Chapter 7

Biomethane Production as an Alternative for the Valorization of Agricultural Residues: A Review on Main Substrates Used as Renewable Energy Sources

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Abstract Biomethane is one of the most promising gases among renewable energy sources. It is a fuel that can be applied to turbines, boilers, domestic stoves, and internal combustion engines to generate heat or electricity. One of the ways to obtain this gas is through biological technologies, such as anaerobic digestion reactors, that can convert agricultural residues like vinasse, swine effluent, and animal manure into biomethane, preventing their inappropriate disposal in the environment. Several studies show that co-digestion, the association of more than one type of waste, can be advantageous for obtaining this gas due to the greater availability of nutrients and diversification of microbial communities. However, obtaining biomethane and using it as a sustainable energy source still has challenges that range from improving the biological process to choosing the most efficient conversion technology. This chapter aims to present the main types and characteristics of agricultural wastes used as substrates and highlight some important factors that can influence the efficiency of biomethane production in anaerobic reactors.

Keywords Biomethane · Agricultural waste · Agro-industrial residues · Renewable energy

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P. Singh (ed.), *Emerging Trends and Techniques in Biofuel Production from Agricultural Waste*, Clean Energy Production Technologies,
https://doi.org/10.1007/978-981-99-8244-8_7

7.1 Introduction

In a sustainable society, wastewater and waste treatments are strategies for pollution control, seeking other benefits through energy recovery and the production of value-added materials. The treatment of effluents containing high easily degradable organic matter, such as agricultural residues, can result in a positive net energy balance.

Energy sources potentially available in the agricultural sector are plant biomass and animal residues, such as crop wastes, animal manure, and agro-industrial effluents like vinasse, cassava wastewater, and dairy wastewater (Pattnaik et al. 2019; Guo et al. 2021). However, most agricultural waste is deposited in landfills or incinerated, promoting adverse consequences for the environment (Obi et al. 2016).

Biological treatments of agricultural waste are attractive because they can provide bioenergy or chemical compounds with associated added value and simultaneously achieve pollution control and recovery of by-products. The choice of bioprocess depends on technical and economic feasibility, operational simplicity, social demand, and political priority (Angenent et al. 2004).

The main bioprocesses to generate bioenergy or biochemical compounds while treating agricultural residues are methanogenic anaerobic digestion, biological hydrogen production, microbial fuel cells, and fermentation. Anaerobic digestion is a process that occurs in the absence of oxygen by the action of microorganisms that degrade organic matter, producing biogas as a by-product of economic value (Silva et al. 2013).

Biogas is a mixture of gases generated during the natural decomposition of organic material through biological processes. Its value is associated with the presence of methane gas, which gives it an approximate calorific value of 5200 kcal/Nm^3 and makes it attractive for applications such as heating and electricity generation, transport, or injection into the natural gas network (Ferreira et al. 2019; Khan et al. 2021).

Biogas is mainly composed of methane (CH_4) at 45–75%, CO_2 between 20–55%, and small amounts of other gaseous compounds (impurities) such as hydrogen sulfide (H_2S), nitrogen (N_2), hydrogen (H_2) and oxygen (O_2) (Rasapoor et al. 2020; Atelge et al. 2021). Classified as impurities, the gases CO_2, H_2S, and NH_3, when in high concentrations, harm the quality of biogas. CO_2, for example, reduces the calorific value, while H_2S gives off an unpleasant odor and makes biogas corrosive to metallic materials (De Farias Silva et al. 2019; Li et al. 2019; Cremonez et al. 2021). Biomethane production happens when these contaminants are removed by purification processes, increasing energy density and improving the efficiency of biogas use (Mulu et al. 2021).

In addition to biogas, the liquid effluent resulting from the anaerobic digestion process can be used as a biofertilizer, making the process an attractive option in the treatment of this type of waste, which has increased worldwide over the years following population growth (Awogbemi and Von Kallon 2022).

