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Review

Biochemical Methane Potential Assays for Organic Wastes as an Anaerobic Digestion Feedstock

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Abstract: The anaerobic digestion process is applied worldwide in the treatment of various organic wastes, allowing energy production from biogas and organic recovery from digested sludge. In the evaluation of suitable substrates for anaerobic digestion, Biochemical Methane Potential assays are the most applied, and, despite several efforts to standardize this method, it is observed that there are still several studies that do not apply all the criteria. This current paper's main goal is to present a review of anaerobic feedstocks, BMP methodologies, experimental conditions, and results of specific methane production from 2008 to 2023. A wide range of anaerobic feedstocks was found, which was divided into five groups: animal manure, sludge, food wastes, energy crops, and other organic wastes. Several parameters were used to characterize the anaerobic feedstocks, like TS, VS, COD, and pH, displaying different value ranges. The number of publications concerning BMP assays increased significantly over the years until 2021, having stabilized in the last two years. This evolution allowed for several attempts to standardize the BMP method with positive developments, but there are still some gaps in the experimental conditions and the determination of specific methane production. All of this makes the comparison of some studies a challenge.

Keywords: anaerobic digestion; biochemical methane potential (BMP); organic wastes; feedstock and review

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1. Introduction

The anaerobic digestion (AD) process is a widely applied technology to treat several feedstocks from different sources, like manure (dairy, pig, sow, chicken, sheep, and goat), agricultural waste (straw rice husk, sugar, dry grass, maize, corn, and potato), organic waste (food waste, fruit and vegetables, organic fraction of municipal solid waste, slaughterhouse waste, and exhaust kitchen oil) and sludge (sewage sludge and food industry sludge) [1,2]. This process also allows for the reduction in greenhouse gas emissions because it treats waste that would otherwise be sent to landfill.

In recent decades, the anaerobic digestion process has received increasing attention due to the recovery of energy [2,3] from biogas production, which contributes to achieving the targets of renewable energies [1,4]. Renewable energy sector growth is extremely important with simultaneous socio-economic development in order for all European member states to become climate neutral. There are several ways to recover energy from biogas; for example, methane can be burned or can be used as a substitute for natural gas and car fuel [5]. Another issue is the industrial gas represented by biogenic CO_2, which can be a feedstock for the food and drinks industry. Moreover, biogenic CO_2 is not a greenhouse gas, because it is produced by a sustainable source, which allows the CO_2 be sequestered in the growing of biomass. By 2030, production of biogas and biomethane of 35 billion cubic meters (bcm) is expected, which may represent 46 Mt of biogenic CO_2 [6].

Also, the solid residue resulting from AD and designated by digestate can be used as a fertilizer or soil additive due to the nutrients and organic contents [2,7], contributing to the circular economy. On average, the digestate amount can be estimated by a factor of 75 t of digestate (dry matter) per GWh of biogas or biomethane produced. Therefore, in Europe, 258 to 222 Mt of fresh matter were estimated for 2021 and 2030, and the digestate can be between 222 and 455 Mt [6].

In recent years, biogas and biomethane have been produced throughout Europe. Biogas can be defined as the gas produced from anaerobic digestion without upgrading the methane content, and biomethane is obtained after the purification of biogas to nearly 100% of methane. In Europe, the number of biogas plants presents a period of rapid growth between 2010 and 2014, with 10,574 and 16,979 plants, respectively. From 2014 to 2021, the growth in the number of plants slowed down, reaching a total of 18,843 plants in 2021 (Figure 1). Also, the number of biomethane plants grew rapidly from 182 in 2011 to 1067 in 2021. In 2022, a significant increase was expected because, until September, 115 new biomethane plants started operation. In terms of energy production, a biogas plant produces around 8 GWh per year, and a biomethane plant produces, on average, 35 GWh per year [6].

