Biogas Production by Anaerobic Digestion of Agricultural Biomass: Factorial Design Analysis

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Abstract. In present times, replacing fossil fuels with renewable ones becomes a problem more and more stringent in the existing society. Related with this topic, biogas production, as one of the potential renewable fuels has an increasing role both at regional and global level in relation to the potential it poses for energetic independence, both for developed and in course of development countries. The present article intends to underline the potential usage of agricultural biomass (namely wheat bran and cereal mix) as renewable source of energy for biogas production. Factorial design of experiments was employed to study the effect of two factors (net calorific value and C/N ratio) on total biogas produced after 65 days of anaerobic digestion. Main effects and interaction effects of these factors were analyzed using statistical techniques. A regression model was obtained to predict the total biogas production and it was found to adequately fit the experimental range studied.

Introduction

Biomass fuels have been used throughout man's long history. Today, with rising prices for crude oil and the existing political instability in oil producing countries, the use of bio-based alcohols is again taken in consideration. This aspect has as main advantages the political independence through diversification and also CO₂ neutral energy production both at local and regional levels [1].

According to literature, anaerobic digestion is the process of decomposition of organic matter by a microbial consortium in an oxygen-free environment [2].

Some of the advantages this process is presented below:

- less biomass sludge is produced in comparison to aerobic treatment technologies;
- successful in treating wet wastes of less than 40% dry matter [3];
- more effective pathogen removal [4];
- minimal odour emissions as 99% of volatile compounds are oxidative decomposed upon combustion, e.g. H₂S forms SO₂ [5];
- the produced residual material is an improved fertilizer in terms of its availability to plants [6];
- in this way it can be produced energy characterized by the presence of neutral carbon.

The elements present inside biogas are: 48–65% methane, ca. 36–41% carbon dioxide, about 1% nitrogen, under 1% oxygen, 32–169 ppm hydrogen sulphide, and traces of other gases [7].

The three main temperature regimes used during biogas production are: cryophilic, mesophilic and thermophilic, each regime having a corresponding temperature domain.

Most fermentation processes work at mesophilic temperatures (25–37°C), but it is known that thermophilic processes (45–55°C) are more productive and for this, the researches are made in this direction for further development. One major difference between the two temperature regimes consists in the fact that for thermophilic processes the microorganisms are more sensitive to the toxicity of the solvent products at high temperatures.

Also, some positive aspects in anaerobic fermentation using the thermophilic regime consists in a high degree of waste stabilization and also more thorough destruction of viral and bacterial pathogens [8].

In spite of these benefits, however, poor operational stability still prevents anaerobic digestion from being widely commercialized [9].

An overall scheme for anaerobic conversion of organic substrates to methane is indicated in figure 1.

Fig. 1 Anaerobe conversion of biomass to methane [10]

Biogas formation is usually a process which consists in the involvement of different types of anaerobic organisms during the stages needed to complete it (hydrolysis, acidogenesis, acetogenesis and methanogenesis).

The bacterial community engaged in these three stages may be similar to those in cows rumen [11] or wastewater treatment plants [12]. However, their composition varies depending on the substrate, the type of fermenter and the process (e.g. mesophilic or thermophilic [13, 14]).

Related with the presented topic, this study will underline the possibilities of using cereal degraded materials inside anaerobic fermentation processes at mesophilic temperature regime, related with the influence of some material parameters (namely net calorific value and C/N ratio) on the total biogas production during a period of 65 days.

Materials and methods

Description of pilot plant. The pilot plant used for producing biogas from biomass through anaerobic digestion is presented in figure 2.