7.2 Anaerobic Digestion Phases

Anaerobic digestion encompasses a set of reactions occurring simultaneously through microbial action and comprises five main steps (Fig. 7.1): hydrolysis, acidogenesis, acetogenesis, sulfetogenesis, and methanogenesis (McCarty 1964).

Hydrolysis is the phase where the conversion of complex organic materials (polymers) into simpler substances (sugars, amino acids, and peptides) occurs. Hydrolytic fermentative bacteria excrete exoenzymes, enabling the transformation of the particulate matter into dissolved constituents. Among the bacteria with hydrolytic capacity, the genera *Clostridium*, *Micrococcus*, *Staphylococcus*, *Bacteroides*, *Butyvibrio*, *Bacillus*, *Acetivibrio*, and *Eubacterium* can be mentioned (Ordaz-Díaz and Bailón-Salas 2020; Kumar Khanal et al. 2021).

In a second phase, called acidogenesis, most microorganisms ferment sugars, amino acids, and fatty acids, producing organic acids (mainly acetic, propionic, and butyric acids), alcohols (ethanol), ketones (acetone), carbon dioxide, and hydrogen. The production of acids promotes a decrease in the pH of the medium (Ordaz-Díaz and Bailón-Salas 2020; Li et al. 2019).

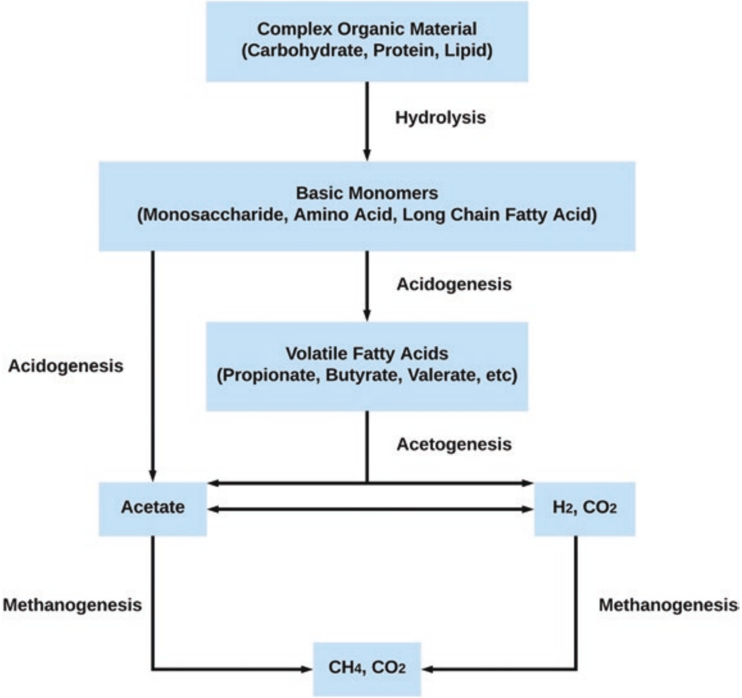

Fig. 7.1 Steps of the anaerobic digestion process. (Source: Adapted from De Farias Silva et al. 2019)

Acetogenesis promotes the oxidation of acidogenesis products yielding a substrate suitable for methanogenic bacteria. In this phase, bacteria act as intermediaries in the acidogenesis and methanogenesis processes, forming hydrogen and carbon dioxide, with a pH decrease caused by acetic and propionic acid generation. In the fourth step, methanogenesis, methanogenic microorganisms consume the H^+ ions in the solution and convert hydrogen and carbon dioxide into methane (Lyu et al. 2018; Li et al. 2019).

When the substrate has the presence of sulfate in its composition, a fifth phase called sulfetogenesis occurs, where sulfate-reducing bacteria (SRB) act by reducing sulfate to sulfide, competing with methanogenic archaea for H_2 and organic matter (Lyu et al. 2018).

7.3 Biomethane Production from Agricultural Wastes

The amount of biomethane produced depends on the composition of the materials involved in the anaerobic process. Numerous organic and inorganic compounds form biomass structures, with carbohydrates, proteins, and lipids as the main organic components. This composition varies significantly for each type of substrate (Rasapoor et al. 2020). A simplified bibliometric analysis encompassing 1000 works between 2019 and 2023 on the production of biomethane from agricultural waste shows that some of the most studied materials with energy purposes are manure (chicken, swine, cattle, dairy and pig), straw, crop residues and food waste (Fig. 7.2).

