



Stoichiometric Analysis of Biogas Production from Industrial Residues

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Abstract

A stoichiometric analysis of biogas production by anaerobic digestion from cassava wastewater, wheat bran, and sewage sludge is proposed. A wide range of methods are available to study stoichiometry of biochemical reactions. This work reported elemental balances method to solve stoichiometric coefficients in biogas production from cassava wastewater, wheat bran, and sewage sludge. The method could be employed for various substrates for biogas production and for other biochemical reactions.

Keywords

Anaerobic digestion · Biogas · Industrial residues · Stoichiometry

6.1 Introduction

Anaerobic digestion is the process of producing biogas from the organic materials such as animal manure, waste paper, and sewage in the absence of oxygen. In India, more than three million small-scale biogas plants are available. In addition, in the Europe Union, biomass could amount to 1545 million tons per year (Igoni et al. 2008). Incorporation of municipal organic wastes such as food to the anaerobic digestion production helps to raise the amount of produced energy. Also, paper waste can be an additional source of enhanced biogas production. Anaerobic digestion is a multistep biological and chemical process that is beneficial in not only waste management but also energy creation (Weiland 2010).

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N. Srivastava et al. (eds.), *Biofuel Production Technologies: Critical Analysis for Sustainability*, Clean Energy Production Technologies,
https://doi.org/10.1007/978-981-13-8637-4_6

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Fig. 6.1 Biogas production process

Table 6.1 Biochemistry of biogas production (Bajpai 2017)

Step	Reaction
Hydrolysis	Carbohydrates + H ₂ O → Glucose Lipids + H ₂ O → Fatty acids Proteins + H ₂ O → Amino acids
Acidogenesis	C ₆ H ₁₂ O ₆ + 2 H ₂ → 2 C ₂ H ₅ COOH + 2 H ₂ O C ₆ H ₁₂ O ₆ → 2 C ₂ H ₅ OH + 2 CO ₂
Acetogenesis	C ₂ H ₅ COOH + 2 H ₂ O → CH ₃ COOH + CO ₂ + 3 H ₂ C ₆ H ₁₂ O ₆ + 2 H ₂ O → 2 CH ₃ COOH + 2 CO ₂ + 4 H ₂ C ₂ H ₅ OH + H ₂ O → CH ₃ COOH + 2 H ₂ C ₆ H ₁₂ O ₆ → 3 CH ₃ COOH
Methanogenesis	2 C ₂ H ₅ OH + CO ₂ → 2 CH ₃ COOH + CH ₄ CH ₃ COOH → CH ₄ + CO ₂ CO ₂ + 4 H ₂ → CH ₄ + 2 H ₂ O CH ₃ COOH + H ₂ SO ₄ → 2 H ₂ O + 2 CO ₂ + H ₂ S 7 CH ₃ COOH + 8 NO ₂ → 2 H ₂ O + 14 CO ₂ + 8 NH ₃
Overall	C _a H _b O _c N _d S _e + f H ₂ O → p CH ₄ + q CO ₂ + r NH ₃ + s H ₂ S

6.1.1 Biogas Production Process

The four fundamental steps of anaerobic digestion include hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Fig. 6.1). Throughout the entire process, large organic polymers that make up biomass are broken down into smaller molecules by chemicals and microorganisms. Upon completion of the anaerobic digestion process, the biomass is converted into biogas consisting of carbon dioxide and methane, and digestate (Dioha et al. 2013).

Table 6.1 shows the biochemical reactions taking place in each step during biogas production, and the overall reaction is shown below:

6.1.1.1 Hydrolysis

In general, hydrolysis is a chemical reaction in which the breakdown of water occurs to form H⁺ cations and OH⁻ anions (Sivamani et al. 2018). Hydrolysis is often used to break down larger polymers, often in the presence of an acidic catalyst. In anaerobic digestion, hydrolysis is the essential first step, as biomass is normally comprised of very large organic polymers. Through hydrolysis, these large polymers, namely, carbohydrates, Lipids, and proteins, are broken down into smaller molecules such as simple sugars, fatty acids, and amino acids, respectively (Sivamani et al. 2018).

6.1.1.2 Acidogenesis

Acidogenesis is the next step of anaerobic digestion in which acidogenic microorganisms further break down the monomer products after hydrolysis. These fermentative bacteria produce an acidic environment in the digestive tank while creating volatile fatty acids, carbonic acids, alcohols, as well as trace amounts of other by-products. While acidogenic bacteria further break down the organic matter, it is still too large and unusable for the ultimate goal of methane production, so the biomass must next undergo the process of acetogenesis (Parawira et al. 2004).

