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# FERTILIZER VALUE AND ENERGY VALUE OF MULTI-COMPONENT DIGESTATE PULP AND THE POSSIBILITY OF ITS AGGREGATION

*Summary*

*The aim of the research was to indicate the possibility of using digestate which was obtained in the process of biogas production and the analysis of the effects of the aggregation process using rolling. The characteristics of fertilizers were determined based on the analysis of the basic properties of post-fermentation pulp such as: dry matter, organic matter, pH, C: N ratio, content of macroelements. The analyzes were carried out for a mixture of excess substrates and elements of food and feed processing. A narrow C: N ratio and a near-neutral pH were indicated. The content of total nitrogen (1.8%) allows the soil application of digestate as a soil improver. The ratio N: P: K (1: 0.53: 0.61) indicates potassium deficiency. The combustion heat was determined with the result of 17.20 MJ·kg-1. The obtained values of humidity and combustion heat were used to determine the net calorific value of digestate equal to 15.80 MJ·kg$^{-1}$. The spherical granulate was obtained using the coating method. For the moisture content of the digestate 30% m / m granules were obtained in the size range from 1.5 to 4.5 mm.*
***Key words**: fermentation pulp, aggregation, fertilizer value, energy value*

# WARTOŚĆ NAWOZOWA I WARTOŚĆ ENERGETYCZNA WIELOSKŁADNIKOWEJ PULPY POFERMENTACYJNEJ ORAZ MOŻLIWOŚCI JEJ AGREGACJI

*Streszczenie*

*Celem badań było wskazanie możliwości zagospodarowania pofermentu pozyskanego w procesie wytwarzania biogazu oraz analiza efektów procesu agregowania obtaczaniem. W ramach realizacji celu pracy wskazano szereg zadań szczegółowych. Analizy przeprowadzono dla mieszanki substratów nadmiarowych i elementów procesów przetwórczych żywności i pasz. Określono cechy nawozowych na podstawie analizy podstawowych własności pulpy pofermentacyjnej takich jak: materia sucha, materia organiczna, pH, stosunek C:N, zawartość makroskładników pokarmowych. Wyznaczono wąski stosunek C:N oraz odczyn zbliżony do obojętnego. Zawartość azotu całkowitego (1,8%) pozwala na doglebowe zastosowanie pofermentu jako ulepszacz glebowy. Stosunek N:P:K (1:0,53:0,61) wskazuje niedobór potasu. Ciepło spalania wyznaczono uzyskując wynik 17,20 MJ·kg$^{-1}$. Za pomocą uzyskanych wartości wilgotności oraz ciepła spalania określono wartość opałową pofermentu wynoszącą 15,80 MJ·kg$^{-1}$. Przy zastosowaniu metody obtaczania uzyskano granulat sferyczny. Dla poziomu wilgotności pofermentu 30% m/m uzyskano granule w zakresie wielkości od 1,5 do 4,5 mm.*
***Słowa kluczowe**: pulpa pofermentacyjna, agregacja, wartość nawozowa, wartość energetyczna*

## 1. Introduction

The development of technologies of organic substrate management has contributed to the popularization of aerobic and anaerobic stabilization processes in the technical scale.

The processes of aerobic and anaerobic biomass stabilization, through the action of bacteria and symbiotic organisms, contribute to the mineralization of organic matter [Parr and Hornick 1993].

Differences between the two processes of biomass stabilization result from the characteristics of biochemical transformation products that result from the digestive properties of the microflora.

A special feature of anaerobic processes is the orientation of biomass processing in order to produce energy products in gas or liquid form [Encyklopedia techniki 1965].

A special feature of the biomass oxygen stabilization process is intensive biomass mineralization with the creation of biological heat as a result of the digestion of organic matter by aerobic bacteria and symbiotic organisms.
In both processes, microorganisms and symbiotic organisms primarily use proteins, fats, carbohydrates and food fiber [Cerda et al., 2018].