Table 7.1 presents some research aimed at the production of methane from the anaerobic digestion of agricultural residues. Fernandes and De Oliveira (2006) studied a two-stage system using a compartmentalized reactor (ABR) followed by a

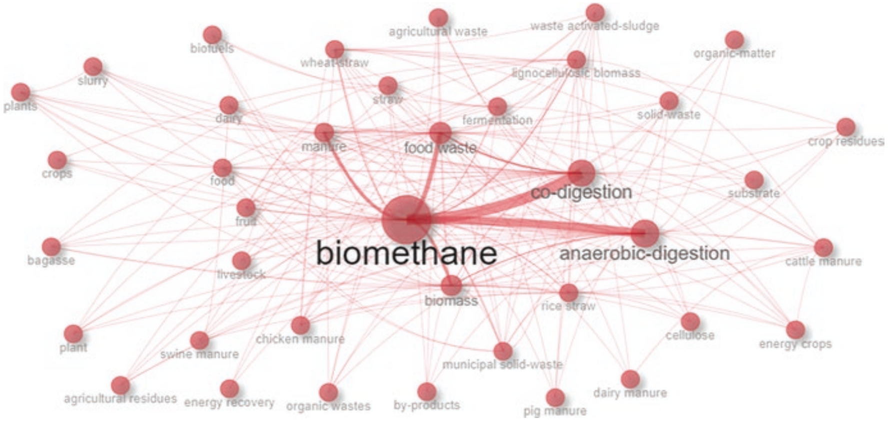

Fig. 7.2 Simplified bibliometric analysis on biomethane production from agricultural waste

Table 7.1 Methane production from agricultural residues

Residue (substrate)	Biodigester type	Results	References
Pig farming wastewater	Compartment reactor (ABR) and UASB reactor	ABR: 0.068 m^3-CH_4/kg-Total $COD_{removed}$	Fernandes and De Oliveira (2006)
		UASB: 0.053 m^3-CH_4/kg-Total $COD_{removed}$	
		System: 0.078 m^3-CH_4/kg-Total $COD_{removed}$	
Swine manure and sweet potato (SP) or cassava wastewater (CW)	Semi-continuous digesters	Biogas yield	Villa et al. (2020)
		SP: 901 L_N/kg-VS_{added}	
		CW: 883 L_N/kg-VS_{added}	
		CH_4	
		SP: 590.5 L_N/kg-VS_{added}	
		CW: 546.8 L_N/kg-VS_{added}	
Cow manure (CM) with grass silage (GS), sugar beet tops (SBT) and oat straw (OS)	Continuously stirred tank reactors (CSTR)	CM-GS: 268 L-CH_4/kg-VS_{added}	Lehtomäki et al. (2007)
		CM-SBT: 229 L-CH_4/kg-VS_{added}	
		CM-OS: 213 L-CH_4/kg-VS_{added}	
Fish waste silage (FWS) and cow manure (CM)	Semi-continuous stirred tank reactors	0.400 L-CH_4/g-VS	Solli et al. (2014)
Sun dried sugar beet pulp and cow manure	Semi-continuous reactor	315 mL-CH_4/g-VS_{added}	Gómez-Quiroga et al. (2022)
Vinasse and molasses	Upflow anaerobic sludge blanket (UASB)	0.245 m^3-CH_4/kg-$COD_{removed}$	Santana Junior et al. (2019)
Manipueira (cassava processing wastewater)	Fixed bed anaerobic reactor	0.430 ± 0.150 $L_{methane}$/g-COD	Oliveira et al. (2017)
Vinasse	Bench scale batch reactor	541.4 L-CH_4/kg-VS	Kiyuna et al. (2017)
Manipueira with addition of cassava waste	Upflow Anaerobic Sludge Blanket (UASB)	259 mL-CH_4/g-$COD_{removed}$	Chavadej et al. (2019)

