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UTILIZATION OF POST-FERMENTATION MAIZE WASTE BY THERMAL CONVERSION

Abstract: One of the essential requirements in the running of a biogas plant is the proper management of the by-product resulting from the production of biogas, i.e. the post-fermentation mass. One of the possibilities for the treatment of a fixed post-fermentation fraction is its combustion. In this study, the basic physicochemical properties of the post-fermented pulp obtained from maize waste are determined in terms of their combustion potential. Parameters such as elemental analysis, moisture content, volatile matter content, ash content, heat of combustion and calorific value were determined for the raw material without pre-treatment, as well as for the raw material after chemical hydrolysis and extrusion. The tested material was subjected to both acidic and alkaline hydrolysis. Acidic hydrolysis was carried out with sulfuric acid (concentration 3% and 7%) and alkaline hydrolysis with sodium hydroxide (concentration 1% and 3%). Under pre-treatment, the raw material was also subjected to low- and high-temperature extrusion. Low-temperature extrusion was carried out at 110°C, and high-temperature extrusion in the range 140–160°C. The purpose of the pre-treatment was to achieve the fragmentation of lignin, a substance not degraded by enzymatic hydrolysis. On the basis of the research, the suitability of the analyzed raw material for thermal utilization was determined. After drying, the residue after fermentation had high calorific value, similar to that of other types of biomass. It also had a lower content of volatile matter and increased ash content compared with the non-fermented raw material. High nitrogen content was a significant parameter distinguishing the studied material from other types of biomass. The decision to burn waste should be preceded by careful analysis of its physical and chemical properties, as this enables appropriate preventive action to be taken.

Keywords: maize waste, physicochemical properties, chemical hydrolysis, mechanical-thermal extrusion

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Introduction

In the process of anaerobic digestion only a part of the input material is transformed into biogas. The unfermented matter – containing organic compounds, microorganisms and mineral compounds – is known as post-fermentation mass, post-ferment, or post-fermentation pulp. That mass constitutes 90-95% of the initial volume of the feedstock put into the reactor. It is characterized by high water content, reaching 90-97%. In recent years it has become a popular practice in biogas plants to separate post-fermentation mass into two fractions, solid and liquid, as such separation widens the scope of its possible further uses.

There are numerous methods of utilization of post-fermentation mass. Those methods have both advantages and disadvantages, and differ with regard to their environmental impact. The main factor that determines the utilizability of post-fermentation mass is its quality, determined on the basis of its chemical composition and physical properties. Local conditions and legal regulations also play an important role [Jędrczak 2008; Czekała et al. 2012]. To some degree, difficulties with post-ferment utilization stem from the fact that its physical and chemical properties may vary significantly depending on the type of feedstock material and the origin of the material used for biogas production [Domińczak-Kuderko et al. 2015].

Post-fermentation mass is most often used as a fertilizer [Koszel and Lorencowicz 2015; Kataki et al. 2017]. It is a natural product that may be used as a substitute for artificial soil fertilizers. However, to be approved for such use the material has to meet restrictive hygienic and sanitary requirements, as well as certain limits related to toxic substance or heavy metal content [Kołodziejczyk 2012]. Another option is to use post-fermentation residue for the production of biofuels [Ray et al. 2008; Sannigrahi and Ragauskas 2011] or to incinerate it in special biomass-burning boilers.

Using post-fermentation material for heating purposes is in line with the current strategy of many countries to produce energy from renewable sources. At present, the energy policy of these countries is directed not only toward increasing the share of energy from renewable sources in their energy balances, but also toward restricting the use of forest biomass resources.

In the process of methane digestion carried out in a typical biogas facility, products of plant origin (maize straw, rape straw, hay) are decomposed only to a small degree. This is connected with their chemical composition, particularly the presence of lignin. Lignin is not easily digested by hydrolytic enzymes, and therefore its decomposition is limited. For that reason, input material is often subjected to pre-treatment that significantly increases the effectiveness of the digestion process [Ferrer et al. 2008; Mohammad and Keikhosro 2008; Wei et al. 2011; Zieminski et al. 2012; Akhtar et al. 2016; Pielhop et al. 2016].