Digestate (fermentation pulp) is a residue after the anaerobic decomposition of organic matter.

The basic factor determining the chemical composition of the digestate includes the type of substrates used in the biogas plant. For this reason, each biogas plant produces a diversified mass. On the basis of the analysis of reactions occurring during methane fermentation, however, it is possible to generally determine the physicochemical properties of the digestate, and what is related to it - its fertilizing value.

During methane fermentation organic matter decomposes. This leads to a narrowing of the ratio (carbon-nitrogen) of C: N in the post-fermentation mass.

The fermentation pulp is composed of the fermented material, (mineral and organic parts) and dead bacteria.

This substance contains nutrients that include macro and microelements that exist in mineral forms that are directly available to plants.

From the point of view of fertilizer values of various waste materials, the following are relevant: carbon content (C), nitrogen content (N), phosphorus content (P), potassium content (K), C: N ratio, pH and micronutrient content (including heavy metals).

All listed properties determine, among other things, the susceptibility of organic materials to mineralization.

As a result of the fermentation process, the C: N ratio of digestate is usually in the range of 20: 1 to 30: 1. This increases the susceptibility to mineralization of organic matter remaining unused previously by anaerobic bacteria. Aminification which processes nitrogen during fermentation leads to the formation of an ammonium nitrogen ($N-NH_4$).

The fermentation pulp, which comes from wet fermentation technology, is a liquid mixture of substrates used in biogas production.

The production of biogas in agricultural and utilization biogas plants requires sustainable technological activities. Technological activities are related to the raw material management and utilization of digestate [KTBL-Heft 84 2009].

In connection with the environmental requirements of the fertilizer management, certain limitations should be adopted in the design solutions of agricultural biogas plants. In winter, the digestate must be collected without the possibility of soil application.

It should be noted that biogas plant constructions are designed for the current possibilities of the process substrate market. Any intervention in the technological conditions of material selection brings environmental and technical consequences.

The environmental consequences include the possible abandonment of post-fermentation soil application as a result of non-compliance with the formal requirements for use. Technical consequences include the case of overfilling of the fermentation storage before the beginning of a planned soil application.

The origin of the topic was indicated by the substrate itself, definitely universal, which unites fertilizer and energy attributes. The low-pressure granulation, proposed in the work, supports a multidirectional model of digestate management.

## 2. The aim of study

The aim of the research was to indicate the possibility of using digestate which was obtained in the process of biogas production and the analysis of the effects of the aggregation process using rolling.

To achieve the aim of the work, a number of specific tasks were indicated.

The characteristics of fertilizers were determined based on the analysis of the basic properties of post-fermentation pulp such as: dry matter, organic matter, pH, C: N ratio, content of macroelements [Szlachta and Fugol 2010].

The energy properties of post-fermentation pulp were determined based on the analysis of the heat of combustion and on the basis of the calorific value.

The effect of aggregation of the dried digestate was obtained using the low-pressure granulation method.

## 3. Methodology

Based on our own research and literature analysis [Jędrczak 2007, Myczko et al., 2011], these factors have been identified that characterize the fermentation pulp [Jones Jr. J.B. 2001, Karczewska, Kabała 2005, Fortuna T. (red.) 2003, DIN 38414 S 8. 2012].

Measurement of substance mass. The mass measurement was carried out by weighing the material on an electronic balance with a measurement resolution of 0.001 g and a measuring range of 0 to 200 g and an electronic weighing machine with a measuring resolution of 0.01 g and a measuring range of 0 to 6 kg. In this way, the composition of each blend and the percentage share of each ingredient were determined.

PH measurement. In the pH tests the potentiometric method was used, consistent with PN-EN 12176: 2004. For the preparation of samples, up to two containers of 250 $cm^3$ each, 15 g of material are taken. Samples are weighed on the precision balance (reading resolution up to 0.01 g).

