

SELECTED METHODS FOR MANAGEMENT OF POST-FERMENTATION
SEDIMENT. THE USE OF EXTRUSION PROCESSING IN DIGESTED
SLUDGE MANAGEMENT (a review)

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Abstract. This review discusses the problem of management of digested sludge with the use of various methods for the management, including extrusion cooking. Extrusion cooking as a method of management of digestate can be an innovative approach to this topic. Until now there have been no studies on the use of the extrusion process to convert anaerobic digestion sludge. The extrusion process plays an important role in the transformation of materials on an industrial scale. An agricultural biogas plant can produce up to several tons of digested sludge per year (depending on the size of the installation). The most common method for utilisation of this kind of material is the use thereof as a fertiliser. However, this solution requires large areas of farmland. The best methods for conversion of digested sludge are those allowing the separation of the solid part from the liquid part. One of these methods consists in obtaining pellets in the extrusion process.

Keywords: agricultural biogas, digested sludge, extrusion

INTRODUCTION

The aim of the study was to indicate the extrusion process as a new method of post-fermentation sludge management. This paper reviews the methane fermentation process and various methods of post-fermentation sludge management.

Agricultural biogas installations, representing the renewable energy sector, serve for the production of biogas which is used primarily for energy purposes. Year by year, the number of these installations increases, which is associated with increasing amounts of digestate produced.

The major component of digested sludge is water (95-97%). The other 3-5% is constituted by incompletely digested bioactive material. This aspect is extremely

relevant, given the important processes taking place in soils. Organic compounds degraded in the methane fermentation process are much more easily available to soil microorganisms and plants. Digestate from an agricultural biogas installation utilising plant and animal production waste requires adequate processing methods. Methane fermentation residues do not differ considerably in terms of the content of mineral compounds from the chemical composition of raw materials used for biogas production. A significant difference can only be expected in the structure of organic compounds which are decomposed into simpler compounds in the fermentation process. In biogas plants that utilise plant raw material originating from agricultural production, the mineral composition of the fermentation residues is formed already at the stage of the yield quality of crops cultivated for the production of appropriate silage. Therefore, the usability of the so-called digestate is not questionable. The problems are related to the physical properties of the digestate.

METHANE FERMENTATION

Methane fermentation is a biochemical process occurring in anaerobic conditions. The entire process is composed of several stages which are carried out by specialised microbial groups comprising hydrolysing and fermentation bacteria (Hans and Cosima 1994, Zhang *et al.* 2006).

Microbiology of the process

Decomposition of organic matter in anaerobic conditions requires synchronised action of many microbial groups, each of which plays a special role (Veeken *et al.* 2000). Hydrolysing and fermentation bacteria are involved in the depolymerisation of high-molecular lignocellulosic compounds and in the hydrolysis of monomers contained in the raw material applied. The end products include mainly acetate and hydrogen, volatile fatty acids, and alcohols. Hydrogen-producing acetogenic bacteria convert fatty acids into acetate and hydrogen. In turn, two groups of methanogenic Archaeobacteria produce methane from acetate and hydrogen (Knol *et al.* 1978, Ince and Ince 2000).

The presence of methanogenic bacteria in the final phase of the fermentation process (e.g. *Methanosarcina*, *Methanobacterium*, *Methanomicrobium*, *Methanococcus*) results in the production of methane from acetic acid. At this stage, organic acids and other compounds produced in the first phase of fermentation are decomposed. This step limits the entire anaerobic process (Hill 1983, Znaheg *et al.* 2006, Amani *et al.* 2010). Depending on the temperature prevailing in the bioreactor, three types of fermentation can be distinguished: mesophilic in temperature range of 35-38°C, thermophilic in temperature range of 42-55°C, and psychro-

philic taking place at a temperature lower than 20°C (Angenent *et al.* 2002). The temperature in the mesophilic range definitely dominates in agricultural biogas plants (Hassan *et al.* 2013).