The aim of this study was to determine physical and chemical properties of corn rachis before and after methane digestion, subjected to various methods of pre-processing, in order to determine its possible use in combustion processes and the resulting environmental threats.

Materials and methods

Research material and pre-treatment

The research material was corn rachis in the < 2.0 mm fraction. In order to disintegrate lignin, corn rachis material was subjected to pre-treatment. Two groups of pre-treatment methods were used: chemical and mechanical-thermal. Chemical pre-treatment was carried out with the use of sodium hydroxide and sulfuric acid. Alkaline hydrolysis was performed with 1% and 3% NaOH, and acidic hydrolysis with 3% and 7% H_2SO_4. Hydrolysis was carried out in laboratory conditions at a temperature of 100°C. After preliminary milling, the material was boiled for 3 h in aqueous solutions of 500 ml volume. After the hydrolysis process was completed, the material was filtered through a Buchner funnel and rinsed with water until neutral pH was reached. Mechanical and thermal treatment was performed in an extruder, in two temperature ranges: 110°C and 140-160°C (in the latter case the initial process temperature was 140°C and the final temperature was 160°C).

Methane fermentation

The methane digestion process was carried out in accordance with the procedure described in DIN 38 414/S8. It was performed in a 21-chamber biofermentor. The material was treated with an inoculum rich in methane bacteria. A diagram of the equipment is shown in figure 1.

The material was inoculated with an inoculum rich in methane bacteria, in the quantities given in table 1. The moisture content in the control material was approximately 7.2%. Dry mass content in the inoculum aqueous solution was approximately 2.7-2.9%, of which about 28–30% was minerals.

The obtained post-fermentation mass was divided into two fractions: solid and liquid. The solid fraction was pre-dried at 50°C and re-dried in laboratory conditions.

The content of the CHNS elements was determined with a FLASH EA 1112 Series (Thermo Electron Corporation), using the procedures set out in two standards: PN-EN ISO 16948:2015-07 and PN-EN ISO 16994:2015-06.

The heat of combustion and calorific value of the material were determined with a Kl-12Mn calorimeter in accordance with the PN-ISO 1928:2002 standard. Moisture was determined by means of the drying method, ash content in accordance with the procedure presented in T 211om-07, and the content of volatile matter according to the guidelines in PN-81/G-04516. All analyses were repeated three times, and the results were averaged.

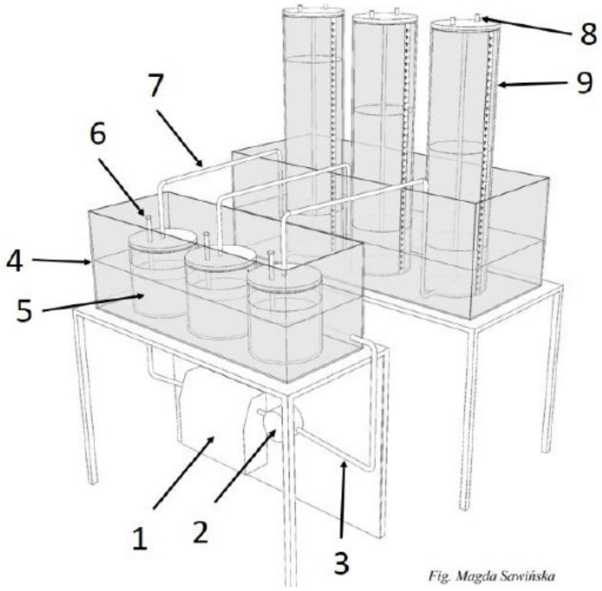

Fig. 1. Diagram of biofermentor used for biogas tests (three-chamber fragment): 1 – water heater with temperature regulator, 2 – water pump, 3 – insulated conductors of calefaction liquid, 4 – water coat, 5 – biofermentor with charge capacity 2 dm^3, 6 – sampling tubes, 7 – biogas transporting tube, 8 – gas sampling valve, 9 – biogas volume-scale reservoir [Lewicki et al. 2013]