6.1.1.3 Acetogenesis

In general, acetogenesis is the creation of acetic acid or acetate, from carbon and energy sources by acetogens. These microorganisms catabolize many of the products created in acidogenesis into acetic acid, CO₂, and H₂. Acetogens break down the volatile fatty acids and alcohols to a point at which methanogens can utilize much of the remaining materials to create methane as a biofuel (Kalia 2007).

6.1.1.4 Methanogenesis

Methanogenesis constitutes the final stage of anaerobic digestion in which methanogens create methane from the final products of acetogenesis as well as from some of the intermediate products from hydrolysis and acidogenesis. While CO₂ can be converted into methane and water through the reaction, the main mechanism to create methane in methanogenesis is the path involving acetic acid. This path creates methane and CO₂, the two main products of anaerobic digestion (Ziemiński and Fraç 2012; An et al. 2008).

6.1.2 Factors Affecting Biogas Yield

The factors affecting the biogas yield are carbon/nitrogen ratio, temperature, pH, dilution and consistency of feed, loading rate, hydraulic retention time, toxicity, agitation, and additives (Mahanta et al. 2005).

6.1.2.1 Carbon/Nitrogen (C/N) Ratio

The relationship between the amount of carbon and nitrogen present in organic materials is expressed by the carbon/nitrogen (C/N) ratio. A suitable C/N ratio plays an important role for the proper proliferation of the bacteria for the degradation process (Augenstein et al. 1976). Depending upon the relative richness in carbon and nitrogen content, feed material can be classified as nitrogen-rich or carbon-rich. It is generally found that during digestion, microorganisms utilize carbon 25–30 times faster than nitrogen, that is, carbon content in feedstock should be 25–30 times of the nitrogen content (Report No. ETSU B 1118 1986; Barnett et al. 1978; Fry and Merrill 1973). To meet this requirement, constituents of feedstock are chosen in such a way to ensure a C/N ratio of 25:1 to 30:1 and concentration of dry matter as

7–10%. Even in situations where C/N ratio is close to 30:1, the biomass can undergo efficient anaerobic digestion only if waste materials are also biodegradable at the same time (Singh 1974; SPOBD 1979).

6.1.2.2 Temperature

Most digesters installed in the field lack mechanisms for temperature control and removal of dissolved oxygen. Hence, efficiency of these digesters is reported to be low, particularly during the winter months. There are different temperature ranges during which mesophilic and thermophilic bacteria are most active causing maximum gas yield. Generally, mesophilic bacteria are most active in the temperature range 35–40 °C and thermophilic bacteria in the range 50–60 °C. Choice between the mesophilic and thermophilic digestions is governed by the natural climatic conditions in which the plant is located. Though, it is possible to create conditions for thermophilic digestion by external heat, but such a method is generally uneconomical. Length of digestion period is linked with the digester temperature.

The methanogens are inactive in extreme high and low temperatures, while the optimum temperature is 35 °C. When the ambient temperature decreases to 10 °C, gas production virtually stops. Satisfactory gas production takes place in the mesophilic range (30–40 °C). Proper insulation of digester helps to increase gas production during the cold weather. When the digester operates at a temperature of 15 °C, it takes nearly a year for the digestion cycle to complete. However, if the temperature is approximately 35 °C, the cycle can be easily completed in less than a month. When the digester temperature is maintained at 25 °C, it takes approximately 50 days for digestion of cattle waste. But, if the temperature ranges between 32 and 38 °C, digestion is complete within 28 days. Mahanta et al. carried out experiments to analyze the effect of temperature variation on anaerobic digestion of cattle wastes (NAS 1977). Smith et al. suggested that at low temperature, biogas plants with some design modifications could also function quite effectively as in a warm climate (Mahanta et al. 2004a).

6.1.2.3 pH

During anaerobic digestion, microorganisms require a natural or mildly alkaline environment for efficient gas production (Smith et al. 1982). An optimum biogas production is achieved when the pH of feed mixture in the digester is between 6.25 and 7.50 (Mahanta et al. 2004b; Wise 1987). The pH in a biogas digester is also a function of retention time. In the initial period of digestion, as large amounts of organic acids are produced by acidogenic bacteria, the pH inside the digester can decrease below 5. This inhibits or even stops the process. Methanogenic bacteria are very sensitive to pH and do not thrive below 6.5. Later, as the digestion process continues, concentration of NH₃ increases due to the digestion of N₂, which can increase the pH to above 8. When the CH₄ production level is stabilized, the pH range remains between 7.2 and 8.2. According to studies in China, during the period when ambient temperature varies between 22 and 26 °C, it takes approximately 6 days for pH to acquire a stable value (SPOBD 1979). Similarly, during the period

when ambient temperature ranges between 18 and 20 °C, it takes approximately 14–18 days for pH to attain a stable value.

