## LABORATORY SCALE ANAEROBIC DIGESTION AT THE ITALIAN BIOMASS RESEARCH CENTRE

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ABSTRACT: Anaerobic digestion is becoming an important source for renewable methane production when disposing of residual biomasses. Nevertheless few data are available in the Literature for mass and energy balances for different feedstocks, which are of great interest both for feasibility studies and design of power plant.

This paper describes the ongoing activities at the Italian Biomass Research Centre on anaerobic digestion, from preliminary results with batch glass vessels on the Lake algae to the design and construction of a laboratory scale monitored steel digester and of its gas-meter.

Keywords: anaerobic digestion, batch reactor, biogas, gas-meter.

#### 1 INTRODUCTION

Anaerobic process has been traditionally used for excess sludge digestion in wastewater treatment plants, achieving useful methane production while reducing the oxygen demand. The process is complex and it is catalyzed and carried out by a consortium of microorganism that in a joined action convert complex macromolecules into low molecular weight compounds such as methane, carbon dioxide, water and ammonia.

Nowadays the process is also convenient for generic residual biomasses, with high water content because the methane produced may be used as a renewable energy source, which can receive public funding.

The technical and economical feasibility of an industrial anaerobic digestion plant is strongly dependent on methane yield and purity, which may vary considerably with biomass chemical composition and process parameters, such as temperature and retention time. These dependencies are poorly available in the Literature and in any case only for few biomasses, while data from producers and plant manufacturing companies are difficult to obtain.

The Italian Biomass Research Centre (Centro Ricerca Biomasse – CRB), funded by the Ministry of Environment at the University of Perugia, has ongoing advising activities on residual biomass use for energy purposes. When humid biomasses were considered, in particular lake algae and Organic Fraction of Municipal Solid Waste (OFMSW), the above mentioned opportunities and lack of data required the construction of an experimental facility, to verify methane yield and composition at different temperatures.

The production of biogas by anaerobic digestion of algae was investigated using batch experimental reactors, with a working capacity of  $1.5 \ l$  at  $37^{\circ}$ C. The biogas produced was significant. Therefore a pilot batch reactor, with a working capacity of  $17 \ l$ , was designed and realized, in order to carry out qualitative and quantitative biogas monitoring. Finally OFMSW was used to calibrate the system.

# 2 FIRST PROTOTYPES OF ANAEROBIC DIGESTER

The eutrophication is a natural process in the aging of lakes, but human activities can greatly accelerate it by increasing the rate of nutrients and organic substances entering the aquatic ecosystems from the surrounding watersheds. The increase in nutrient concentration can stimulate the growth of algae, creating conditions that interfere with the health and diversity of indigenous fish, plant and animal populations and also with the recreational use of lakes. Growth of algae makes harvesting essential, but the main problem becomes the final use of algae.

The paper investigates the anaerobic digestion of the algae harvested from Trasimeno Lake, a mesotrophic lake near eutrophication in Umbria, a Region in Central Italy. Three different species of algae were harvested in two different points of the lake, nearby Polvese island (Figure 1), in Dicember 2004. Species 1 and 2 were collected in Point 1, while Specie 3 in Point 2. Sampling sites and their coordinates are shown in Fig. 1.

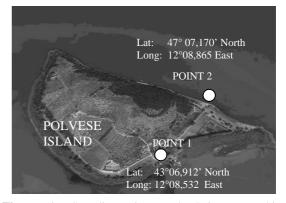


Figure 1: Sampling sites and their geographic coordinates

Four kilograms of each sample were collected and their chemical and physical characteristics were analysed at the CRB laboratory [1]. In particular, Total Solids (TS) were determined, drying the samples at 105°C for 12 hours, in compliance with UNI 10458 [2]. Then the samples were ground and sieved, to measure the ash content in a Thermogravimetric Analyzer (TGA-701 LECO) at 600°C. Volatile components were determined as difference between TS and ash. The Higher Heating Value (HHV) was meausered with Isoperibolic Calorimeter (AC-350 LECO), according to UNI 9017 [3], and the Nitrogen, Hydrogen and Carbon content was determined with Elementary Analyzer (Truspec CHN LECO), according to ASTM D-5373 [4]. Table I lists the characteristc of the three different species of algae.

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Table I:	Composition	of the three	species of algae	

Parameter	Unit	Specie 1	Specie 2	Specie 3
Total Solids	%	90.00	91.00	95.00
Volatile	% <sub>db</sub>	86.25	72.26	47.97
Solids	/ • 00	00.20	/ =:=0	
Ash	% <sub>db</sub>	13.75	27.73	52.09
HHV	MJ/kg <sub>db</sub>	15.10	13.60	8.900
С	% <sub>db</sub>	37.50	33.00	27.20
Н	% <sub>db</sub>	5.720	4.830	3.320
Ν	% <sub>db</sub>	2.050	1.910	1.310
C/N	-	18.29	17.28	20.76

Eventually the digestion of algae was carried out in four anaerobic glass vessel reactors, with a working capacity of 1,5 l, shut by a PVC lid sealed with silicone (Figure 2): three of them were used, one for each species of algae, and one for a mixture of the three types; Table II shows the quantity of algae introduced in each reactor.