Table 1. Quantity of raw material and inoculant used in the fermentation process

Material	Quantity of raw material (g)	Quantity of inoculant solution (g)
Control	13.5	1186.5
Hydrolysis 1% NaOH	51.0	1149.0
Hydrolysis 3% NaOH	60.0	1140.0
Hydrolysis 3% H_2SO_4	59.0	1141.0
Hydrolysis 7% H_2SO_4	62.0	1138.0
Extrusion 110°C	10.0	1190.0
Extrusion 140–160°C	10.0	1190.0

Statistical analysis

The statistical analysis was performed using STATISTICA 10, and consisted of ANOVA followed by a post hoc Tukey's HSD test. Identical letters in columns represent no differences at the significance level $p = 0.05$.

Results and discussion

Tables 2 and 3 present the content of basic elements (C, H, N, S) in the examined material before and after the digestion process. A desired feature of

Table 2. Elemental analysis of maize waste before fermentation

Material	Before fermentation			
	C (%)	H (%)	N (%)	S (%)
Control	46.22 ±0.0^{A}	6.17 ±0.1^{D}	0.60 ±0.1^{D}	0.15 ±0.0^{B}
Hydrolysis 1% NaOH	44.49 ±0.1^{D}	6.51 ±0.0^{A}	0.02 ±0.0^{A}	–^{A}
Hydrolysis 3% NaOH	44.19 ±0.1^{C}	6.55 ±0.0^{A}	–^{A}	–^{A}
Hydrolysis 3% H_2SO_4	46.03 ±0.1^{A}	6.30 ±0.0^{BC}	0.44 ±0.0^{C}	0.14 ±0.0^{B}
Hydrolysis 7% H_2SO_4	46.11 ±0.1^{A}	6.36 ±0.0^{C}	0.15 ±0.0^{B}	–^{A}
Extrusion 110°C	46.09 ±0.2^{A}	6.26 ±0.1^{B}	0.90 ±0.0^{E}	–^{A}
Extrusion 140-160°C	46.20 ±0.2^{A}	6.50 ±0.0^{A}	1.13 ±0.0^{F}	–^{A}

– not detected (<0.01%).
Homogeneity groups: same letters in each column indicate that there is no statistical difference between the samples according to Tukey's HSD test at $P < 0.05$.

Table 3. Elemental analysis of maize waste after fermentation

Material	After fermentation			
	C (%)	H (%)	N (%)	S (%)
Control	43.78 ±01^{E}	5.12 ±0.0^{C}	3.11 ±0.0^{E}	0.33 ±0.0^{AB}
Hydrolysis 1% NaOH	37.60 ±0.2^{A}	4.40 ±0.1^{AD}	3.03 ±0.0^{C}	0.31 ±0.1^{A}
Hydrolysis 3% NaOH	36.93 ±0.2^{B}	4.32 ±0.1^{D}	2.97 ±0.0^{BC}	0.26 ±0.1^{AD}
Hydrolysis 3% H_2SO_4	40.31 ±0.2^{C}	4.76 ±0.1^{ABC}	2.76 ±0.0^{F}	0.32 ±0.0^{AB}
Hydrolysis 7% H_2SO_4	41.48 ±0.2^{D}	4.92 ±0.1^{BC}	2.54 ±0.0^{D}	0.39 ±0.0^{B}
Extrusion 110°C	37.71 ±0.2^{A}	4.54 ±0.0^{AB}	2.92 ±0.0^{AB}	0.20 ±0.0^{CD}
Extrusion 140-160°C	37.80 ±0.3^{A}	4.47 ±0.0^{AB}	2.88 ±0.0^{A}	0.17 ±0.0^{C}

– not detected (<0.01%).
Homogeneity groups: same letters in each column indicate that there is no statistical difference between the samples according to Tukey's HSD test at $P < 0.05$.