Next, the material placed in the beakers is flooded with 200 cm3 of demineralised water and allowed to stand for 0.5 h. The samples are mixed every 10 minutes with a glass rod. During this time, the measuring equipment should be prepared (Laboratory pH meter with pH range from 1 to 14, Elmetron CP-215, measuring accuracy 0.1 and with temperature compensation function). Before pH measurement, pH-meter calibration should be performed according to buffer solutions at pH 4, 7 and 10 at 20ºC.

In the dry matter content tests, a drying-weight method was applied in accordance with the PN EN 12880: 2004 standard.. The measurement of dry matter content was carried out using the dryer-weight method. The substrate sample was dried at a temperature of 105ºC to a constant mass and weighed.

Determination of the content of organic matter. To determine the content of organic matter, the method of thermal mineralization in a muffle furnace was used in accordance with PN-75 / C-04616/01 and PN-EN 12879: 2004.

Thermal mineralization involves the destruction of the organic structure by incineration of the sample at high temperature (usually 500ºC to 600ºC).

Under these thermal conditions, the organic substance is completely degraded. The organic matter is charred and oxidized with atmospheric oxygen to $CO_2$ and $H_2O$.

The parameters describing the energy properties include: humidity, heat of combustion and calorific value.

The determination of total nitrogen was made using the elemental analysis method on the VarioMax apparatus. The determination of ammonium nitrogen was performed using the Kjeldahl method.

To determine the total phosphorus, the mineralized sample of the analyte was extracted in the presence of concentrated acid. Total phosphorus in homogenized filtrate was determined by colorimetry on a UV-VIS Cary 60 spectrophotometer.

To determine the total potassium, the mineralized sample of the analyte was extracted in the presence of concentrated acid. The total potassium in the homogenized filtrate was determined on a Varian 220 FS flame spectrophotometer.

The total content of trace elements - Cu and Mg was determined in the aqua regia extract according to ISO 11466 1995 (E). "Solid quality - extraction of trace elements soluble in aqua regia" Detection by atomic spectrophotometer (ASA) was performed on the Varian 220 FS.

The determination of these parameters was made at the Wood Chemical Technology Institute at the University of Life Sciences in Poznań. The moisture content was determined using a weight dryer method in compliance with PN-EN ISO 18134-3 standard. This method is based on measuring the loss in mass caused by evaporation of water during drying at 105°C-110°C.

The humidity test was carried out in three repeat tests each time. The mass of wet and dry samples was determined with an accuracy of 0.01 g.

Due to the high water content, the fermentation pulp was dried to an air-dry dry state, enabling determination of combustion heat and calorific value. The fermentation pulp was subjected to a milling process for examination and then sieving to obtain a representative sample with a 0.2 mm fraction. The dust obtained was used to make a 1 g tablet of the combustion heat used for the test. The examination of combustion heat was carried out at the Institute of Chemical Wood Technology with the use of computer calorimeter KL-12Mn2 based on PN-81/G-04513 and PN-ISO 1928 standards (Fig. 1). The test consisted in the total combustion of the sample in an oxygen atmosphere, under pressure in a cal**o**rimetric bomb immersed in a water jacket, with simultaneous measurement of the increase in water temperature. To determine the calorific value, the moisture content was determined with the use of the drying and drying-weight method in accordance with the PN-EN ISO 18134-3 standard.

**4. Results**

The post fermentation pulp was obtained from a biogas plant with an electrical power of 1.2 MWe which is owned by Biowatt S.A.

For the research we used the fermented pulp, produced during the anaerobic digestion of a mixture of substrates such as cattle slurry, waste from food production, waste feed, and redundant and defective substrates from the processing of fruit and vegetables (Fig. 2).

The composition of the mixture of fermenting substrates is presented in Table 1.

