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## FROM BIOMASS TO BIOGAS: A PILOT PLANT TECHNOLOGY

### OD BIOMASY DO BIOGAZU: TECHNOLOGIA PILOTAŻOWA

#### Abstract

The pilot plant technology was developed in order to improve biomass digestibility and to evaluate biogas yields. It is composed of two main sections namely pretreatment and fermentation. The pretreatment units include using of the new type of mill – macerator, ball mill and thermal-expansionary hydrolysis. To evaluate biogas potential batch tests according VDI 4630 are used. They can be carried out either in lab-testing unit (8x volume 0.5 liters) or in lab-fermenter (20 ÷ 35 liters).

*Keywords: biogas batch test, fermenter, size reduction, macerator, thermal-expansionary hydrolysis*

#### Streszczenie

Technologia instalacji pilotażowej została opracowana dla poprawy przetwarzania biomasy i oceny wydajności produkcji biogazu. Składa się z dwóch głównych etapów: obróbka wstępna i fermentacja. Obróbka wstępna polega na mieleniu za pomocą maceratora nowej konstrukcji i młyna kulowego oraz technologii TEH rozprężania rozdrobnionego materiału. Potencjał produkcji biogazu określano testami zgodnymi ze standardem VDI 4630. Mogą być one przeprowadzane w laboratorium jednostkowym (8x objętość 0,5 l) lub w fermentorze laboratoryjnym (20 ÷ 35 litrów).

*Słowa kluczowe: badania biogazu, fermentor, rozdrabnianie, macerator, instalacja rozprężna TEH*

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## 1. Introduction

The application of anaerobic digestion technology is actually rapidly growing around the world because of environmental and economic benefits. For its implementation is necessary to determine biogas potential for various types of wastes which can be found in such materials as agriculture and forestry wastes, municipal solid waste, waste paper, wood and herbaceous energy crops. Incidentally, the production of these lignocellulosic substrates has been estimated in  $1 \cdot 10^{13}$  tons worldwide [1]. This feedstock is generally composed of cellulose, hemicellulose, lignin and wide variety of organic and inorganic compounds. Both cellulosic and hemicellulosic fractions are converted to monosaccharides that can be subsequently fermented to biogas. However, the inherent properties caused by composite structure make them resistant against enzymatic attack, see Fig. 1.

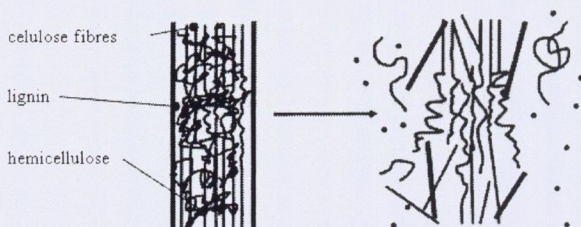


Fig. 1. The effect of pretreatment on lignocellulosic structure [3]

Rys. 1. Wpływ obróbki wstępnej na strukturę lignocelulozową

Biodegradation of these substrates is very slow and extent does not exceed 20% [2]. Therefore, pretreatment of biomass is an essential step in order to increase biomass digestibility. Various pretreatment technologies have been developed including dilute acid, ammonia, steam explosion, ethanol solvolysis, ammonia fiber explosion or sulfite pretreatment. However, they generally entail the use of either chemicals or high energy demand which makes them not economically feasible [4].

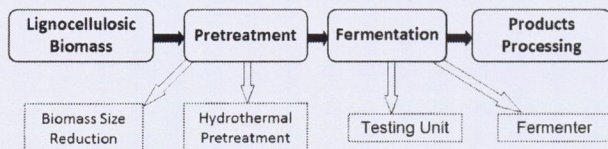


Fig. 2. Lab-technology for biogas yield evaluation

Rys. 2. Schemat technologii określania wydajności laboratoryjnej produkcji biogazu

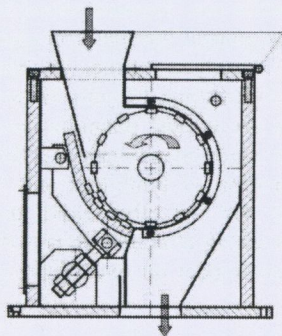
The pilot-plant biogas technology as presented in Fig.1 was developed in order to evaluate influence of various pretreatments on biodegradability and biogas production. The first part of lab-technologynamely the pretreatment sectionallows the using of size reduction machines and hydrothermal pretreatment. Afterwards, treated material can be

anaerobically digested either in testing unit or fermenter where extent and quality of biogas are evaluated in relation to energy requirement of previous pretreatment.