6.1.2.4 Dilution and Consistency of Feed

All waste materials fed to a biogas plant consist of solid substance, volatile organic matter and nonvolatile matter, and water. During anaerobic digestion process, volatile solids undergo digestion and nonvolatile solids remain unaffected. According to a finding by The Energy and Resources Institute (TERI), fresh cattle waste consists of approximately 20% total solids (TS) and 80% water (TERI 1987). TS, in turn, consists of 70% volatile solids and 30% fixed solids. For optimum gas yield through anaerobic digestion, normally, 8–10% TS in feed is required. This is achieved by making slurry of fresh cattle dung in water in the ratio of 1:1. However, if the dung is in dry form, the quantity of water has to be increased accordingly to arrive at the desired consistency of the feed (i.e., ratio could vary from 1:1.25 to even 1:2). If the dung is too diluted, the solid particles will settle down into the digester and if it is too thick, the particles impede the flow of the gas formed at the lower part of the digester. In both cases, gas production will be less than optimum. It is also necessary to remove inert materials such as stones from the feed before sending the slurry into the digester. Otherwise, the effective volume of digester will decrease.

6.1.2.5 Loading Rate

Loading rate is defined as the amount of raw materials fed per day per unit volume of digester. It is an important parameter that affects gas yield. If the plant is overfed, acids will accumulate and methane production will be inhibited since bacteria cannot survive in acidic conditions. Similarly, if the plant is underfed, the gas production will also be low because of alkaline solution, which is also not a favorable condition for anaerobic bacteria. The effect of daily and alternate day loadings on biogas yield was also studied (Gore 1981). It was found that a 50 kg charge on daily basis and 100 kg charge on alternate day basis produced 2.9043 and 2.9285 m³ of gas, respectively. Also, for a particular size of plant, there is an optimum feed of charge rate that will produce maximum gas and further quantity of charge will not proportionately produce more gas. A daily loading rate of 16 kg of volatile solids per m³ of digester produces 0.04–0.074 m³ of gas per kg of raw dung fed (Moharao 1975). The recommended loading rates for plants working on night soil range from 1.04 to 2.23 kg of volatile solids per m³ of digester (Moharao 1974). Higher loading rates are recommended only in cases where mean ambient temperature is high.

6.1.2.6 Hydraulic Retention Time

Hydraulic retention time (HRT) is the average period that a given quantity of input material remains in the digester to be acted upon by the methanogens. In a cattle dung plant, the retention time is calculated by dividing total volume of the digester and volume of feed per day. The rate of gas generation is initially high and then, gradually, declines as the digestion approaches completion. Thus, the time required for 70–80% digestion is considerably less than that needed to achieve complete digestion. HRT is chosen to achieve at least 70–80% digestion. Langrage (Langrage

1979) suggested that HRT depends upon the interior temperature of the digester. Higher the temperature of the digester, lower is the retention time. HRT varies between 20 and 120 days, depending upon the design and operating temperature of the digester. For digesters operating in countries of tropical region, HRT is usually taken as 40–60 days. In countries of colder region, digesters are designed for HRT of about 100 days (UN Guidebook on Biogas Development 1980).

6.1.2.7 Toxicity

Mineral ions, heavy metals, and detergents are toxic materials that inhibit the normal growth of pathogens in the digester. Small quantity of minerals (e.g., sodium, potassium, calcium, magnesium, ammonium, and sulfur) also stimulates the growth of bacteria, while high concentration leads to toxic effects. For example, presence of ammonium from 50 to 200 mg/L stimulates the growth of anaerobic microbes, whereas its concentration above 1500 mg/L produces toxic compounds. Similarly, heavy metals, such as copper, nickel, chromium, zinc, lead etc., in small quantities, are essential for the growth of bacteria, but their higher concentration has toxic effects (Moharao 1975). Detergents, including soap, antibiotics, organic solvents, etc. also inhibit the activity of methane-producing bacteria, and hence, addition of these substances in the digester should be avoided.