Fig. 2 Pilot installation general schematics

From the biomass deposit, the used material is passed through a mill, and then it's sent to the tank where the preparation of the suspension of biomass is made (1). The biomass suspension is transported with the help of the pump (2) and introduced into the fermentation reactors (3). The correction agent for the pH assures, through the control system, the conditions for the process of anaerobic fermentation. The resulted biogas is passed through a filter for partially retaining the H_2S (5) and after that, through a system used for partially retaining CO_2 (6), after which takes place the CO_2 desorption and the compression of the CO_2 in the adjacent system and the purified biogas is sent for being used (8). The used material is discharged through the means of a gravimetric system (9), and the solid material is retained for being dried using the natural drying, and after that is sent to a compost deposit for being used as a soil fertilizer. A part of the resulting liquid is neutralized when the case, in the system (10) and sent to the sewerage network, or is transported by the recirculation pump (2) from the suspension preparation tank (1). The fermentation reactors are thermostat heated with the system (11). For the homogenization of the suspension is used a bubbling system (12) made by polypropylene pipes to avoid the possible corrosion. Also, for depositing small quantities of biogas of the purpose of analyzing, the installation is equipped with a small tank (13) positioned at the top of the reservoirs.

The reactors were fed at the beginning of the experiment with approximately 75 kg dry biomass and 2000 l water. Biogas production was measured daily, the pressure difference being dropped with the help of a semi-automated system and afterwards through a gas counter. Methane (CH_4) and carbon dioxide (CO_2) compositions (v/v) were measured using a Delta 1600 IV gas analyzer. Temperature and pH were also continuously measured.

Factorial design. A factorial design of experiments was applied to find out the influence of biomass properties, such as net calorific value and C/N ratio, on the total biogas produced after 65 days of anaerobic digestion (Q). The effect of selected factors was studied by means of a 2^2 full factorial design (two factors each at two levels). The high and low levels of the factors were selected according to some preliminary experiments and their values are listed in table 1.

Table 1 Factors and levels used in the factorial design

Factors	Low level (-1)	High level (+1)
(A) Net calorific value (db) [J/g]	16820	17520
(B) C/N	20	35

The factorial design matrix and the results of Q measured for each experiment are shown in table 2. The values of Q listed in the table represent the average of two parallel experiments.

Table 2 Design matrix of the 2^2 full factorial design and the results of dependent variable

Experiment	A	B	Q [m^3]
1	-1	-1	8.0
2	1	-1	18.0
3	-1	1	10.0
4	1	1	25.0
5	-1	-1	9.0
6	1	-1	17.0
7	-1	1	10.5
8	1	1	26.0

The results were studied and interpreted with MINITAB 16 software. The mean of the experimental results for the low and high levels of studied factors are shown in figure 3.

Fig. 3 Cube plots for Q

Results and discussion

The influence of biomass properties, such as net calorific value (A) and C/N ratio (B), on the total biogas produced after 65 days of anaerobic digestion (Q) was evaluated by means of factorial plots: main and interaction effects, Pareto chart, normal probability plots and surface plot. A regression first-order polynomial model with interaction terms was developed to describe the relationship between selected factors and the response (Q). The effect of those factors on the response was performed by analysis of variance (ANOVA).

The general form of mathematical model used is given in eq. 1:

$$Q = \alpha_0 + \alpha_1 * A + \alpha_2 * B + \alpha_3 * A * B. \quad (1)$$

where: Q is the response, A and B are the independent variables, α_0 is the intercept of the model, α_1 and α_2 are the linear coefficients in the regression models and α_3 is the interaction coefficient between factors.

The effects, regression coefficients, standard errors, t and P are shown in table 3. The significance of the regression coefficients was determined by applying a student's t-test. The P values were used as a tool to check the significance of each of the interaction among the variables. The term is more significant as the value of t is higher and the value of P is smaller [15]. According to this rule, all effects were significant with 95% confidence level.

Table 3 Estimated effects and coefficients for Q [m³]

Term	Effect	Coef	SE Coef	t	P
Constant		15.438	0.2253	68.51	0.000
A	12.125	6.062	0.2253	26.90	0.000
B	4.875	2.437	0.2253	10.82	0.000
A*B	3.125	1.563	0.2253	6.93	0.002

$$S = 0.6374 \quad R^2 = 99.55\% \quad R^2_{(adj)} = 99.22\%$$

The main effects represent deviations of the average between high and low levels of each factor and are presented in figure 4 [16, 17].