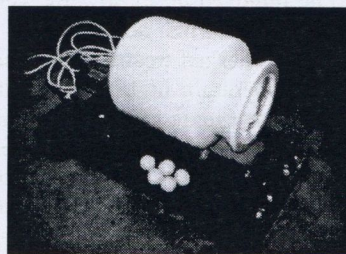
## 2. Pretreatment methods in the pilot plant technology

### 2.1. Mechanical comminution

Biomass size reduction technology is an essential part of each biogas plant. The goal of this pretreatment is to disrupt inherent structure of lignocelluloses, decrease particle size and degree of cellulose crystallinity. In general, the recommended final particle size is  $1 \div 2$  mm for effective digestibility. The size reduction increases total hydrolysis yield by  $5 \div 25\%$  and also reduces digestion time by  $23 \div 59\%$ . But on the other hand, comminution is a very expensive operation that consumes about 33% of total electrical demand [5]. Thus reducing the energy requirement as well as the right solution for disintegration of biomass would improve the whole process economics.



A) Macerator



B) Ball mil

Fig. 3. Size reduction machines used in lab-scale technology

Rys. 3. Młyny laboratoryjne

Knife, hammer, roll and colloid mills are commonly used for disintegration of biomass. In the pilot plant technology, the new prototype of mill called *MACERATOR* has been tested, see Fig. 3A. The idea of this machine is to combine the most efficient milling principles being in knife and roll mill. The macerator consists of one horizontal roll and drum sieve both with sharp-edged segments. The roll-sieve gap is easily adjustable by screws. In principle, lignocellulosic biomass is fed into the gap between roll and sieve where becomes soaked in hot water. Due to high shear and cut forces biomass is disrupted and reduced in size goes away through the holes in sieve into storage vessel. The variable parameters are processing amount of biomass, gap size, rotational roll speed, flow rate and temperature of hot water. The effectiveness of macerator is evaluated by energy demand for comminution, structure and biogas tests of treated material.

The second size reduction machine used in lab-scale technology is ball mill, see Fig. 3B. This universal equipment has been found to be a very efficient machine for disintegration of lignocelluloses and it can be used both for wet and dry way of milling. The shear and compressive forces cause disruption of lignocellulosic matrix, reduction in size and decrease in cellulose crystallinity. Nevertheless, ball milling has been also found to be a time-consuming operation with the highest energy demand for comminution [5]. The effectiveness of ball mill is determined by the same parameters as macerator before.

## 2.2. Thermal-expansionary hydrolysis

Both agriculture and municipal biogas plants especially are currently endeavored to implement a new physical pretreatment technology called as thermal-expansionary hydrolysis (TEH), liquid hot water (LHW) or hot compressed water (HCW) pretreatment technologies well. During this treatment, lignocellulosic biomass is heated in water maintained by pressure in liquid state. Over the temperature 160°C passive lignocellulosic structure is provoked to make alterations causing increase in accessibility of nutrients for enzymatic attack. The objective of TEH is to solubilize mainly hemicellulose, to make cellulose more accessible and to avoid formation of inhibitors. Hemicellulose has been shown to be removed up to 80% by weight. Nevertheless, during thermal processes a part of hemicellulose forms acids which are assumed to catalyze further hydrolysis of hemicellulose. Thermal treatment also causes partial solubilization of lignin. However, for processing temperature higher than 220°C, the products are almost phenolic compounds having primarily a toxic effect on methanogens [1]. The major advantages of TEH are no addition of chemicals, no sludge is generated, no formation of inhibitors and low-cost reactor construction because of low-corrosion potential. On the other hand, the major disadvantage is heat-energy demand caused by high processing temperature [6]. Pérez [7] reported that thermal-expansionary hydrolysis has a great impact on overall process cost accounting up to 33% of total cost.