Inhibitors of the fermentation process

The process can be disturbed by several factors (Chen *et al.* 2014). The critical points include temperature, pH, formation of ammonia and hydrogen sulphide, and errors occurring while loading the substrate into the digester. Any failure in the fermentation process may lead to spoilage of the entire feedstock; therefore, monitoring of the process and appropriate choice of the substrates loaded into the digester are essential. The C/N ratio is of great importance, particularly when substrates containing substantial amounts of protein, lipids or carbohydrates are applied (Siles *et al.* 2010, Gutser *et al.* 2005). An increased content of nitrogen leads to the generation of ammonia (NH₄), which may limit bacterial growth.

Table 1. Summary of the results of the investigations of the impact of ammonia on the methane fermentation process (Chen *et al.* 2014).

| Substrate | Temperature (°C) | pH | Total ammonia (mg dm ⁻³) | Ammonium nitrogen (m dm ⁻³) | Publication |
|----------------------------------|------------------|---------|--|--|--------------------------|
| Industrial waste (glucose) | 35 | 6.8-7.0 | 2480 (50% inhibition of methane production) | – | Zhou and Qui 2006 |
| Industrial waste (yeast extract) | 35 | 7.7 | 1445 (50% inhibition of methane production) | 27 (50% inhibition of methane production) | He <i>et al.</i> 2011 |
| Pig slurry | 35 | 7.6 | > 5200 (100% inhibition of methane production) | 200 (100% inhibition of methane production) | Garcia and Angenent 2009 |
| Industrial waste (glucose) | 52 | 7.8 | 7000 (75% inhibition of methane production) | 620 (100% inhibition of methane production) | Siles <i>et al.</i> 2010 |
| Cattle slurry | 55 | 7.2 | 4000 (50% inhibition of Acetoclastic methanogens) 7500 (50% inhibition of Hydrogenotrophic methanogens) | 0% inhibition of Acetoclastic methanogens) 520 (50% inhibition of methanogens) | Borja <i>et al.</i> 1996 |
| Industrial waste (yeast extract) | 35 | 8.1 | – | 800 (78-96% COD removal efficiency) | Calli <i>et al.</i> 2005 |
| Municipal waste | 55 | 7.5 | 5600 (50% inhibition of methane production) | 635 (50% inhibition of methane production) | Bayr <i>et al.</i> 2012 |

Table 1 presents the results of investigations of the effect of ammonia on the methane fermentation process (Jin *et al.* 2012). For a majority of bacterial groups, ammonia is a source of N_2 , but at low concentrations (from 0.15 g dm^{-3}), it has an inhibitory effect on microorganisms. High total concentrations of NH_3 and NH_4 (above 3000 mg dm^{-3}) may result in inhibition of the biogas production process. Installations loaded with high-protein substrates and those working in thermophilic conditions are particularly exposed to this problem, since the inhibitory action of ammonia increases with the rising temperature (Wellinger *et al.* 1991).

Post-fermentation sludge

Proper sludge management is a very important aspect in the entire field of biogas production (Wong *et al.*, 2006). The main factors determining the use of digestate include its quality, local conditions, and applicable legal regulations.

The post-fermentation sludge produced by biogas plants has to be managed in such a way that the selected disposal mode is in agreement with the environmental conditions and legal provisions (Alburquerque *et al.* 2012). A biogas plant with a capacity of 1 MW utilising agricultural waste (manure, slurry) in a mixture with energy plant silage generates from 20 to 30 tons of digestate per year (Lansing *et al.* 2010, Goberna *et al.* 2011).

To manage such an amount, 2-3 thousand ha of arable land should be available for application of the digestate. The exact surface area depends on the total nitrogen content in the digestate, since the fertiliser dose is limited to 170 kg ha^{-1} . In many cases, biogas plants are planned to be constructed on farms focused on livestock production, which generate waste (manure or slurry) with high nitrogen contents.

QUALITY OF SLUDGE

The quality of post-fermentation sludge is associated with many factors, e.g. the content of dry organic matter, the quality of raw materials used for fermentation, and the presence of inhibitors (pesticides, growth hormones, heavy metals, antibiotics).