Table 1. Composition of a fermenting substrate mix
*Tab. 1. Skład mieszanki fermentujących substratów*

| | Component of the mixture | Dry matter | Percentage share |
|---|---|---|---|
| No | - | % | % |
| 1. | Cattle slurry | 3.26 | 55 |
| 2. | Meat pulp | 10.89 | 8 |
| 3. | Re-food homogen | 8.71 | 8 |
| 4. | Potatoes and potato peelings | 20.31 | 3 |
| 5. | Vegetable waste | 9.93 | 3 |
| 6. | Post flotation residue | 6.65 | 6 |
| 7. | Fatty sediment | 4.85 | 11 |
| 8. | distillery | 17.48 | 3 |
| 9. | Bakery waste | 75.47 | 1 |
| 10. | Coffe pomace | 33.64 | 2 |
| | Total of shares | | 100 |
| | Dry matter | 7.20 | |
| | Humidity | 92.80 | |

*Source: own study / Źródło: opracowanie własne*

The digestate (post fermentation pulp) taken from the biogas plant was dried at 50°C and obtained a loose and powdery form. The concentrated digestate by low-pressure granulation obtained the form of spherical granules. The parameters of dry matter and dry organic matter were compared for non-aggregated and aggregated material. The results of the tests are presented in the Table 2. The increase of the organic matter content in aggregate samples was obtained thanks to the doping of the starch binder.

Table 2. The content of dry matter and dry organic matter in conditioned post fermentation pulp
*Tab. 2. Zawartość materii suchej i suchej materii organicznej w kondycjonowanym pofermencie*

| | Form of digestate | Dry matter | Dry organic matter |
|---|---|---|---|
| No | - | % m/m | % m/m |
| 1. | Granulated | 92.6 | 71.2 |
| 2. | Dried | 89.3 | 64.1 |

*Source: own study / Źródło: opracowanie własne*

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Fig. 1. Computer Calorimeter KL-12Mn2 (on the left), general view of the test stand (on the right)
*Rys. 1. Kalorymetr KL-12Mn2 (po lewej), ogólny widok stanowiska badawczego (po prawej)*


*Source: own study / Źródło: opracowanie własne*

Fig. 2. View of feed mix substrates (from the left - coffee pomace, pulp meat, potato peels)
*Rys. 2. Widok substratów pokarmowych (od lewej – wytłoki kawy, pulpa mięsna, łupiny ziemniaka)*

The dried digestate was analyzed for the content of selected macronutrients and micronutrients [Mercik et al. 2003]. The average values of the analyzed contents of biomass components are presented in Table 3.

Table 3. The content of macroelements and micronutrients in the post fermentation pulp
*Tab. 3. Zawartość makroskładników i mikroelementów w pulpie pofermentacyjnej*

| | Nutrients | Content |
|---|---|---|
| No. | - | $kg \cdot Mg^{-1}$ |
| 1. | Total nitrogen | 17.90 |
| 2. | Ammonium nitrogen | 0.82 |
| 3. | Total phosphorus | 9.57 |
| 4. | Total potassium | 11.00 |
| 5. | Magnesium | 2.80 |
| 6. | Calcium | 36.00 |
| | Parameters | [-] |
| 7. | C:N ratio | 9.6 |
| 8. | pH | 7.14 |

*Source: own study / Źródło: opracowanie własne*

The next step was to analyze the dried digestate sample in terms of energy parameters.

Samples were taken in the form of tablets with a weight of about 1 g (Fig. 3).

For this purpose, the previously obtained fragmented digestate samples were used.

The obtained tablets were placed each time in a crucible being part of the calorimetric bomb.

The sample ignition was induced by a resistance wire touching the samples in at least two places.

Moisture of the tableted samples after preparation was 4.32%.

The combustion heat was determined with the result of 17.20 $MJ \cdot kg^{-1}$.

The obtained values of humidity and combustion heat were used to determine the net calorific value of digestate equal to 15.80 $MJ \cdot kg^{-1}$.

At the same time, a significant ash residue was observed during the study (Fig. 4).

Due to the lack of this part of the sample in the oxidation reaction, it decreased the calorific value and combustion heat of the post-fermentation pulp.

Due to the limited participation of part of the sample mass (ash residue) in the oxidation reaction, it reduced the calorific value and the heat of the thermal mineralization of digestate.