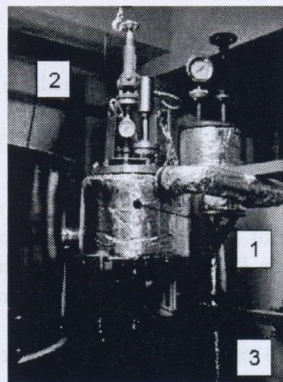
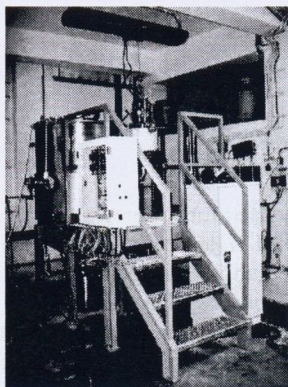


Fig. 4. The apparatus for thermal-expansionary hydrolysis (TEH)  
1 – hydrolyser, 2 – expansion vessel, 3 – ball valve with pneumatic actuator

The lab TEH in batch mode can be generally used for treatment of various biomasses. It is composed of three main parts namely hydrolyser, expansion vessel and ball valve equipped with pneumatic actuator, see Fig. 4. The hydrolyser is double-jacketed pressure vessel which allows treatment of biomass with volume up to 8 liters at maximum processing temperature 200°C and pressure 1.6 MPa. Substrate is indirectly heated by oil circulating in double jacket. Incidentally, the electrical spiral with power 12 kW is used for oil heating. The expansion vessel is an apparatus where atmospheric pressure is maintained inside and it is used for storage of expanded substrate. The third main part namely ball valve keeps pressure space in hydrolyser separate from atmospheric space in expansion vessel.

The processing of biomass by batch TEH is based on this principle. The hydrolyser is stuffed with a suspension containing lignocelluloses and hot water at elevated temperatures. Substrate is heated and afterwards, when a processing temperature is reached, it is kept constant for a processing time. As follows, the ball valve is rapidly opened and substrate immediately expands to expansion vessel. Two products are formed during expansion namely vapor and hydrolyzate. After vapor condensation, expanded material is removed out of the expansion vessel and fermentation tests are carried out.

### 2.2.1. The impact of expansion on particle size distribution

Because of temperature resistance up to 250°C [2], purge microcrystalline cellulose was used as a model material in order to determine the impact of rapid decompression on particle size distribution.

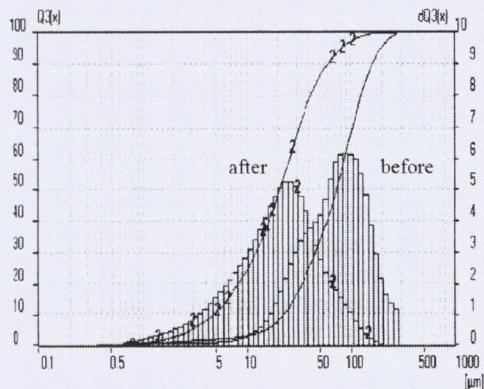


Fig. 5. Cellulose size distribution before and after expansion

Rys. 5. Rozkład wielkości celulozy przed i po rozprężeniu

The suspension containing 10% of cellulose by weight was treated in hydrolyser at processing temperature  $T=200^{\circ}\text{C}$  and residence time  $t=5$  min. The particle size distribution before and after expansion as shown in Fig. 5 was investigated by *FRITSCH analysette 22 COMPACT*. In detail, the measured values of mode for particle size distribution before and after expansion were 87.433  $\mu\text{m}$  and 25.753  $\mu\text{m}$ . Therefore, the decrease in particle size caused entirely by expansion was 3.5 times. Thus this experiment

bears out supposition that not only the effect of temperature causes decrease in particle size. The expansion of material and its implementation in real technologies was found out to be as an intensive step for reduction biomass in size.

### 2.2.2. The treatment of wheat straw

For example in first experiments, the suspension containing non disintegrated wheat straw with 5% by weight was treated. This material was initially incubated at temperature 60°C to reach good straw maceration. Then the substrate was filled into hydrolyser. The initial pH value was  $7.14 \pm 0.05$  and the glucose yield  $0.14 \pm 0.02 \text{ g} \cdot \text{l}^{-1}$ .