The fermentation rate and efficiency as well as the rate of proliferation of methane bacteria is dependent of the substrate quality, which has been confirmed in investigations based on spectroscopic techniques (Provenzano *et al.* 2011), and, to a large extent, on the quality the entire process. Stability of such parameters as temperature, pH, proper stirring of the mass, and inhibitory factors ensures long-term growth of normal bacterial flora. A sustainable process has a direct effect on the level of organic matter biodegradability, which in turn is reflected in the quality of the resultant digestate. Microorganisms present in bioreactors are often con-

tained in the digestate. These most frequently include *Pseudomonas*, *Klebsiella*, *Salmonella*, *Penicillium*, *Shigella*, *Bacteriodes*, *Aspergillus*, and *Bacillus* (Linderoth *et al.* 2008).

Changes in the quality of substrates after the methane fermentation process

Changes that can be noted during the transformations of substrates subjected to methane fermentation mainly involve a nearly 60% reduction of the organic matter content per dry weight, fragmentation of solid compounds, full or partial hygenisation, and degradation of compounds responsible for the oppressive smell. The degree of each transformation is primarily related to the ratio of organic substances contained in the material. Substrates rich in lignocellulosic compounds are the most difficult to degrade (El-Mashad and Zhang 2010, Möller *et al.* 2010). Table 2 shows an example of the composition of post-fermentation sludge.

Table 2. Chemical composition of post-fermentation sludge compared with cattle and pig slurry (according to Gutser 2005, El-Mashad *et al.* 2010, Möller 2010, Svoboda 2013a)

| Composition | Pig slurry | Cattle slurry | Post-fermentation sludge |
|-----------------------------|------------|---------------|--------------------------|
| Dry matter % | 8.80 | 9.80 | 1.5-40 |
| Dry organic matter % | 81.00 | 84.00 | 38-75 |
| Nitrogen g kg d.m. | 63.00 | 40.00 | 3.5-130 |
| Ammonium nitrogen g kg d.m. | 41.00 | 20.00 | 35-95 |
| Phosphorus g kg d.m. | 23.00 | 7.60 | 0.2-12.2 |
| Potassium g kg d.m. | 37.00 | 40.00 | 0.12-36 |
| Cd mg kg d.m. | 0.17 | 0.13 | 0.30 |
| Cr mg kg d.m. | 4.10 | 2.30 | 9.30 |
| Cu mg kg d.m. | 178.00 | 49.00 | 113.00 |
| Pb mg kg d.m. | 0.95 | 0.92 | 4.10 |
| Ni mg kg d.m. | 3.20 | 3.60 | 9.70 |
| Zn mg kg d.m. | 635.00 | 190.00 | 375.00 |

MANAGEMENT OF SLUDGE AS A FERTILISER

Unprocessed digestate is a liquid and it is often characterised by a disagreeable smell. It can be used directly as a fertiliser in periods in which fertilisation is made possible by weather conditions, soil conditions, presence of plants in the fields, and vegetation condition. Digestate has to be stored in periods that exclude application thereof on fields. In the liquid form, it must be stored in large-volume containers. The use of unprocessed digestate in fertilisation may provoke conflicts associated with the odour nuisance in rural areas. Additionally, unprocessed digestate with a high hydration level can only be used in close proximity to a biogas plant, since transportation thereof is cost inefficient.

The main factors determining the application of digestate include its quality, local conditions, and applicable legal regulations. In Poland, legal provisions, which currently do not favour easy digestate management, determine the use of the by-product of biogas plants. However, together with an increase in the number of biogas installations, there will be a possibility of regulation of the laws governing digestate disposal.

Legal aspects

Material derived from the methane fermentation process is called digestate or post-fermentation sludge. National laws strictly regulate the production and disposal of this waste. Currently, the Act of 14 December 2012 on wastes (Journal of Laws 2013, item 21) and the Act of 10 July 2007 on fertilisers and fertilisation (Journal of Laws 2007 No. 147, item 1033 as amended) are in force. Two other Regulations should also be mentioned here. These include the Regulation of the Minister of the Environment on the Catalogue of Waste, of 27 September 2001 (Journal of Laws 2001 No. 112, item. 1206), and the Regulation of the Minister of the Environment on the R10 recovery process, of 5 April 2011 (Journal of Laws of 2011 No. 86, item. 476).