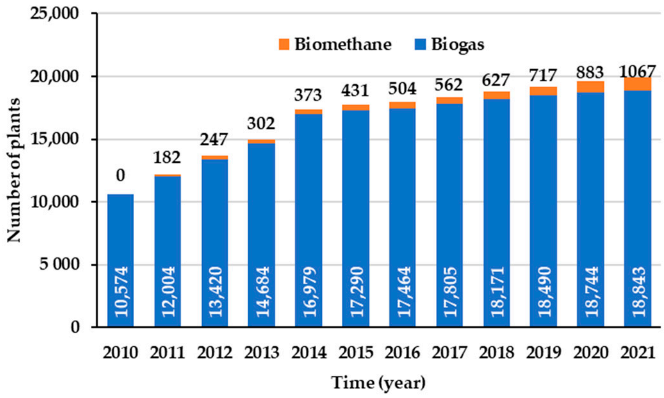

Figure 1. Number of biogas and biomethane plants in Europe between 2010 and 2021, data from [6].

The selection of a suitable substrate for anaerobic digestion is based on the assessment of its physicochemical characteristics and composition which influence the anaerobic degradability [8,9].

The most used methodologies to gauge biodegradability are the biochemical methane potential (BMP) assays [10,11]. These tests may also allow the determination of optimal conditions for the anaerobic process [9,12] and several factors, like pH, temperature, and substrate–inoculum ratio, that affect biogas quality [13]. In addition, the BMP assays are an effective method for selecting potential substrates for anaerobic digestion.

Despite the widespread use of BMP tests to measure the ultimate methane production from various organic substrates and several attempts to obtain a standard methodology, such as ASTM D 5511 (1994) [14], ISO 11734 (1995) [15], ISO 15985 (2004) [16], and VDI 4630 (2016) [17], there is still no generally accepted experimental procedure, which is confirmed by the variability of BMP tests results presented in the papers published between 2007 and 2018 [18]. The variability of results is due to several factors, such as different experimental conditions, methodologies applied, and measuring equipment [4].

This current paper's main goal is to present an extensive review of anaerobic feedstocks, BMP factors, methodologies, and results. Therefore, in Section 2, five groups of AD feedstocks are described and characterized. In Section 3, the evolution of the AD process and BMP assays in the last four decades is presented, as well as the different methodologies used in recent years. In Section 4, BMP assay experimental conditions and results are described. Finally, in Section 5, different models are applied to determine the methane productivity of feedstocks and to predict the cumulative methane production.

2. Feedstocks for Anaerobic Digestion

Suitable feedstocks for anaerobic digestion can be classified according to the moisture content as solids, slurries, and liquids (concentrated and diluted). According to the biodegradable fraction, feedstocks can be classified from readily degradable wastewater up to complex high-solid waste [19]. Usually, feedstocks include animal manure (e.g., dairy, swine, beef, poultry), sludge (e.g., sewage sludge, industry sludge), food wastes (e.g., household, restaurant, grocery, food production), energy crops (e.g., maize silage, Napier grass, energy cane, switchgrass, wheat) [2,20], and other organic wastes (e.g., fats, oils, grease, crop residue, winery/brewery waste) [21].

Feedstock biodegradability is very important for the AD process design because it influences the biogas or methane yield and the percentage of solids (total or volatile) that are destroyed [22].

The most used parameters for characterizing these feedstocks are total solids (TS), volatile solids (VS), chemical oxygen demand (COD), and pH. In addition, total Kjeldahl nitrogen, ammonium, and alkalinity can be used to predict a potential inhibition problem in feedstocks [23].

In this present work, an extensive literature review was performed to identify the potential feedstocks sources for AD. A wide range of feedstocks was found and classified into five groups, as shown in Figure 2.

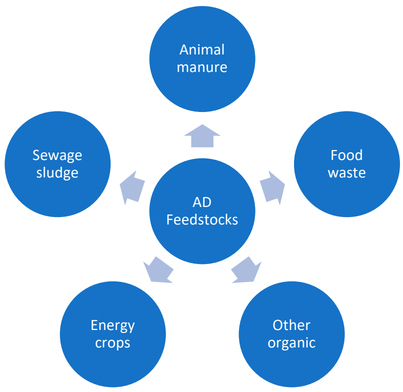

Figure 2. Anaerobic feedstock groups.