Fig. 4 Main effects plot for Q

Substituting the regression coefficients from table 3 in equation (1), it was obtained the following model equation:

$$Q = 15.438 + 6.062 \cdot A + 2.437 \cdot B + 1.563 \cdot A \cdot B \quad (2)$$

The net calorific value (A) had the greatest effect on Q, followed by C/N ratio (B) and net calorific value - C/N interaction (AB). The positive values of the effect bring out that the increase of parameter increase the response Q. Contrary, negative values reveal that the increase of parameter decrease the response Q. According to equation (2), the net calorific value (A), C/N ratio (B) and the interaction between (AB) had a positive effect on Q.

The value of adjusted square correlation coefficient $R^2_{(adj)}$ of 99.22%, certify that the model in equation (2) fitted the data adequately.

The surface plot of total biogas produced after 65 days of anaerobic digestion (Q) as a function of net calorific value (A) and C/N ratio (B) are reported in figure 5.

Fig. 5 Surface plot of Q

ANOVA results, presented in table 4, showed that net calorific value and C/N ratio of biomass influence biogas production. Furthermore, two-factor interactions exhibited a significant effect on the response Q. This indicates that the two factors do not work independently, the effect of each factor being affected by the level of other factor.

Table 4 Analysis of variance for Q [m³]

Source	Degrees of freedom	Seq SS	Sum of squares adj (SS)	Mean square adj (MS)	F	P
A	1	294.03	294.03	294.03	723.77	0.000
B	1	47.53	47.53	47.53	117.00	0.000
A*B	1	19.53	19.53	19.53	48.08	0.002
Error	4	1.63	1.63	1.63		
Total	7	362.72				

Interaction plot of effects is shown in figure 6. An interaction is effective when the change in the response from low to high levels of a factor is dependent on the level of second factor. On the interaction figure, the two lines that represent the variation of factors are not parallel [17].

Fig. 6 Interaction effect plot for Q

The relative importance of the main effects and their interactions was also observed on the Pareto chart, presented on figure 7. For the 95% confidence level and four degrees of freedom, the t-value is 2.78 (marked on the figure by a vertical line). According to figure 4, all factors and their interactions were statistically significant at 95% confidence. The net calorific value (A) represented the most significant effect on Q.

Fig. 7 Pareto chart of the standardized effects

In order to determine the significance of the effects results, the normal probability plot of standardized effects was used (figure 8). If the effects are not significant, the variations are due to random error and this can be tested with the plots. According to this theory, the points which are close to a line fitted to the middle group of points represent those estimated factors that do not demonstrate any significant effect on the response variable [18, 17].

Fig. 8 Normal probability plot of standardized effects

The factors (A and B) and their interaction are far away from the straight line, which means that their effects are significant on the response Q. Because A, B and AB lie to the right of the line, their contribution had a positive effect. The effects decreased as follow: A > B > AB. These results confirm the previous Pareto chart and the values from table 3.

Conclusions

In the present study, the anaerobic digestion of some agricultural biomass was investigated. The effects of biomass net calorific value (A) and C/N ratio (B) were studied using the factorial design and the results showed that these parameters has a significant influence on the total biogas produced after 65 days of anaerobic digestion (Q). A regression first-order polynomial model has been generated to describe the relationship between selected factors and the response (Q). The value of correlation coefficient (R^2) was 0.99, which means the model adequately describe the observed data. The statistical analysis proved that the two factors have a positive influence on the response (Q), within the experimental range studied. The biomass net calorific value (A) is the most important factor.