UASB reactor to treat swine wastewater. ABR reactor was operated with hydraulic retention times (HRT) between 56 and 18 h, and from 13 to 4 h for the UASB reactor. The total COD ranged from 7557 to 11,640 mg/L, and methane content was above 70% for both reactors. The highest specific methane yield of 0.068 m^3-CH_4/kg-$COD_{total\text{-}removed}$ occurred in ABR with an OLR of 5.05 kg-COD_{total} (m^3/day). UASB reached a maximum of 0.053 m^3-CH_4/kg-$COD_{total\text{-}removed}$ at 2.84 kg-COD_{total} (m^3/day).

Some studies indicate that the use of pig manure as a substrate in the mono-digestion process difficulted the process due to the presence of nitrogen concerning available organic carbon (Wang et al. 2012; Yin et al. 2015). The high nitrogen content can generate an elevated level of toxic ammonia. Thus, materials rich in organic carbon need to be added to swine manure to provide the necessary organic carbon to improve methane production (Ojediran et al. 2021).

Villa et al. (2020) used semi-continuous reactors to analyze the co-digestion of swine manure (SM), sweet potato (SP), and cassava wastewater (CW) in different proportions. Initially, the authors tested a batch reactor with C/N ratios of 10/1, 13/1, 17/1, and 22/1. Based on the results of the first stage, the C/N ratio of 10/1 showed greater reductions of volatile solids and specific biogas productions. In a second step, the authors concluded that co-digestion of swine manure with sweet potato and cassava wastewater resulted in increased methane yields of 31.5% and 21.8% (SP: 590.5 ± 23.9 L_N/kg-VS_{added}; CW: 546.8 ± 14.9 L_N/kg-VS_{added}) compared to single digester (SM: 449.5 ± 23.0 L_N/kgVS_{added}).

Anaerobic digestion of cattle farming residues has also been widely adopted in co-digestion with other agricultural waste. Lehtomäki et al. (2007) analyzed the co-digestion of cow manure with different residues from plant production (grass, beet husk, and oat straw—grass silage, sugar beet tops, and oat straw) in continuously stirred tank bench-scale reactors (CSTRs). The highest specific methane yields were 268, 229, and 213 L-CH_4/kg-VS_{added} when adding 30% of grass, beet husk, and oat straw to cow dung, respectively. Co-digestion with 30% plant material promoted an increase in methane production from 16% to 65% concerning manure mono-digestion. The addition of 40% of plant material resulted in a decrease in specific methane yield (4–12%).

Solli et al. (2014) evaluated the co-digestion of cow manure (CM) with fish waste silage (FWS) in increasing volumes (3%, 6%, 13%, 16%, and 19%). The highest methane production of 0,400 L-CH_4/g-VS was obtained when adding 16% of FWS, corresponding to twice the methane production obtained from CM mono-digestion.

Gómez-Quiroga et al. (2022) observed the effect of HRT (30–3 days) and OLR (2–24 g-VS/$L_{reactor}$/day) in semi-continuous reactors on sugar co-digestion sun-dried sugar beet with cow manure. The highest methane yield was 315 mL-CH_4/g-VS_{added}, obtained in the 5-day and OLR of 12.47 g-VS/$L_{reactor}$/day. The results demonstrated the possibility of obtaining great efficiency and stability in the co-digestion of agro-industrial residues and cattle manure in short HRTs.

Vinasse is a residue from sugarcane processing quite studied for bio-methane production. This wastewater has application as a biofertilizer in the cultivation of sugarcane, supplying the needs of the soil with some minerals such as potassium, nitrogen, and phosphorus, being an alternative to synthetic fertilizers, mainly to the supply of potassium (Ferraz Júnior et al. 2016; Janke et al. 2016). However, an incorrect and indiscriminate application can result in damage to the soil and groundwater due to the high organic load and low vinasse pH (Fuess and Garcia 2014). Thus, anaerobic digestion is an alternative for the correct disposal of vinasse, reducing its polluting load and producing bioenergy.