solid fuels is a high content of elemental carbon, as this directly influences the energy indices. Elemental carbon content in the control sample, i.e. the sample not pre-treated and not subject to fermentation, was measured at 46.22%. The tested raw material had a high carbon content compared with other types of biomass. The content of carbon in biomass is about 38% to 50% [Cuiping et al. 2004]. Cuiping et al. [2004] report that the carbon content of maize waste is slightly lower than that indicated (42.69%). Pre-treatment, both chemical and mechanical-thermal, produced little change in carbon content in the treated material; only alkaline hydrolysis caused a drop in the elemental carbon content, by 3.7-4.4%. Hydrogen content in the control sample was 6.17%. Following pre-treatment, it increased slightly (within the range 1.5-6.1%). Nitrogen content in the material before fermentation ranged from 0 to 1.13%. The nitrogen content depends on the type of biomass, and varies from 0.1% to 2.0% [Cuiping et al. 2004]. Following hydrolysis carried out with 1% and 3% NaOH and 3% and 7% H_2SO_4 it dropped to 0.15-0.44%, but it increased to a level of 0.9-1.13% after extrusion. The presence of elemental sulfur was detected in two samples only, the control and the sample after hydrolysis with 3% H_2SO_4, with levels of 0.15% and 0.14%, respectively. The literature shows that biomass has a higher content of hydrogen and a smaller content of elemental carbon than coal, which results in a lower energy value [Cuiping et al. 2004].

The fermentation process to which the investigated material was subjected caused changes in its elemental composition. Elemental carbon and hydrogen content dropped, while nitrogen and elemental sulfur content increased. This was observed both for the control sample and for the examined material after pre-treatment. Carbon content in the control sample after the fermentation process dropped from 46.22% to 43.78%, i.e. by approximately 5.3%, while for the pre-treated material carbon content decreased by 15.5-18.8%. Hydrogen content in the control sample dropped from 6.17% to 5.12% (by 17%), and in the other materials after chemical treatment and extrusion from 6.2-6.55% to 4.32-4.92%. Nitrogen content after fermentation increased from a level of 0-1.13% to 2.54-3.11%, and sulfur content increased from 0-0.15% to 0.17-0.39%.

From the point of view of energy generation, a decreased content of elemental carbon in post-fermentation material is undesirable, as the value of that parameter is in a directly proportional relation to the gross calorific value of the fuel type [Cichy and Prądzyński 1995]. A drop in hydrogen content, on the other hand, has positive consequences. In the thermal process, hydrogen is oxidized, forming a water molecule, and thus lowering the net calorific value of the fuel. Increased sulfur and nitrogen content in a material meant for combustion may have negative consequences for the natural environment. The combustion of a material that contains elemental sulfur may result in higher atmospheric emissions of SO_2, while nitrogen content may result in emissions of the fuel-related nitrogen oxides (NO_x).

The results of determination of the fueling properties of the examined materials are presented in tables 4 and 5.

Table 4. Content of moisture, volatile matter, ash and heat of combustion of maize waste before fermentation

Material	Moisture (%)	Volatile matter (%)	Ash (%)	Gross calorific value (MJ/kg)	Net calorific value (MJ/kg)
Control	7.2 ±0.1	73.99 ±0.1^{A}	0.69 ±0.0^{A}	17.22 ±0.0^{B}	15.70
Hydrolysis 1% NaOH	6.6 ±0.1	78.97 ±0.1^{F}	0.99 ±0.1^{C}	16.18 ±0.0^{A}	14.60
Hydrolysis 3% NaOH	6.7 ±0.1	79.03 ±0.1^{G}	0.78 ±0.1^{AB}	16.19 ±0.2^{A}	14.60
Hydrolysis 3% H_2SO_4	5.2 ±0.1	78.73 ±0.1^{D}	0.90 ±0.1^{BC}	17.67 ±0.1^{E}	16.17
Hydrolysis 7% H_2SO_4	5.8 ±0.2	78.74 ±0.1^{E}	0.75 ±0.0^{A}	17.47 ±0.1^{D}	15.94
Extrusion 110°C	6.7 ±0.1	73.80 ±0.1^{C}	2.76 ±0.1^{D}	17.08 ±0.1^{C}	15.55
Extrusion 140-160°C	6.5 ±0.1	73.10 ±0.3^{B}	2.69 ±0.1^{D}	17.20 ±0.0^{B}	15.62

Homogeneity groups: same letters in each column indicate that there is no statistical difference between the samples according to Tukey's HSD test at $P < 0.05$.