Post-fermentation mass is qualified by the Polish legislation in the catalogue of waste, and its use in agriculture is regulated by the Regulation on R10 recovery. As specified in this Regulation, “substances generated in the process of anaerobic decomposition of manure, liquid manure, slurry, and plant waste from agriculture and agri-food industry” can be used for soil fertilisation or amendment. However, the Regulation outlines strict conditions that have to be fulfilled prior to the application of post-fermentation sludge for fertilisation.

Principles for natural fertilisers specified in the Act on fertilisers and fertilisation shall be employed. Animal-origin materials shall comply with the requirements specified in the European Parliament and Council (EC) Regulation No. 1069/2009. Wastes shall be applied evenly over the soil surface.

Wastes shall be spread over land at a maximum depth of 30 cm. The regulation substantially relaxes the requirements for digestate used as a fertiliser. An important amendment is the waiving of the requirements for post-fermentation sludge that were similar to those for municipal sewage sludge. Furthermore, it is not mandatory to assess the heavy metal content prior to the application of digestate and to fragmentise digestate derived from anaerobic decomposition of plant and animal wastes (code: 19 06 06).

Microbiological quality

Since the bioreactor feedstock can only comprise organic matter (material for proliferation of methanogenic bacteria), post-fermentation sludge is rich in easily available nutrients. During the anaerobic decomposition of organic compounds, long-chain proteins, lipids, and carbohydrates undergo a process of mineralisation (Masse and Droste 2000). Nitrogen and phosphorus are mineralised into NH_4^+ and PO_4^{3-} , and they are more easily available for plants in this form. Digestate accounts for 90-95% of the initial volume of the bioreactor feedstock. The exact composition of the digestate in a biogas plant is closely related to the bioreactor feedstock (Gujer and Zehnder 1983). In bioreactors, reduction of pathogens, weed seeds, fungal spores, and other undesirable matter takes place. Hence, post-fermentation sludge is a considerably more attractive fertilising material than agricultural wastes such as liquid manure, slurry, or manure, which contain great amounts of both pathogens and weed seeds (Martinez *et al.* 2012).

Microorganisms present in the bioreactor digestate serve important functions, e.g. *Klebsiella* and *Clostridium* spp. fix nitrogen, and *Bacillus* and *Pseudomonas* spp. produce phosphorus-releasing enzymes (Alfa *et al.* 2014).

SLUDGE DRYING AND INCINERATION

Another form of digestate management is dehydration, drying, and pelleting followed by incineration. The most popular methods of thermal treatment of sludge include incineration, pyrolysis, and gasification (Manya 2012, Lech-Brzyk 2014).

Pyrolysis and gasification are endothermic processes taking place under anaerobic conditions. Pyrolysis starts at a temperature of 200°C, but the upper value is unlimited, reaching even plasma temperature. The end product of biomass combustion is charcoal and pyrolytic gases. Gas obtained from organic waste can have a calorific value of approx. 12 MJ kg⁻¹ (Guiot *et al.* 2011, Andreoni *et al.* 1990, Basu 2013, Mohan *et al.* 2006).

Digestate is a difficult material for thermal treatment. During drying, it shrinks intensively and cracks on the surface or forms a hard layer impeding drying. As in the case of the application of digestate as a fertiliser, the composition of ash depends on the composition of the substrate used in the fermentation process. As indicated by Kratzeisen *et al.* (2010), pellets from post-fermentation sludge contain high concentrations of nitrogen, sulphur and chlorine, that exceed several-fold the allowable values. The zinc content slightly exceeds the limit value, whereas the levels of arsenic, mercury or cadmium are close to the limits. The high content of sulphur and nitrogen obstructs combustion. Emission of dust, CO and NO_x is higher than that from the conventional pellet. Table 3 shows an ele-

mental analysis of pellets obtained from post-fermentation sludge and wood pellets. The greatest differences can be noted in the calcium content. In the wood pellets it reached 13.60%, while the calcium content in the post-fermentation sludge ash was nearly 42%. The content of phosphorus was evidently the highest among the analysed elements. It was the highest, i.e. 26.7%, in ash derived from incinerated post-fermentation sludge, compared with that from wood (0.6%).