The obtained results of the content of macronutrients and microelements prove the suitability of the digestate as soil improvers. Valuable are: the content of organic matter, the content of lime and the possibility of assimilation and maintenance of moisture.

Possibility of compacting post fermentation pulp.

Fermented pulp may be aggregated as a result of: high-pressure aggregation, intermediate pressure aggregation, low-pressure aggregation [Kłassien and Griszajew 1989].

The assumption was to create repetitive, spherical granules with a diameter of 3 to 6 mm.

In research, the method of low-pressure granulation by coverting was used.

Preliminary tests of the granulation process were carried out as part of the research and development project "Development of production technology for high-quality organic fertilizer and plant growth accelerator from the digestate of biogas plant".

In the process of forming the granules a granulator with a diameter of 80 cm and a speed range from 0 to 220 rpm was used.

A laboratory grinder with ceramic mills and a sieve separating from 0.25 to 0.5 mm was used to grind the digestate.

The dripper formed drops with a diameter of 3mm.

The mixture of water and starch binder had a moisture content of 88,5 % m / m and maintained the possibility of pumping and drop formation. The droplets gravitationally dropped onto the rotating surface (Fig. 5).

The process of forming the granules was carried out by combining a wet drops of binder with a fragmented digestate onto a rotating surface.

The rotation of the wet fraction of digestate caused by the influence of the stationary side surface formed granules.

The tests were carried out for the nominal rotation speed of 125 rpm.

For this rotational speed, the moment of initiating the granulation process was experimentally determined.

The granules varied in size and shape depending on the degree of moisture content of the post fermentation pulp (Fig. 6).

Lowering the moisture content of the digestate (post fermentatio pulp) from 40 to 30% allowed observation of the research results.

Lowering the moisture content of the post-fermentation pulp to 30% allowed for the formation of granules with dimensions from 1.0 to 4.5 mm.

Further reduction in the moisture content of the mixture prevented the aggregation processes and the formation of granules.

*Source: own study / Źródło: opracowanie własne*

Fig. 3. Sample of digestate in a ceramic crucible weighing 1 g±10% (left), ash after combustion of three samples (right)

*Rys. 3. Próbka osadu pofermentacyjnego w tyglu ceramicznym o masie 1g±10% (po lewej), popiół po spaleniu trzech próbek (po prawej)*

*Source: own study / Źródło: opracowanie własne*

Fig. 4. Dried digestate shredded with a ceramic mill

*Rys. 4. Suszony poferment rozdrobniony młynem ceramicznym*

*Source: own study / Źródło: opracowanie własne*

Fig. 5. The process of forming granules by the method of turning

*Rys. 5. Proces formowania granul metodą obtaczania*

*Source: own study / Źródło: opracowanie własne*

Fig. 6. Changes in the shape and size of granules depending on the humidity of the mixture (from the left humidity: 30% m / m, 33.5% m / m, 36.5% m / m, 40% m / m)
*Rys. 6. Zmiana kształtu i wielkości granul w zależności od wilgotności mieszanki (od lewej wilgotność 30; 33,5; 36,5; 40% m/m)*

Table 4. Dependence of the size of granules on the moisture of the aggregate mixture
*Tab. 4. Zależność wymiaru granul od wilgotności mieszanki agregowanej*