Table 5. Content of moisture, volatile matter, ash and heat of combustion of maize waste after fermentation

Material	Moisture (%)	Volatile matter (%)	Ash (%)	Gross calorific value (MJ/kg)	Net calorific value (MJ/kg)
Control	9.8 ±0.1	48.24 ±0.1^{B}	26.30 ±0.2^{C}	14.69 ±0.0^{B}	13.33
Hydrolysis 1% NaOH	11.0 ±0.2	45.77 ±0.1^{A}	31.10 ±0.2^{A}	13.30 ±0.1^{A}	12.07
Hydrolysis 3% NaOH	10.1 ±0.2	46.17 ±0.1^{D}	31.22 ±0.2^{A}	13.31 ±0.0^{A}	12.12
Hydrolysis 3% H_2SO_4	11.2 ±0.2	47.20 ±0.1^{E}	27.38 ±0.0^{D}	14.61 ±0.0^{B}	13.30
Hydrolysis 7% H_2SO_4	11.8 ±0.1	48.40 ±0.1^{B}	25.74 ±0.2^{B}	15.24 ±0.0^{D}	13.87
Extrusion 110°C	12.8 ±0.2	45.30 ±0.0^{C}	31.48 ±0.1^{A}	14.54 ±0.0^{E}	13.23
Extrusion 140-160°C	10.7 ±0.1	45.93 ±0.1^{A}	30.04 ±0.1^{E}	13.90 ±0.0^{C}	12.67

Homogeneity groups: same letters in each column indicate that there is no statistical difference between the samples according to Tukey's HSD test at $P < 0.05$.

The humidity of the examined material prior to the fermentation process was between 5.2% and 7.2%, and the humidity of the examined solid fraction of post-fermentation mass ranged from 9.8% to 12.8%.

The gross calorific value (GCV) of the input material was fairly high, ranging from 16.18 to 17.67 MJ/kg. The net calorific value (NCV), reflecting the actual thermal effect, ranged between 14.60 and 16.17 MJ/kg, and is comparable to the NCV of other fuels. The GCV of different types of biomass ranges from 14 to 23 MJ/kg [Erakhrumen 2009; Gravalos et al. 2010; Telmo and Lousada 2011; Günther et al. 2012; Patel and Gami 2012; Schmatz et al. 2016]. The differences are mainly due to variable carbon and ash content [Gravalos et al. 2016]. However, it should be remembered that the net calorific value of every material is closely correlated with water content. Moisture is considered a contaminant for thermochemical processes [Núñez-Regueira et al. 2002] and reduces the efficiency of the energy system. There is a practical limit of autogenous combustion at about 67% moisture [Bahadori et al. 2014]. The NCV of the feedstock material was 15.70 MJ/kg, close to the NCV determined for the material after extrusion and acidic hydrolysis. The lowest NCV was found for material after alkaline hydrolysis. This was correlated with the content of elemental carbon in the raw material after the alkaline treatment, which was also the lowest. According to the literature, the content of elemental carbon is directly proportional to the heat of combustion of a fuel [Cichy and Prądzyński 1995]. Forestry biomass fuels have a slightly higher calorific value than agricultural biomass [Cuiping et al. 2004].

After the fermentation process, the GCV and NCV were much lower than the values recorded for the feedstock. The gross calorific value for the post-fermentation mass was from 13.30 to 15.24 MJ/kg, and the net calorific value from 12.07 to 13.87 MJ/kg. This is related to changes in the elemental composition caused by microbiological decomposition of organic substances, which took place during the fermentation process.