The typical characteristics of animal manure feedstocks are presented in Table 1. It can be observed that the animal manure feedstocks presented a pH between 6.3 and 8.87. The feedstock content of TS and VS is expressed in different units, possibly due to the variability of the moisture content. This makes the comparison of the different feedstocks more difficult, since it is necessary to use the density of each material. According to the different units, the TS ranges from 3.8% to 79.86% of TS; from 47.67 g/kg to 937.98 g/kg, and from 3.97 g/L to 73.6 g/L. The values of VS contents are presented on different bases, e.g., related with TS, dry mass, and wet weight. These values are also presented with different units such as %, g/L and g/kg, corresponding to the ranges from 2.8% to 89.96%, from 1.73 g/L to 64.8 g/L, and from 26.07 g/kg to 794 g/kg. The organic matter represented by COD is expressed in mass per volume (g/L) and mass per mass (g/kg) ranging from 24.6 g/L to 307 g/L and from 71 g/kg to 915 g/kg, respectively. According to the feedstock database created by Moretta et al. (2022) [2], the TS and VS contents in animal manure range from 19.15% to 64.76% and from 64.69% to 76.00%, respectively.

The great variability in the characteristics of the animal manure shown in Table 1 is probably due to the differences technics of animal husbandry concerning cow, pig, goat, poultry, and buffalo.

Mata-Alvarez et al. (2014) [24] mentioned that in most publications, pig and cow manures are the most used as feedstocks for the AD process. In this present review, the most frequently animal manure feedstocks were from pig (eight references), dairy (seven references), and cow (six references), as shown in Table 1.

Table 1. Characteristics of animal manure feedstocks for anaerobic digestion.

Feedstocks	TS	VS	COD	pH	Reference
Cattle manure	3.8–9.3%	2.8–7.4% (wet weight)	na	na	[25]
Cow dung	19.02 wt.%	11.84 wt.%	109.2 g/L	na	[26]
Cow manure	3.97 ± 0.09 g/L	1.73 ± 0.09 g/L	307 ± 2 g/L	7.24	[27]
Cow manure	28.81 (1.07) g/L	18.50 (0.84) g/L	na	7.05 (0.1)	[28]
Cow manure from slaughterhouse	221.6 g/kg	208.5 g/kg	258.8 g/kg	na	[29]
Cow slurry	78 g/kg	782 g/kg TS	na	7.7	[30]
Dairy manure	13.6 ± 0.4%	11.9 ± 0.4%	na	na	[31]
Dairy manure	10.2%TS/FM	83.6%VS/TS	na	na	[32]
Dairy manure	124.0 g/kg	102.1 g/kg	128.9 g/kg	na	[33]
Dairy manure	26.62 ± 0.86%	19.37 ± 0.43%	na	na	[34]
Dry cow manure	937.98 ± 3.82 g/kg	463.02 ± 5.93 g/kg	na	8.87 ± 0.24	[35]
Fresh buffalo manure	109.6 (0.6) g/kg wet	89.1 (0.7) g/kg wet	na	7.05 (0.06)	[36]
Goat manure	79.86 ± 1.78%	66.72 ± 1.45%	na	na	[34]
Liquid pig manure	26.5 ± 5.3 g/L	18.6 ± 4.3 g/L	24.6 ± 4.0 g/L	8.2 ± 0.3	[37]
Liquid poultry manure	47.67 ± 2.64 g/kg	26.07 ± 1.52 g/kg	na	8.39 ± 0.31	[35]
Livestock residues on-farm	42–45 wt%, wet basis	31–35 wt%, wet basis	na	na	[38]
Manure separated liquid	57.5 g/kg	40.5 g/kg	71.0 g/kg	na	[33]
Pig slurry	69.9 g/kg	794 g/kg TS	na	7.0	[30]
Pig slurry	1.42 (70)% FM, *w*/*v*	1.04 (61)% FW, *w*/*v*	na	6.73 (3.9)	[39]
Pig slurry	13.0–18.0 g/L	7.6–12.9 g/L	27.7–33.1 g/L	6.3–6.5	[40]
Poultry litter	77 ± 1.3%	70 ± 1.5%	915 ± 67 g COD/kg_{waste}	na	[41]
Separated dairy manure	41.1 ± 0.06 g/L	32.4 ± 0.1 g/L	52.1 ± 0.4 g/L	6.82	[42]
Slurry from dairy farm	87.5 ± 2.1 g/kg	66.9 ± 1.8 g/kg	na	na	[43]
Solid fraction of dairy manure	25.8 ± 0.3%	23.3 ± 0.4%	na	na	[44]
Solid fraction of pig manure	166.4 ± 0.2 g/kg	138.6 ± 0.2 g/kg	197 ± 3 gO_2/kg	na	[45]
Solid waste produced in RAS	11.65 ± 1.15 g TS/L	7.57 ± 0.87 g TVS/L	10.95 ± 0.09 gCOS/L	na	[46]
Swine manure	23.58 ± 1.06%	89.86 ± 2.15% TS	na	na	[47]
Swine manure	31.22 ± 3.97%	23.27 ± 2.61%	na	na	[34]
Swine manure	23.34 ± 0.24 g TS/L	15.49 ± 0.43 g VS/L	na	7.5 ± 0.1	[48]
Unseparated dairy manure	73.6 ± 2.0 g/L	64.8 ± 1.9 g/L	55.9 ± 2.5 g/L	6.93	[42]