| No | Sieve size | The type of the sample according to the humidity of the mixture | | | | |
|---|---|---|---|---|---|---|
| | | <30 [% m/m] | 30 [% m/m] | 33.5 [% m/m] | 36.5 [% m/m] | 40 [% m/m] |
| 1. | < 1[mm] | 23.8± 0.03 | 4.7±0.05 | 3.2±0.03 | 2.6±0.02 | 0.7±0.01 |
| 2. | 1÷2 [mm] | 29.7±0.45 | 7.1±0.40 | 5.3±0.80 | 5.2±1.00 | 3.2±0.12 |
| 3. | 2÷3 [mm] | 29.6±0.01 | 10.7±0.02 | 8.6±0.07 | 6.1±0.10 | 4.1±0.09 |
| 4. | 3÷4 [mm] | 11.4±0.08 | 15.9±0.20 | 10.2±0.25 | 9.4±0.45 | 4.7±0.40 |
| 5. | 4÷5 [mm] | 3.1±5.60 | 28.8±8.80 | 22.7±9.10 | 18.6±8.20 | 5.8±7.70 |
| 6. | 5÷7 [mm] | 2.4±0.10 | 29.6±0.50 | 29.5±2.80 | 21.3±5.10 | 15.4±5.10 |
| 7. | 7÷8 [mm] | 0 | 3.2±1.00 | 18.6±0.80 | 27.7±0.25 | 34.6±2.20 |
| 8. | 8÷10 [mm] | 0 | | 1.9±0.50 | 6.7±0.10 | 23.8±1.00 |
| 9. | >10 [mm] | 0 | | | 2.4±0.30 | 7.7±0.50 |

*Source: own study / Źródło: opracowanie własne*

Sieve analysis was performed on samples of the granulate with a mass of 250 grams (Tab. 4).

For the aggregate mix moisture content below 30% there was no aggregation process in the structures above the average droplet size of the binder.

Only 5.5% of the weight of the granules in the sample with a moisture content below 30% formed a dimension greater than 4 mm.

Increasing the moisture content of the digestate to the level of 40% resulted in the connection of binder drops and digestate into structures larger than the average droplet size of the binder.

## 5. Summary

Knowing the benefits of different applications can help you make a decision.

The choice of a particular solution may be determined by economic or technical criteria [Fuldauer et al., 2018].

The conducted studies indicate the possibility of using the fermentation according to energy or fertilizer demand.

Post fermentation pulp tests showed that the content of nutrients and the level of organic matter allows for management as a fertilizing and structure-forming factor.

The calorific value (15.80 MJ $kg^{-1}$) obtained during the combustion of dried digestate indicates that it is a material similar to wood pellets (from 16 to 20 MJ $kg^{-1}$).

The problem with the use as fertilizer and energy consists in the low level of dry matter content. This makes it especially difficult to use energy [Törnwall et al. 2017].

The tested aggregation solution indicated the possibility of creating a compact spherical structure even at a moisture content of up to 40% m / m.

Dried and shredded post-fermented pulp can be mixed with a binder to obtain a granulated product of a controlled size [Törnwall et al. 2017].

The possibility of obtaining granules, with a spherical shape, is influenced by the moisture of the digestate pulp in contact with the binder.

It should be pointed out that preforming of spherical granules with a higher than 40% moisture of digestate means limiting the share of liquid binder.

The element that substitutes for the external binder should be spontaneous adhesion properties in the dried digestate.

Adhesive properties of organic matter may result from the content of humic acids [Kwiatkowska-Malina 2015].

The pre-treatment of digestate in an aerobic stabilization process can result in adhesive properties.

Compost binds moisture and retains adhesive properties even with humidity above 50% [Maciejewska 1993].

Giving the post-digestate pulp the characteristics of compost will allow for the formation of granules at a moisture content above 40% m / m [Senesi and Loffredo 1998].

The conducted studies indicate the possibility of using the fermentation according to energy or fertilizer demand.

The test product is suitable for both fertilizer and renewable solid fuel production in the form of pellets or briquettes.

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[24] PN-EN 12879: 2004. Charakterystyka osadów ściekowych -- Oznaczanie strat przy prażeniu suchej masy osadu.

[25] PN-EN ISO 18134-3. Biopaliwa stałe -- Oznaczanie zawartości wilgoci -- Metoda suszarkowa. Część 3: Wilgoć w próbce do analizy ogólnej.

[26] PN-ISO 1928: 2002. Paliwa stałe. Oznaczanie ciepła spalania metodą spalania w bombie kalorymetrycznej i obliczanie wartości opałowej.