Table 3. Comparison of the ash composition of pellets obtained from post-fermentation sludge and wood pellets (Kratzeisen *et al.* 2010).

| Element | Ash from incinerated post-fermentation sludge | Ash from incinerated wood pellet | Limit value |
|------------------------|---|----------------------------------|-------------|
| P % | 26.70 | 0.60 | - |
| K % | 15.50 | 6.40 | - |
| Mg % | 8.40 | 6.00 | - |
| Na % | 0.80 | 0.70 | - |
| Ca % | 13.60 | 41.70 | - |
| Si % | 30.40 | 25.00 | - |
| S % | 0.90 | 1.90 | - |
| Fe % | 1.80 | 2.30 | - |
| Al % | 1.20 | 4.60 | - |
| As mg kg ⁻¹ | 1.10 | 4.10 | 40.00 |
| B mg kg ⁻¹ | 2.30 | 13.60 | 150.00 |
| Cd mg kg ⁻¹ | < 0.5 | 1.20 | 1.50 |
| Cr mg kg ⁻¹ | 184.00 | 325.50 | 2.00 |
| Ni mg kg ⁻¹ | 285.00 | 66.00 | 80.00 |
| Hg mg kg ⁻¹ | < 0.1 | 0.01 | 1.00 |

SLUDGE EXTRUSION

Introduction

Since the time it was first used for extrusion of food products in the 20s of the 20th century, the process has become a modern approach facilitating the production of new foods from traditional raw materials. Extruded products are mainly characterised by high durability and microbiological purity. Moreover, they have a porous structure and high water capacity, which ensures a relatively high degree of digestibility (Camire 1998). The extrusion process involves pressing a material at high pressure (up to 20 MPa) and high temperature (up to 200°C). During the process, the material undergoes considerable physicochemical changes, hygienisation, and structural changes (Rosentrater *et al.* 2005). The process is run in extruders. A screw mounted in a cylinder is the main operating element in extruders. Physical and chemical changes occurring in the loaded material are associated with the extrusion parameters as well as with the structure of the device itself.

Several types of extruders can be distinguished. One of the classifications is based on the structure of the plastifying units, i.e. single- or twin-screw (Mościcki and Mitrus 2007, Malyon and Malik 2007, Amornthewaphat and Attamangkune 2008). In comparison with devices equipped with two screws, single-screw extruders are simple machines. Processed material is transported, compacted, and forced under high pressure through a die orifice located at the end of the cylinder. Materials with a high coefficient of friction are easy to process in these extruders. The disadvantage of single-screw extruders is the poor mixing of raw materials and their limited effectiveness (Mościcki and Mitrus 2007, Bruin *et al.* 1978).

Twin-screw extruders are divided into co-rotating and counter-rotating devices. In co-rotating extruders, screws rotate in the same direction, whereas the screws in counter-rotating ones rotate in opposite, clockwise or counter-clockwise, directions (Colbert 1990). Counter-rotating extruders are most commonly used due to their versatility. The high screw rotation velocity (up to 300 r min^{-1}) results in good efficiency of material flow and uniform extrusion. The rotating screws and their overlapping threads push the material forwards. This arrangement of the screws prevents the material from being deposited in the space between the screws and the surface of the cylinder.

To ensure that the extruded material has an appropriate and desirable structure, the process has to take place in conditions that are strictly associated with the structure of the extruder and its parameters. First of all, an adequate time of material residence in the extruder has to be specified (Mościcki and Mitrus 2007, Secker *et al.* 2003). Temperature, which influences the condition of the extruded mass and inactivates microorganisms that could adversely affect its microbiological quality, is also important. Other factors that have great significance for the extrusion process include pressure, shear stress, and process variables (type of extruder, screw geometry, moisture content of the raw material, temperature profile of the cylinder, screw rotation velocity, material loading rate). The appearance, structure, and texture of extruded products are influenced by physicochemical changes in sugars, cellulose, and proteins. These changes are caused by combined thermal energy supplied and generated by the extruder with adequate shear strength (Chen *et al.* 1991, Janssen *et al.* 2002).

The three basic nutrients (fat, protein and fibre) undergo different changes during the extrusion process (Alonso *et al.*, 2000). It has been assumed for a long time that the lipid content in the components impedes extrusion (IloandBerghofer, 1999). This view, however, has changed after long-term research. It has been proved that lipid compounds contribute to positive modification of the properties of extrudates (Zeitoun *et al.* 2010).