COD—chemical oxygen demand; FM—fresh matter; na—not available; RAS—recirculating aquaculture systems; TS—total solids; VS—volatile solids.

The characteristics of sludge, food waste, energy crops, and other organic feedstocks are presented in Tables 2–5, respectively.

Table 2. Characteristics of sludge feedstocks for anaerobic digestion.

Feedstocks	TS	VS	COD	pH	Reference
Aeration basin sewage sludge	14.98 g/L	6.41 g/L	na	na	[49]
Aerobic granular sludge	29.6–106.1 g/L	27.3–60.1 g/L	39.7–85.7 g/L	na	[50]
Biological sludge from WWTP	71.2 g/kg	54.9 g/kg	83.9 g/kg	na	[29]
Dehydrated sludge	19.17%	7.95%	na	na	[49]

Table 2. *Cont.*

Feedstocks	TS	VS	COD	pH	Reference
Excess sludge (dewatered sludge)	97.9 ± 0.525 g/L	37.2 ± 0.250 g/L	48.34 ± 0.952 g/L	6.5 ± 0.1	[51]
High solid sludge from municipal WWTP	16.7 ± 0.5%, *w*/*w*	70.5 ± 0.1 VS/TS	166.0 ± 2.3 g/L	na	[52]
Oxidized sludge	6.53 g/L	2.04 g/L	na	na	[49]
Pharmaceutical sludge	3.1%	94.7%	36.64 g/L	7.09	[53]
Primary sludge from a municipal WWTP	26.3 ± 0.26 g/L (TSS)	20.0 ± 0.250 g/L (VSS)	42.8 ± 0.18 g/L	5.0 ± 0.1	[54]
Primary Sludge from municipal WWTP	3.2 ± 0.30%	82.6 ± 0.40 TS%	na	na	[55]
Pulp and paper industry WWTP biosludge	1.1–1.5%	0.7–1.0%	12 (1) g/L	7.4	[56]
Refinery waste -Waste activated sludge	0.4%	77%	5 g/L	na	[57]
Refinery waste-Flotation sludge	10.1–16.9%	74–85%	228–406 g/L	na	[57]
Secondary sewage sludge from WWTP	19.05 ± 1.21 g/L	13.99 ± 1.05 g/L	20.593 ± 2.513 gO_2/L	6.98 ± 0.17	[58]
Sewage sludge	3.67 ± 0.01 g/L	2.69 ± 0.03 g/L	53.9 ± 1.2 g/L	6.9 ± 0.2	[59]
Sewage sludge from a WWTP	33.56 (1.06) g/L	25.9 (0.66) g/L	37.81 (0.13) g/L [1]	6.23 (0.11)	[28]
Thickened sludge	30.3 ± 0.216 g/L	20.05 ± 0.145 g/L	44.8 ± 0.281 g/L	7.6 ± 0.1	[60]
Thickened sludge from a WWTP	4.98 ± 0.6%	3.68 ± 0.6%	51.6 ± 0.7 g/L	6.7 ± 0.1	[61]
Thickened waste activated sludge	14.18%	6.72%	37.04 g/L	6.40	[62]
Thickened waste activated sludge	14.2 ± 0.16%	6.7 ± 0.09%	37.04 ± 1.332 g/L	6.4 ± 0.00	[63]
Waste activated sludge	47.3 ± 0.4 g/kg	40.5 ± 0.1 g/kg	69.9 ± 0.5 (gO_2/L)	5.9	[64]
Waste activated sludge from a WWTP	2.97%	2.49%	49.7 g/L	7.15	[65]
Waste mixed sludge from a WWTP	1.73 (0.01)%	78.6 (0.17)% TS	na	6.49	[66]
Wastewater treatment sludge from a WWTP	1.01%	0.66%	9.43 g/L	7.48	[65]

