	2153	64.0	688	88.5	
Sulphates [mgSO ₄ ²⁻ /dm ³]	7100	2050	71.1	140	98.0	500
COD [mgO ₂ /dm ³]	13700	6794	50.4	110	99.2	125

Dependency of the volumetric permeate fluxes on the length of nanofiltration and reverse osmosis processes is presented in Figure 4. In the beginning of the process, volumetric permeate fluxes exhibited the value of $7.4 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$ and $3.5 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$ for the NF and RO respectively. As it was expected, the volumetric flux of the nanofiltration exhibited a higher value. After 5 hours of the process the value decreased significantly and amounted to $1.3 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$.

Due to the high content of pollutant, the volumetric flux characterizing the RO process decreased sharply throughout the process. Its value amounted to $1.0 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$ and $0.7 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$ after 2 and 4 hours of the process respectively – Figure 4.

Changes in quality indicators characterising the digested manure after membrane treatment are presented in Table 4. The RO process turned out to be very effective in terms of COD and phosphorus removal (>99%). Their final values were at the level allowing the release of the treated permeate to a natural reservoir. Despite a relatively high degree of ammonia nitrogen removal – 86%, its concentration in the permeate amounted to $794 \text{ mg NH}_4^+/\text{dm}^3$, which is much higher than the normalized value allowing release to the natural reservoir.

The treatment of the manure by means of nanofiltration turned out to be ineffective. The degree of COD and nitrogen compounds removal amounted to 50.4% and 62.3÷64.0% respectively. In spite of the fact that a degree of phosphorus removal exhibited a high value, i.e. 96.5%, its concentration in the permeate was several times higher ($6.5 \text{ mg}/\text{dm}^3$) than the norm.

The research project allows to draw the following conclusions:

4. CONCLUSIONS

The research project allows to draw the following conclusions:

1. The process of methane fermentation of the analysed manure conducted under high ammonia concentration turned out to be effective and stable for the HRT in the range of 25 and 35 days. When the process was carried out for the HRT below 25 days, a significant decrease in biogas production as well as organic matter decomposition was noticed. Moreover, the VFA/TA ratio increased above the critical value indicating instability of the process.

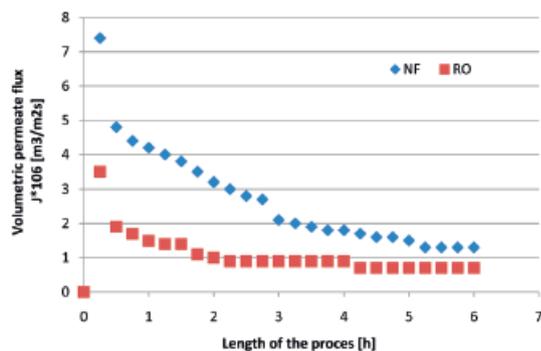


Figure 4.
Dependency of the volumetric flux on the length of NF and RO

2. The most appropriate value of HRT amounted to 30 days, which corresponded to the OLR at the level of 1.57 kg/(m³×d). Under those conditions, the highest biogas production (0.86 m³/(m³×d) as well as organic matter bioconversion (VS: 45%; COD: 57%) were achieved.
3. The methane fermentation affected the separation properties of the digested manure in a positive way. As compared to the raw manure (524s), the digested manure was characterized by a significantly lower values of CST (274-310s).
4. Application of the nanofiltration process (NF) for the treatment of the analysed manure turned out to be ineffective, as regards of all degrees of pollutant removal.
5. The treatment of the analysed leachate by means of reverse osmosis (RO) ensured a high degree of pollutant removal. The concentration of COD and total phosphorus decreased below the levels allowing its release to the natural reservoir. However, in spite of a relatively high degree of ammonia removal, i.e. 86%, its concentration in the permeate after the process exhibited too high value (794 mgNH₄⁺/dm³) which did not allow to release the treated permeate to the natural reservoir. The concentration of ammonia-nitrogen in the permeate strongly depends on its initial value before the membrane process. It shows that the application of purification methods geared towards the removal of ammonia-nitrogen, will play a key role. A controlled ammonium magnesium phosphate (struvite) precipitation seems to be an interesting solution, as it enables a concurrent binding of both nitrogen and phosphorus. Besides, manure pre-treatment will most likely allow to obtain a higher volumetric membrane flux and to lengthen the membrane life cycle.

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