Each reactor was linked to a 3,0 l sample bag, to collect the produced biogas.



Figure 2: Anaerobic glass vessel reactors in climatic chamber

 Table II: Quantity of algae introduced in the reactors

	Introduced quantity (g)		
Digestor 1	Specie 1	285.080	
Digestor 2	Specie 2	441.230	
Digestor 3	Specie 3	1189.48	
	Specie 1	83.770	
Disaster 4	Specie 2	247.070	
Digestor 4	Specie 3	359.020	
	Total Dig. 4	689.860	

## **3 FIRST EXPERIMENTAL RESULTS**

The digesters were put in climatic chamber, a Mazzali C330G55, at constant temperature of 35°C for thirty days. The sample of produced biogas was collected with a syringe. Biogas composition was measured by a gas chromatograph DANI GC 1000 DPC, fitted with a thermal conductivity detector. The composition of biogas was monitored weekly, during the total period of investigation.

Analytical results show a good methanisation capacity of Trasimeno Lake algae, with significant variation depending on feedstock, as in Figure 3. Specie 1 shows the lowest quantity of methane, but an air leak problem was experienced on the digester. This accounts for the 6% vol. of methane at the end of the process. Digestor  $n^{\circ}$  2 and 4 show an increasing methane production reaching 50% vol. at the end of process.

The biogas produced by Digestor 3 shows the highest methane concentration (60% vol.), which is consistent with the highest value of C/N ratio of its feedstock, in agreement with the Literature [5].

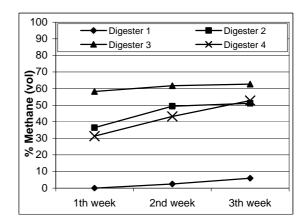


Figure 3: Methane percentage in volume produced by the four digesters

The preliminary activity on algae pointed out some critical aspects of the test facility, such as: the impossibility to measure the quantity of the produced biogas and the heat provided to the system, which are essential quantities to carry out a mass and energy balance. Moreover no temperature or pH measures of the feedstock could be taken, which are also important variables that affect biogas production. These considerations were taken as design specification of a 2nd generation anaerobic digester, described in the following section.

#### **4 LABORATORY DIGESTER**

A pilot batch digester was designed and built as a cylindrical vessel equipped with an airtight lid. The steel AISI 304 vessel has a working volume of 17 l, with 30 cm inner diameter and a height/width ratio of 5/6 (Figure 4).

The cylindrical shape was chosen because of its simplicity from the construction point of view and because it requires less material with respect to other shapes. Moreover, it guarantees a better homogeneity of the biogas flow in the reactor. Steel AISI 304 was used as building material for its strength and durability in acid or basic environments.

Flexible silicon rubber heaters, with a maximum operating temperature of 260 °C and an electrical power density of 12.5 W/cm<sup>2</sup> (depending on temperature), were fitted on the external surface of the vessel to provide the heat necessary to maintain the feedstock at the required temperature.

The heating system was equipped with a AF thermocouple, connected to a PID temperature controller, introduced in the digester through a hole on the lid, closed by threaded steel adapter (Figure 5 a).



Figure 4: Pilot batch digester

Five holes were drilled on the lid of the digester; four side holes are used to insert probes to measure temperature and pH through threaded steel adapters and rubber stoppers to avoid gas leakage. Through the central hole the shaft of a stirring system RW 16 Basic IKA with a speed range of 40-1200 rpm was introduced to allow the mixing of the feedstock and therefore increasing biogas production, as in existing plants (Figure 5 b).

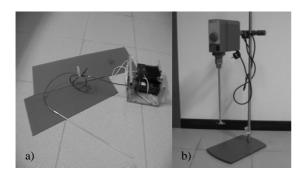


Figure 5: Silicon heaters (a) and stirring system (b)

Inside temperature is measured by  $n^{\circ}$  3 PT 100 probes namely: one with the pHmeter, one feedbacks the heating system and a third one for redundancy. This allows a consistent confidence on temperature and also verifies its uniformity in the reactor.

Finally the digester was equipped with a tap placed at the side to collect the biogas produced.



Figure 6: Batch digester equipped with probes, heating and stirring systems.

# 5 PRELIMINARY ACTIVITY ON ORGANIC WASTE

Organic Fraction of Municipal Solid Waste (OFMSW) was chosen as a substrate for digester calibration. This waste was fresh collected from a nearby dump and analyzed at the CRB laboratory, as previously described for algae. Average values of the chemical and physical parameters are shown in Table III.