However, the interruption of industrial operation during the off-season period requires a new start of the reactors at each harvest, hindering the viability of using vinasse for bio-methane production on a real scale. Some authors have already reported difficulties in resuming sugarcane from full-scale reactors in the anaerobic treatment of vinasse of each crop (Souza et al. 1992; Aguiar et al. 2011).

The use of sugarcane molasses, a by-product of the sugar refining process, during the off-season period is an option to ensure continuous bio-methane production. Santana Junior et al. (2019) used a two-stage UASB reactor (R1 and R2) operated at thermophilic temperature to treat molasses. The highest methane yield was 0.245 m^3-CH_4/kg-$COD_{removed}$ in reactor R2 (using vinasse as substrate) with OLR between 0.15 and 3.50 kg/m^3/day. When fed with molasses, yields were 0.056 and 0.090 m^3-CH_4/kg-$COD_{removed}$ (OLR of 7.1–7.5 kg/m^3/day in R1) in R1 and R2, respectively. The authors observed a 58% increase in energy production for the two-stage system compared to the single stage.

Another concern about anaerobic digestion of vinasse is the use of sulfuric acid by sugarcane distilleries to avoid microbial contamination and yeast flocculation in fermentation vessels (Barth et al. 2014). This procedure can entail sulfate concentrations up to 9 g/L in vinasse (Kiyuna et al. 2017), which potentially inhibits the development of anaerobic microbial populations responsible for bio-methane production, such as methanogenic Archaea.

Kiyuna et al. (2017) evaluated the influence of sulfate on the anaerobic digestion of vinasse using batch reactors under thermophilic conditions. The authors adopted three COD/sulfate ratios (12.0, 10.0, and 7.5) to analyze COD removal and bio-methane production. The system achieved COD removals above 80%, indicating that interference of sulfetogenesis was negligible to the degradation of organic matter. The authors observed a reduction in bio-methane production of 35% for the COD/sulfate of 7.5 (351.5 L-CH_4/kg-VS) concerning the ratio of 12.0 (541.4 L-CH_4/kg-VS).

Oliveira et al. (2017) used an anaerobic fluidized bed reactor (AFBR) fed with cassava to produce hydrogen in the acidogenic phase. Later, a fixed bed reactor (FBR) was fed with RALF effluent to produce methane in the methanogenic phase. Expanded clay and shells of *sururu* (Mytella falcata) were used as support material in AFBR and FBR, respectively. The highest hydrogen yield was 1.91 mol-H_2/mol-glucose in the HRT of 2 h, and the highest methane yield was 0.430 ± 0.150 L-methane/g-COD in the HRT of 12 h. The authors also observed that the shells of *sururu* neutralized the pH in the fixed bed reactor efficiently.

Chavadej et al. (2019) analyzed UASB reactors to produce biohydrogen and bio-methane in separate phases. The systems were operated at a thermophilic temperature (55 °C) using different concentrations of cassava residues. The concentration of 1200 mg/L resulted in the best biogas compositions: 42.3% H_2, 55% CO_2, and 2.70% CH_4 for the acidogenic reactor, and 70.5% CH_4, 28% CO_2, and 1.5% H_2 for the methanogenic reactor. The maximum biohydrogen and bio-methane yields were 15 mL-H_2/g-$COD_{removed}$ and 259 mL-CH_4/g-$COD_{removed}$, displaying augmentations of 45.2% and 150% in H_2 and CH_4 production, respectively, compared to the system without the addition of cassava residue.

7.4 Substrate Pre-treatment

Most agricultural waste used in anaerobic digestion is of animal origin, especially from swine and cattle farming (Filho et al. 2018). According to Avaci et al. (2013), animal manure has already undergone a digestion process in the animal's intestine, which would facilitate treatment.

In the case of vegetable waste, the lignin presence hinders the anaerobic digestion process since it is a compound difficult to digest, despite the high fermentation potential. The high lignocellulosic structure resistance to hydrolysis causes operational system instability and restrains biodegradability (Yang et al. 2015). Thus, it is necessary to adopt some pre-treatment of this type of waste so that the microorganisms involved in the process can decompose the biomass more efficiently and quickly, increasing the methane contents in the biogas composition at the end of the process (Tian et al. 2018).