Apart from the lower gross and net calorific value, the post-fermentation mass also had a lower content of volatile matter. The volatile matter content for the input material was between 73.10% and 79.03%, and after the fermentation process it dropped to values between 45.30% and 48.40%. Volatile matter content in fuel is a critical parameter for the combustion process. The higher the content, the easier the ignition and the faster the combustion. Volatile matter content in biomass ranges from 61.10% to 76.50% [Cuiping et al. 2004]; in conventional fuels it is 20–40% for hard coal and about 50% for brown coal [Kucowski et al. 1994; Cuiping et al. 2004], i.e. it is lower than for the input material and similar to the content in the post-fermentation material.

Fermentation had a significant impact on ash content. Ash content in biomass is varied, ranging from 0.89% to 15.24% [Cuiping et al. 2004]. The control sample contained 0.69% ash; the material after pre-treatment contained from 0.75% to 2.76%. Of the pre-treatment methods used, the extrusion process had the greatest impact on ash content, causing it to increase to 2.69% and 2.76%. Pérez-Navarrete et al. [2007] also found an increase in ash content after extrusion. The increase in the ash content caused by the extrusion process may be due to changes in polysaccharides. Polysaccharides can be fragmented to form reactive molecules that can create new bonds [Camire 1998]. According to Karunanithy and Muthukumarappa [2010], extrusion causes the depolymerization of cellulose, hemicelluloses, lignin and protein. Chemical treatment, both alkaline and acidic, did not bring about any major changes in that parameter. The post-fermentation material, on the other hand, contained significantly more ash, from 25.74% to 31.48%. This significant growth in ash content in post-fermentation mass probably results from the inoculum (fermentation-facilitating substance) added to the material in the digestion process, particularly in the first hours of fermentation. Sannigrahi and Ragauskas [2011] presented the ash contents of the fermentation residues from four feedstocks (switchgrass, miscanthus, hybrid poplar and red maple). The ash contents in the residues from miscanthus and switchgrass (11.8% and 18.95%) were much higher than in the two hardwood feedstocks (4.3% and 7.1%). Ash is considered as a byproduct, and may create serious problems in the combustion process [Gravalos et al. 2016]. The ash content in biomass is linked to problems with the equipment, such as slag formation, fouling, sintering and corrosion [Cuiping et al. 2004; Biedermann and Obernberger 2005; Gravalos et al. 2016].

Ash from biomass contains K, Mg and P and heavy metals, which means that it is not possible to recycle it on agricultural fields or forests [Gravalos et al. 2016].

Conclusions

Methane fermentation, consisting in anaerobic digestion of organic matter, is an efficient process yielding biogas. The waste that is left after that process, i.e. post-fermentation material, may also be used for energy-generating purposes. This is confirmed by examination of post-fermentation material obtained from fermented corn rachis.

The fermentation process caused significant changes in the elemental composition of the examined material, altering its carbon, hydrogen, nitrogen and sulfur contents. Although the direction of the changes is generally disadvantageous, the material can still be used to substitute other fuel types. The adverse changes are decreased elemental carbon content and increased nitrogen and sulfur content. The change in hydrogen content is advantageous. The drop in hydrogen content in post-fermentation material means a smaller energy demand, as hydrogen is an additional source of water that has to be evaporated in the thermal process. The decrease in the gross calorific value of the post-fermentation material to a level of 13.30-15.24 MJ/kg does not preclude its use as a fuel, as this is similar to the levels found for various types of untreated biomass. The same applies to the drop in volatile material content from 73.99--79.03% to 45.30-48.24%. Conventional fuels such as hard coal or brown coal have a volatile matter content similar to that found for the material obtained after fermentation.

The significant increase in ash content following fermentation is undoubtedly a problem. Ash is a by-product that has to be utilized after the thermal process; it also adversely influences the energy value of fuel and the course of the combustion process.

Human development is generating constant demand for energy, while traditional fuel reserves (mainly fossil fuels such as coal, oil and gas) are running out and their use increases environmental pollution; therefore, the search for alternative fuel materials is a necessity. As research on clean biomass materials such as wood progresses, scientific interest is currently shifting towards materials that are harder to utilize, even those with high ash or humidity content.

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T 211om-07 Ash in Wood, Pulp, Paper, and Paperboard: Combustion at 525°C

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