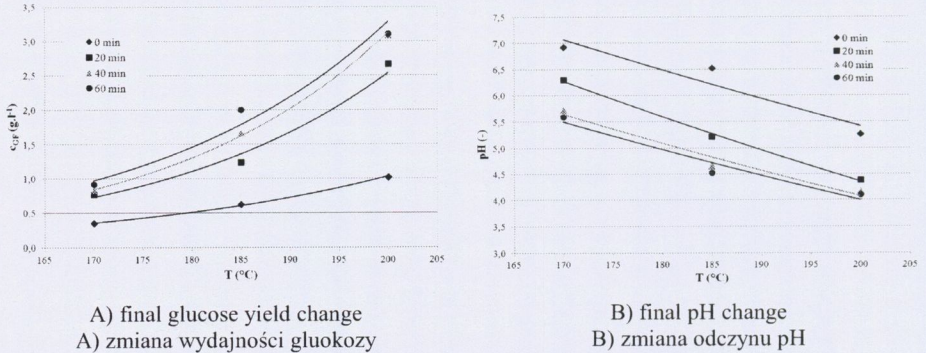


Fig. 6. The dependence of final glucose yield, pH on temperature and processing time

Rys. 6. Zależność wydajności glukozy i odczynu pH od temperatury i czasu trwania procesu

The Fig. 6 plots the dependencies of final glucose yield and pH on thermal conditions 170 ÷ 200°C and processing time 0 ÷ 60 min. The glucose yield (Fig. 6A) grows up with increasing temperature and time. Whereas the pH values (Fig. 6B) grow down with increase in temperature and time. Generally, the TEH pretreatment causes that liquid water under pressure penetrates into pores in biomass. Because of rapid expansion liquid water changes phase to vapor and associated volumetric change causes disruption of substrate and cell walls especially. These effects cause not only increase in glucose yield and pH changes but primarily increase in biodegradation rate of biomass. The influences of TEH on structure of wheat straw and boxboard are shown in Fig. 7.

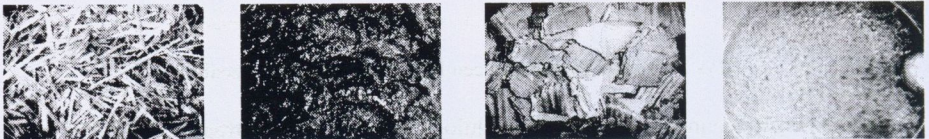


Fig. 7. Influence of LHW on structure of biomass – processing parameters 200 °C, 20 min

Rys. 7. Wpływ TEH na strukturę biomasy – parametry procesu 200 °C, 20 min

### 3. Fermentation tests in the pilot plant technology

To evaluate an anaerobic biodegradability of wastes a test known as Biochemical Methane Potential (BMP) is commonly used. Generally known, determination of biogas potential can be carried out in batch or continuous mode. Nevertheless, batch systems are more usual because of its easier set up and simplicity in monitoring and evaluation. All batch tests are based on the same principle – measuring of biogas/methane production. The basic approach is to incubate a waste with an anaerobic inoculum and measure biogas/methane production [8].

#### 3.1. The lab-testing unit

The lab-testing unit shown in Fig. 8 is almost used for primary testing of BMP. Anaerobic digestion experiments are carried out in accordance with European standards VDI 4630 [9] and ČSN EN ISO 11734 [10]. In detail, 8 glass batch digesters of 0.5 liters capacity are used. These bottles are filled up with mixture of tested waste with seeding sludge from an agriculture biogas plant processing primarily straw and corn silage. To prevent inhibition in fermentation batch, the substrate should not be overlarge in proportion to the seeding sludge. Thus, the follow condition has to be respect [9]:

$$\frac{VS_{SUB}}{VS_{SLU}} \leq 0.5 \quad (1)$$

The fermentation batch has to also contain organic mass from the seeding sludge in range  $1.5 \div 2\%$   $VS$  by weight [9]. Therefore, the ratios of  $VS_{SUB}$  to  $VS_{SLU}$  in values 0.3 and 0.5 are being used in experiments.