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References

- [1] D. Antoni, V.V. Zverlov, W.H. Schwarz, Biofuels from microbes, Appl. Microbiol. Biotechnol. 77 (2007) 23 – 35.
- [2] B.F. Pain, R.Q. Hephherd, Anaerobic digestion of livestock wastes, in: B.F. Pain, R.Q. Hephherd (Eds.), Anaerobic Digestion of Farm Waste, NIRD Technical Bulletins Reading, 1985, pp. 9–14.
- [3] J. Mata-Alvarez, Biomethanization of the Organic Fraction of Municipal Solid Wastes, IWA Publishing, London, 2002.

- [4] L. Sahlstrom, A review of survival of pathogenic bacteria in organic waste used in biogas plants, *Bioresour. Technol.* 87 (2003) 161–166.
- [5] E. Smet, H. Van Langenhove, I. De Bo, The emission of volatile compounds during the aerobic and the combined anaerobic/aerobic composting of biowaste, *Atmos. Environ.* 33 (1999) 1295–1303.
- [6] S. Tafdrup, Viable energy production and waste recycling from anaerobic digestion of manure and other biomass materials, *Biomass Bioenerg.* 9 (1995) 303 – 314.
- [7] S. Rasi, A. Veijanen, J. Rintala, Trace compounds of biogas from different biogas production plants. *Energy* 32 (2007)1375–1380.
- [8] K.V. Lo, P.H. Liao, A.C. March, Thermophilic anaerobic digestion of screened dairy manure. *Biomass* 6 (1985) 301–315.
- [9] M. Dupla, T. Conte, J.C. Bouvieu, N. Bernet, J.P. Steyer, Dynamic evaluation of a fixed bed anaerobic-digestion process in response to organic overloads and toxicant shock loads, *Water Sci. Technol.* 49(1) (2004) 61–68.
- [10] B. Demirel, P. Scherer, The roles of acetotrophic and hydrogenotrophic methanogens during anaerobic conversion of biomass to methane: a review, *Rev. Env. Sci. Biotechnol.* 7 (2008) 173–190.
- [11] R. Einspanier, B. Lutz, S. Rief, O. Berezina, V. Zverlov, W.H. Schwarz, J. Mayer, Tracing residual recombinant feed molecules during digestion and rumen bacterial diversity in cattle fed transgene maize, *Eur. Food Res. Technol.* 218 (2004) 269–273.
- [12] H.D. Ariesyady, T. Ito, S. Okabe, Functional bacterial and archaeal community structures of major trophic groups in a fullscale anaerobic sludge digester, *Water Res.* 41 (2007)1554–1568.
- [13] C.A. O’Sullivan, P.C. Burrell, W.P. Clarke, L.L. Blackall, Structure of a cellulose degrading bacterial community during anaerobic digestion, *Biotechnol. Bioeng.* 92 (2005) 871–878.
- [14] K. Syutsubo, Y. Nagaya, S. Sakai, A. Miya, Behavior of cellulose degrading bacteria in thermophilic anaerobic digestion process, *Water Sci. Technol.* 52 (2005) 79–84.
- [15] R. Krishna Prasad, S.N. Srivastava, Electrochemical degradation of distillery spent wash using catalytic anode: Factorial design of experiments, *Chem. Eng. J.* 146 (2009) 22–29.
- [16] V. Ponnusami, V. Krithika, R. Madhuran, S.N. Srivastava, Biosorption of reactive dye using acid-treated rice husk: Factorial design analysis, *J. Hazard. Mat.* 142 (2007) 397–403.
- [17] D. Bingol, N. Tekina, M. Alkan, Brilliant Yellow dye adsorption onto sepiolite using a full factorial design, *Appl. Clay Sci.* 50 (2010) 315–321.
- [18] K. Palanikumar, J.P. Dawim, Assessment of some factors influencing tool wear on the machining of glass fibre-reinforced plastics by coated cemented carbide tools, *J. Mater. Process Technol.* 209 (2009) 511–519.