6.1.2.8 Agitation

Agitation or mixing of digester contents significantly helps to ensure intimate contact between microorganisms, which leads to improved fermentation efficiency. Coppinger (Coppinger 1979) suggested that effect of varying degrees of mixing of digester contents improves biogas production. The major problem associated with the different designs of biogas plant is that a thick layer of scum formation appears at the top of the digester which blocks the gas from coming out of the upper free portion of the digester. Thus, no gas is available at the utility point. The effects of recirculation of gas to break the scum formation were investigated by Mahanta et al. They found that recirculation of gas improves the biogas yield. The recirculation of gas increases the biogas production by three times. The gas production with circulation is much more than that without recirculation at the same pH.

6.1.2.9 Additives

The additives seem to play an important role in biogas yield. Addition of 5% commercial charcoal to cattle dung slurry on dry-weight basis raised the yield by 17 and 35% in batch and semicontinuous processes, respectively. Madamwar and Mithal (Madamwar and Mithal 1986) performed two sets of experiments: one at controlled temperature of 38 °C and the other at ambient temperature of 15 °C to find the impact of adding pectin to cattle dung slurry as feed on biogas yield. Pectin not only enhances gas yield but also imparts process stability during the periods of fluctuating temperature. The impact of adding inert materials, such as vermiculite, charcoal, and lignite bovine excreta, as feed on biogas yield has also been reported (Geeta et al. 1986). These additives increased biogas yield by 15–30%. Pebbles,

glass marbles, and plastic mesh when suspended in digester slurry reportedly led to an increase in the gas yield by 10–20%.

Prasad (Prasad 1985) studied the effects of adding bagasse, Gulmohar leaves, wheat straw, groundnut shells, and leguminous plant leaves as additives to cattle dung on the biogas yield, gas composition, and extent of biodegradation. These additives were separately mixed with cattle dung in the ratio of 1 part (oven dry) to 10 parts of fresh dung containing 19.2% of TS on weight basis. Anaerobic fermentation was carried out under batch process in bottles in laboratory at ambient temperature between 30 and 32 °C for 9 weeks. The volume of biogas generated in 24 h was measured every day and gas composition analyzed periodically. It has been concluded that addition of additives is advantageous for obtaining a high gas yield.

6.2 Materials and Methods

6.2.1 Materials

Cassava was purchased from local supermarket in Salalah, Sultanate of Oman. Wheat bran was kindly provided by Salalah Mills Company SAOG, Raysut, Sultanate of Oman. Sewage sludge was kindly supplied by Salalah Sanitary Drainage Services Company SAOC, Raysut, Sultanate of Oman. Samples were used without further treatment.

6.2.2 Methods

6.2.2.1 Preparation of Cassava Wastewater

Fresh cassava was peeled and 3 kg of peeled cassava root was weighed. The roots were washed and cut into pieces. The cut roots were crushed with water in blender, and the final mixture was filtered. The filtrate was used as cassava wastewater for further analysis without any further dilution (Liu et al.).

6.2.2.2 Analytical Procedures

The total carbohydrate, lipid, and protein contents in the samples were estimated by anthrone (Hedge et al. 1962), Soxhlet extraction (Saim et al. 1997), and micro-Kjeldahl (Pellet and Young 1980) methods.

6.2.2.3 Stoichiometric Analysis

The procedure for stoichiometric analysis of biogas production is as follows:

1. The contents of carbohydrates, lipids, and proteins were estimated in cassava wastewater, wheat bran, and sewage sludge.
2. The percentages of carbon, hydrogen, oxygen, nitrogen, and sulfur present in carbohydrates, lipids, and proteins are known from the literature.

3. The concentrations of carbon, hydrogen, oxygen, nitrogen, and sulfur present in cassava wastewater, wheat bran, and sewage sludge were calculated.
4. The gram atoms of carbon, hydrogen, oxygen, nitrogen, and sulfur present in cassava wastewater, wheat bran, and sewage sludge were calculated.
5. Consider the overall reaction for biogas production,



Writing balances for each element, we get

$$\text{Carbon : } a = p + q \quad (6.E1)$$

$$\text{Hydrogen : } b + 2f = 4p + 3r + 2s \quad (6.E2)$$

$$\text{Oxygen : } c + f = 2q \quad (6.E3)$$

$$\text{Nitrogen : } d = r \quad (6.E4)$$

$$\text{Sulfur : } e = s \quad (6.E5)$$

Knowing the values of a, b, c, d, and e from step (4) above, the values of f, p, q, r, and s can be calculated solving five simultaneous Eqs. (6.E1, 6.E2, 6.E3, 6.E4 and 6.E5) (Tchobanoglous et al. 1991).