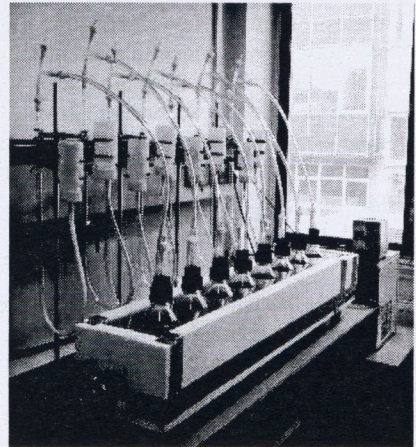
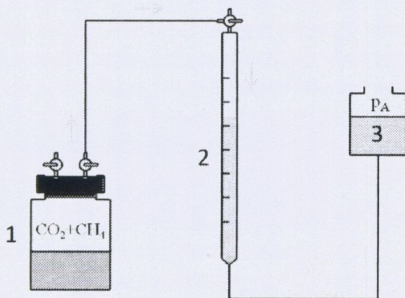


Fig. 8. The lab-testing unit for biogas yield evaluation  
1 – bottle with substrate, 2 – burette, 3 – balancing bottle

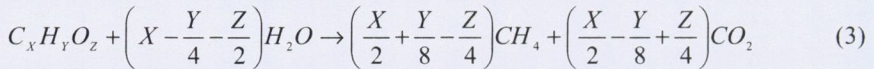
Rys. 8. Stanowisko laboratoryjne do określania wydajności produkcji biogazu  
1 – zbiornik substratu, 2 – biureta, 3 – zbiornik wyrównawczy

As for experimental set up, the 3 digesters are used only for liquid phase, next 2 bottles only for treated solid phase, 1 bottle for both liquid and solid phases, 1 digester for untreated biomass and the last one only for seeding sludge itself. The digesters are incubated under mesophilic conditions at constant temperature of 35°C which is maintained through a water bath. The gas measuring system is based on simple volumetric method. The biogas produced inside moves to external burette where displaces an equivalent volume of barrier solution from balancing bottle providing constant pressure conditions, see Fig. 8. The amount of biogas quantity is monitored daily except the beginning of the test when increase in biogas volume is evaluated more often. The anaerobic digestion test is proclaimed to be finished when volumetric changes are lower than 1% of total biogas volume. The quality of biogas is analyzed by absorption of carbon dioxide CO<sub>2</sub> into potassium hydroxide KOH.

The evaluations and characteristics of tested material and anaerobic sludge are based on initial and final analyzes of pH, chemical oxygen demand, total solid and volatile solid content. Incidentally, theoretical methane extent can be easily expressed as volume of methane related to weight of biomass calculated as [12]:

$$Y_{CH_4g} = 0.35 \cdot COD \quad (2)$$

If the elementary composition of substrate (C, H, O, nutrients) is known, methane yield can be calculated by using of Bushwell equation [12]:



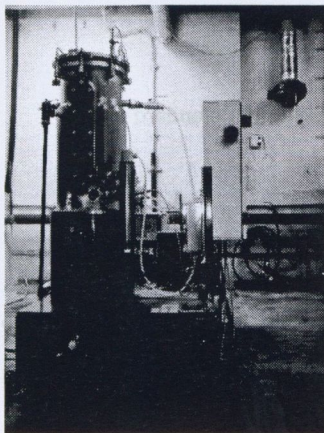
Nevertheless, these reviewed equations do not take in account the real biodegradability of biomass caused by lignocellulosic structure. This limitation gives rise to lower methane yields achieved by batch tests in relation to theoretical calculations. However the estimation of methane production according (2) or (3) is accurate only for easily digestible materials with simple structure [12]. In addition to waste in initial state, elementary composition (C, N, S, P, Mg, K) or analysis of fats, carbohydrates, proteins and lignin contents are also investigated.