Biomass extrusion

The use of extrusion in processing raw material for methane fermentation has been a common method used in investigations for many years (Karunanithy and Muthukumarappan 2012, Novarino and Zanetti 2012, Chen *et al.* 2014, Zheng *et al.* 2014, Williams *et al.* 1997, Cesaro and Belgiorno 2014, Hjorth *et al.* 2011). In their research, Hjorth *et al.* (2011) extruded five types of agricultural biomass. Methane production was measured over a period of 90 days. Fragmented biomass processed in the extruder became more digestible for bacteria and enzymes during the fermentation process. Lamsal *et al.* (2010) compared two pre-treatment methods, i.e. milling and extrusion. The researchers were the first to show extrusion-induced major changes in reducing sugars. Both methods were regarded as alternatives to pre-treatment of lignocellulose-rich biomass, and the extrusion process was shown to result in higher yields of reducing sugars in the analysed material. The best results were obtained with parameters 3.7 Hz/150°C and 3.7 Hz/110°C. This material processing method contributes to easier microbial degradation in bioreactors during the fermentation process.

Post-fermentation sludge extrusion

Research on extrusion of post-fermentation sludge seems to be an entirely novel approach to the processing of this type of waste. The versatility of the extruder allows the selection of parameters (pressure, temperature, screw rotations) that will ensure high quality of extrudates. The possibility of supplementation of the extruded material with additives, e.g. minerals, fertiliser enhancers, dyes etc., can yield a product that will meet the requirements of a wide range of customers. High temperature and pressure help neutralise any pathogens and undesirable microflora in an effective manner. Since such organic compounds as protein, sugars and lipids undergo considerable changes during the extrusion process, post-fermentation sludge treated with this method will have a structure that allows easier degradation thereof.

Another positive side of the use of extrusion in the processing of post-fermentation materials is the easier storage of pellets. The pellet form ensures successful storage in big-bags, sacks, and easily stackable packages (e.g. containers). Obviously, the conditions of storage of such fertilising material should meet the standards for storage of organic-mineral fertilising materials.

SUMMARY

Besides biogas production, management of post-fermentation sludge is another important aspect requiring continuous research and improvement of methods used currently. Reduction of the surface area of the post-fermentation sludge by separation of the effluent allows further processing of solid organic matter. The introduction of extrusion as a new method for processing solid organic matter remaining after the separation may contribute to the production of a fertilising material with a completely new form. The presented issue requires a number of detailed studies that will help to evaluate these parameters of the derived pellets that will define the usefulness of the material in the best way. Yet, pot and field experiments assessing the application value of the pellets will be the most important indicator.

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WYBRANE METODY ZAGOSPODAROWANIA OSADU POFERMENTACYJNEGO.
WYKORZYSTANIE EKSTRUZJI DO PRZETWARZANIA POFERMENTU
(artykuł przeglądowy)

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Streszczenie. Artykuł omawia temat zagospodarowania osadu pofermentacyjnego za pośrednictwem różnych metod, w tym ekstruzji. Wykorzystanie ekstruzji jako metody zagospodarowania osadu może być innowacyjnym podejściem do tego tematu. Nie odnotowano do tej pory żadnych badań na temat stosowania procesu ekstruzji do przekształcania osadów fermentacji beztlenowej. Ekstruzja spełnia ważną rolę w przemyśle przekształcania materiałów. Jedna biogazownia rocznie może produkować (w zależności od wielkości instalacji) blisko kilkadziesiąt ton osadu rocznie. Najczęstszym sposobem zagospodarowania osadu jest wykorzystanie go jako materiału nawozowego. Takie rozwiązanie wymaga jednak znacznej powierzchni pól uprawnych. Najlepszymi metodami do zagospodarowania osadu są te, które pozwalają na odseparowanie części stałej od

płynnej. Jedną z takich metod polega na otrzymaniu peletu w procesie ekstruzji. Tak zagospodarowany osad, po wzbogaceniu go w mikro- i makroskładniki, może być łatwo przechowywany i z powodzeniem stosowany jako pełnowartościowy nawóz organiczno-mineralny.

Słowa kluczowe: biogazownia rolnicza, osad pofermentacyjny, ekstruzja