Table III: Composition of the OFMSV	Table	III:	Com	position	of the	OFMSV
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Parameter	Unit	Value
M	%	49.250
VS	% <sub>db</sub>	82.060
Ash	% <sub>db</sub>	17.930
HHV	MJ/kg <sub>db</sub>	12553
С	% <sub>db</sub>	31.900
Н	% <sub>db</sub>	5.9000
Ν	% <sub>db</sub>	1.2200
O (difference)	% <sub>db</sub>	43.050
C/N	-	26.150

A sample of 4.615 kg of substrate was added to the reactor at a pH between 5,5 and 5,7. The anaerobic batch digestion was carried out at  $37^{\circ}$ C for 8 days. The process was monitored during the first acidogenic phase: the values of temperature, pH and the biogas production were determined. The temperature was maintained at 37-38 °C, but in the two last days of the process it was increased at 40 °C, to improve digestion.

Biogas production was measured each hour, through the elevation of a Plexiglas cylinder inside a water filled vessel, as in industrial gas-meter. Biogas production ranged from 0,043 to 2.64 l, during the eight days.

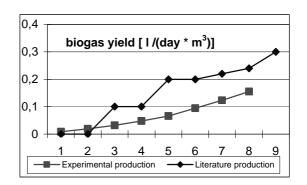


Figure 7: Comparison between experimental biogas production and the Literature values.

When comparing experimental values with Literature results obtained for OFMSW in a 10 m<sup>3</sup> digester [4] (values normalized to digester volume), trends are similar, but experimental values are much lower ( $\sim$ 50% less).

The fact that no substrate pre-treatment or inoculum were considered may account for the discrepancy together with a scale effect due to reactor dimensions.

Further experimentation is however required.

### 6 GASMETER DESIGN AND ERROR EVALUATION

In order to measure biogas volume with more precision, a dedicated gas-meter was designed, made of

two cylindrical coaxial chambers which communicate as shown in Figure 8. The inner chamber is open in the upper area, so that liquid is at atmospheric pressure. The external vessel is closed and linked to the digester through a plastic pipe to collect the biogas produced.

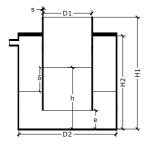


Figure 8: Schematic diagram of gas-meter

At the beginning of the process, the surface of the water is equal in both chambers. When biogas is produced, it is collected in the external chamber of the gas-meter, increasing the pressure and causing the rise of the liquid level (d) in the internal vessel.

The height variation of the liquid in the inner cylinder may be measured with a magneto-strictive level sensor and it is proportional to the volume of biogas produced. The error on biogas volume is therefore a function of the error of the level measuring sensor, but also of the  $D_1/D_2$ ratio of the gas-meter and of the density of the liquid. A home made software was developed to evaluate the measure error on biogas volume as a function of the above mentioned quantities and of time.

The biogas production vs time curve was obtained from Literature data [7] and may be expressed in volume as follows with time in seconds.

$$V_{ex} = 2.69122 \cdot t^5 - 0.001951 \cdot t^4 + 0.037722 \cdot t^3 \tag{1}$$

Biogas was considered a perfect gas therefore for every increase in volume a pressure increase can be measured through the height of the column, also considering the error introduced by different level sensors.

Different simulation were run for different  $D_1/D_2$  ratios of the gas-meter, different sealing liquids and different sensors: error obviously decreases abruptly as time increases. Best performing architecture, obtained varying the dimensions indicated in Figure 8 shows an error below 2% after 1,5 days (Figure 9). It also guarantees a compact layout, and considers water as a sealing liquid and a magneto-strictive level measuring device with an error of 0,025 %.

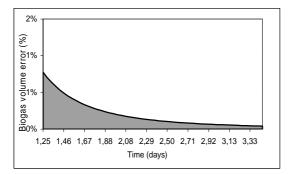


Figure 9: Percentage biogas volume error vs time

## 7 CONCLUSIONS

The ongoing activity in anaerobic digestion at the Italian Biomass Research Centre was described in the present paper. Preliminary activity on lake algae with sealed glass vessels digesters in a climatic chamber showed encouraging results in terms of methane production. Eventually an electrically heated stainless steel digester was designed and built, with temperature control and pH measure. A stirring system was also added to reply real working conditions. Testing activity on organic waste showed the necessity of a reliable biogas metering system. A gas-meter to be connected to the digester was then designed, to guarantee an error on biogas volume measure below 2% after 1,5 days of digestion.

Future activity on the pilot digester will determine mass and energy balance and biogas composition for different feedstocks, in order to build a reliable database.

#### 8 ACKNOWLEDGMENTS

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## 10 NOMENCLATURE

- %TS percentage of Total Solids;
- VS Volatile Solids;
- HHV High Heating Value;
- M moisture;
- db dry basis;
- OFMSW organic fraction of municipal solid waste;
- $V_{ex}$  experimental volume (m<sup>3</sup>);
- t time (s).