The methods of substrates pre-treatment for anaerobic digestion can be classified as mechanical, thermal, chemical, enzymatic, or combinations of these. Mechanical pre-treatments are ultrasound, high pressure, grinding, and extrusion. They aim to reduce the size of waste particles, increasing their solubility and the contact surface with the microorganisms. This type of pre-treatment is generally adopted when the waste has large particles, such as food-industry, agricultural, and household organic residues (Appels et al. 2008; Carlsson et al. 2012).

Thermal pre-treatments employ extreme temperatures and high pressures, seeking to avoid evaporation. In this case, the objective is the solubilization or degradation of components with a high molecular weight into simpler substances that can be decomposed more easily. Freezing cycles (from −10 to −80 °C) and thawing are also adopted as a pre-heat treatment in search of greater solubilization and reduction in the size of the waste particles (Wang et al. 1999; Montusiewicz et al. 2010; Carlsson et al. 2012).

The chemical pre-treatment aims to destroy the cell wall and membrane present in the waste by the addition of acids or bases, increasing its solubilization. Before the start of the operation, it is necessary to neutralize the substrate pH to promote methane production. The pre-treatment combining chemical and thermal techniques is an advantageous option that employs lower temperatures (Appels et al. 2008; Carlsson et al. 2012).

Oxidation of substrates, another form of chemical pre-treatment, can be carried out using oxygen or air, at high temperatures and pressures, or ozone. Despite the efficiency of substrate solubilization, oxidation can be unfeasible due to high energy costs (Appels et al. 2008; Carlsson et al. 2012). Enzymatic pre-treatment aims to hydrolyze organic waste, facilitating the action of microorganisms (Pinheiro 2021).

The requirement to adopt a substrate pre-treatment constrains the use of plant residues in the anaerobic digestion process. In general, its disposal in the soil is a more accessible alternative with a low environmental impact, unlike animal waste. Plant residues provide nutrients and help maintain soil moisture, besides protecting against erosion (Ramalho Filho and Beek 1995).

However, the co-digestion of plant waste with other types of residues can overcome the need for pre-treatment. This technique associates two or more substrates to accelerate the anaerobic digestion of complex compounds, increasing biogas production from 25% to 400% compared to mono-digestion of the same substrates (Siddique and Wahid 2018). The co-digestion strategy can bring advantages such as reduction of toxic compound concentration, better loading of the biodegradable substrate, improved digestion rate, and increased biogas production (Cook et al. 2017; Neshat et al. 2017; Bedoić et al. 2019).

7.5 Conclusions

The anaerobic digestion process is a suitable alternative for biomethane production using different types of waste. The foremost agricultural residues studied as substrates are leftovers, crop residues, agro-industrial effluents, and animal manure such as swine and cattle. Animal composts are largely applied in anaerobic reactors since it is a material that has already gone through a digestion process in the animal's intestine, which would facilitate the treatment.

Despite the high fermentation potential of vegetal wastes, lignin is a plant constituent that hampers the anaerobic digestion process, requiring the adoption of pretreatment techniques, which restricts its use. However, the co-digestion of materials of plant origin with other types of residues is a promising alternative for biomethane production. Co-digestion is an efficient and economical alternative, replacing the need to adopt substrate pre-treatments and overcoming the obstacles of mono-digestion. Another strategy that can result in elevated energy gain is separate acidogenic and methanogenic phases in different reactors, producing bio-hydrogen and biomethane separately.

Therefore, this chapter presented a waste and wastewater variety from the agricultural sector commonly unused or inappropriately discarded in the environment. The treatment of agricultural residue by anaerobic digestion can generate value-added products such as biogas and natural biofertilizer to enrich soils lacking in organic matter. Anaerobic digestion presents economic advantages, reducing costs with the gas purchase and waste transport, and environmental benefits, avoiding the disposal of pollutants and generating a renewable energy source.

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