### 3.2. The lab-fermenter

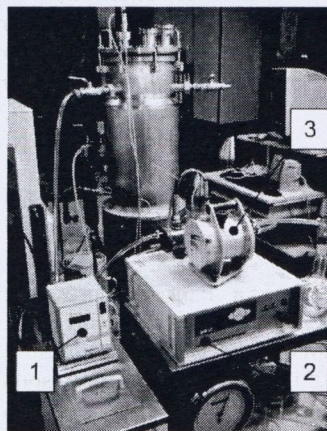
Secondly, the lab-fermenter as presented in Fig. 9 is used for more detailed BMP tests and for scale-up authentication especially. These tests are carried out according the same processing parameters and rules as in testing units. The lab-fermenter allows an investigation of BMP tests with volume of substrate in range 20 ÷ 35 liters. Tested substrate is also indirectly incubated under mesophilic conditions usually at temperature 35°C by hot water which circulates in external double-jacket of fermenter. Owing to adjustable pressure difference, produced biogas is moved to the part of biogas analysis, see Fig. 9B. At first, biogas is cooled because of humidity removal, then quality and flow rate of biogas are analyzed in detail. The evaluation and characteristics of waste during biodegradation process are provided by continual measurement of process parameters both in liquid and



gas phase. In liquid phase, total organic carbon, total nitrogen, pH, chemical oxygen demand, redox potential and temperature are measured. In gas phase, pressure, temperature, humidity, amount and quality of biogas are analyzed. The control and monitoring of anaerobic process is available via websites [11].



A – equipment  
A – wyposażenie



B – part of biogas yield investigation  
1 – cooling, 2 – gas analyzer, 3 – flow meter  
B – układ pomiaru wydajności biogazu  
1 – chłodzenie, 2 – analizator gazu, 3 – przepływomierz

Fig. 9. The lab-fermenter

Rys. 9. Fermenter laboratoryjny

#### 4. Conclusion

The lab-pilot plant technology was developed to determine biogas potential in various types of wastes. This technology provides several methods of pretreatments aiming to enhance anaerobic biodegradability of substrate and increase biogas yield including its quality. These methods are based on hydrothermal pretreatment or mechanical comminution.

- The lab-ball mill and the new prototype called macerator are used for biomass comminution. The energy demands to reach required final particle size and structure are investigated.
- The thermal-expansive hydrolysis is used to obtain more digestible biomass. Its effectiveness grows up with increasing processing temperature and time. The TEH pretreatment ensures increase in biogas extent, decrease in residence time in fermenter, homogenization and pumping are easier.
- Biochemical methane potential tests can be carry out either by testing units in simply way of use or by lab-fermenter laboriously. The testing units are primarily used for biogas yield investigation, whereas the lab-fermenter for a scale-up investigation.

### Symbols

$COD$	– chemical oxygen demand of tested biomass	$[g \cdot l^{-1}]$
$T$	– processing temperature	$[^{\circ}C]$
$t$	– residence time	$[-]$
$VS$	– volatile solid content	$[-]$
$VS_{SLU}$	– volatile solid matter in seeding sludge by weight	$[-]$
$VS_{SUB}$	– volatile solid matter in feedstock by weight	$[-]$
$Y_{CH_4g}$	– specific methane ( $CH_4$ , substrate) yield under standard conditions ( $0^{\circ}C$ , 101.325 kPa)	$[l \cdot g^{-1}]$

### Literature

- [1] Alvira P., et al.: *Bioresource Technology*, 101, (2010), 4851-4861.
- [2] Pandey A.: *Handbook of plant-based biofuels*, CRC Press, New York 2009.
- [3] Mosier N., et al.: *Bioresource Technology*, 96, (2005), 673-686.
- [4] Xiao L.O., et al.: *BioResources*, 6/2, (2011), 1576-1598.
- [5] Krátký L., Jirout T.: *Chem. Eng. and Tech.*, 34/3, (2011), 391-399.
- [6] Taherzadeh M.J., et al.: *Inter. Jour. Mol. Sci.*, 9, (2008), 1621-1651.
- [7] Pérez J.A., et al.: *Jour. of Chem. Tech. and Biotech.*, 82, (2007), 929-938.
- [8] Raposo F., et al.: *Renew. and Sustain. Ener. Rev.*, 11, (2001), 861-877.
- [9] VDI 4630: *Fermentation of org.materials*, Verein Deutscher Ingenieure, 2006.
- [10] ČSN EN ISO 11734: *Evaluation of the "ultimate" anaerobic biodegradability of organic compounds in digested sludge*, Czech Office for Standards, 1999.
- [11] Skočilas J.: 56<sup>th</sup> National Congress of Chemical and Process Engineering CHISA 2009, 2009, 16.
- [12] Procházka J., Dohányos M.: *Paliva*, 3, (2011), 47-52.

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