6.3 Results and Discussion (Tables 6.2, 6.3 and 6.4)

mg atom of carbon in cassava wastewater a_1	= 1139.37/12	= 94.95
mg atom of hydrogen in cassava wastewater b_1	= 88.25/1	= 88.25
mg atom of oxygen in cassava wastewater c_1	= 1380.83/16	= 86.30
mg atom of nitrogen in cassava wastewater d_1	= 16.29/14	= 1.16
mg atom of sulfur in cassava wastewater e_1	= 1.09/32	= 0.03
mg atom of carbon in wheat bran a_2	= 115.51/12	= 9.63
mg atom of hydrogen in wheat bran b_2	= 9.94/1	= 9.94
mg atom of oxygen in wheat bran c_2	= 94.09/16	= 5.88
mg atom of nitrogen in wheat bran d_2	= 8.66/14	= 0.62
mg atom of sulfur in wheat bran e_2	= 0.58/32	= 0.02
mg atom of carbon in sewage sludge a_3	= 141.44/12	= 11.79
mg atom of hydrogen in sewage sludge b_3	= 12.34/1	= 12.34
mg atom of oxygen in sewage sludge c_3	= 101.49/16	= 6.34
mg atom of nitrogen in sewage sludge d_3	= 12.13/14	= 0.87
mg atom of sulfur in sewage sludge e_3	= 0.81/32	= 0.03

For cassava wastewater, substituting the mg atoms in Eqs. (6.E1, 6.E2, 6.E3, 6.E4 and 6.E5),

Table 6.2 Biochemical composition of carbohydrates, lipids, and proteins in cassava wastewater, wheat bran, and sewage sludge

Constituents	Carbohydrates (ppm)	Lipids (ppm)	Proteins (ppm)
Cassava wastewater	2507.36	19.74	98.72
Wheat bran	146.28	30.0	52.50
Sewage sludge	148.00	46.7	73.50

Table 6.3 Percentage of carbon, hydrogen, oxygen, nitrogen, and sulfur in carbohydrates, lipids, and proteins

Elements	Carbohydrates	Lipids	Proteins
Carbon	42.7	84.6	52.7
Hydrogen	3.2	5.1	7.1
Oxygen	54.1	10.3	22.6
Nitrogen	–	–	16.5
Sulfur	–	–	1.1

$$94.95 = p + q \quad (6.E6)$$

$$88.25 + 2f = 4p + 3r + 2s \quad (6.E7)$$

$$86.30 + f = 2q \quad (6.E8)$$

$$1.16 = r \quad (6.E9)$$

$$0.03 = s \quad (6.E10)$$

Solving above equations, the values of f , p , q , r , and s were found to be

$$f = 30.62$$

$$p = 36.49$$

$$q = 58.46$$

$$r = 1.16$$

$$s = 0.03$$

Similarly, for wheat bran,

$$9.63 = p + q \quad (6.E11)$$

$$9.94 + 2f = 4p + 3r + 2s \quad (6.E12)$$

Table 6.4 Concentration of carbon, hydrogen, oxygen, nitrogen, and sulfur in cassava wastewater, wheat bran, and sewage sludge

Elements	Cassava wastewater			Wheat bran			Sewage sludge		
	Carbohydrates	Lipids	Proteins	Carbohydrates	Lipids	Proteins	Carbohydrates	Lipids	Proteins
Carbon (ppm)	1070.64	16.70	52.03	62.46	25.38	27.67	63.20	39.51	38.74
Hydrogen (ppm)	80.24	1.01	7.01	4.68	1.53	3.73	4.74	2.38	5.22
Oxygen (ppm)	1356.48	2.03	22.31	79.14	3.09	11.87	80.07	4.81	16.61
Nitrogen (ppm)	–	–	16.29	–	–	8.66	–	–	12.13
Sulfur (ppm)	–	–	1.09	–	–	0.58	–	–	0.81

$$5.88 + f = 2q \quad (6.E13)$$

$$0.62 = r \quad (6.E14)$$

$$0.02 = s \quad (6.E15)$$

Solving above equations, the values of f, p, q, r, and s were found to be

$$f = 4.68$$

$$p = 4.35$$

$$q = 5.28$$

$$r = 0.62$$

$$s = 0.02$$

Similarly, for sewage sludge

$$11.79 = p + q \quad (6.E16)$$

$$12.34 + 2f = 4p + 3r + 2s \quad (6.E17)$$

$$6.34 + f = 2q \quad (6.E18)$$

$$0.87 = r \quad (6.E19)$$

$$0.03 = s \quad (6.E20)$$

Solving above equations, the values of f, p, q, r, and s were found to be

$$f = 6.20$$

$$p = 5.52$$

$$q = 6.27$$

$$r = 0.87$$

$$s = 0.03$$

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