[1] Calculated; COD—chemical oxygen demand; na—not available; TS—total solids; VS—volatile solids; VSS—volatile suspended solids; WWTP—wastewater treatment plant.

Table 3. Characteristics of food wastes feedstocks for anaerobic digestion.

Feedstocks	TS	VS	COD	pH	Reference
Agro-food industry organic waste	72.1–209 g/kg	51.5–200.3 g/kg	90.5–342.8 g/kg	3.3–6.7	[67]
Banana waste	9.70–17.90% (fresh mass)	83.35–92.98% (dry mass)	na	na	[68]
Bovine slaughterhouse waste	25.6 (0.18)%	95.6 (0.04)% TS	na	6.14	[66]
Bread waste	67.4%	65.5%	na	na	[69]
Cocoa shell	89.9 ± 1.1%	82.3 ± 1.2%	na	na	[31]
Commercial food waste	7.7–92.7%TS/FM	90.6–100% VS/TS	na	na	[32]
Fish waste	31.4–38.5%	27.63–36.19%	na	na	[69]
Food and vegetable waste	70.5 ± 0.20%	89 ± 0.30% TS	na	na	[55]
Food residues	71.4–991.0 g/kg	59.8–988.8 g/kg	90.9–2880.0 g/kg	na	[33]
Food waste	24.1 wt.%	88.2% dry weight	na	na	[70]
Food waste	20.05%	19.21%	na	na	[11]
Food waste	29.4%	95.3% TS	na	4.1	[71]
Food waste	48.4 ± 2.7 g/L	27.9 ± 1.3 g/L	113.0 ± 2.8 g/L	4.6 ± 0.2	[54]
Food waste	111.8 (0.9) g/L	103.2 (0.9) g/L	144.3 (5.0) g/L	na	[72]
Food waste	13% *w*/*w*	11% *w*/*w*	na	na	[73]
Food waste from restaurant	174.12 ± 17.20 g/L	168.61 ± 18.46 g/L	187.20 ± 31.68 g/L	4.01 ± 0.01	[9]

Table 3. *Cont.*

Feedstocks	TS	VS	COD	pH	Reference
Fruit and vegetable waste	23.83 ± 0.13%	91.67 ± 0.12% of TS	na	na	[74]
Fruit and vegetable waste	144.81 ± 1.80 g/kg	133.18 ± 0.22 g/kg	na	4.24 ± 0.19	[35]
Fruit and vegetable waste	155.7 (0.5) g/kg wet	113.6 (0.4) g/kg wet	na	na	[36]
Meat processing waste	9.26%	7.07%	188.86 g/L	5.36	[65]
Meat-processing wastes	65–88%	65–86%	1774–1846 g/kg	na	[75]
Mixture of cooked food waste and raw vegetables	30.42 (1.79)%	94.52 (3.11)% TS	na	na	[28]
Municipal solid waste	351.4 g/kg	246.0 g/kg	332.5 g/kg	na	[29]
OFMSW	109.9 g/kg	105.1 g/kg	150 g/kg	na	[44]
OFMSW	23.3 ± 0.34%	20.2 ± 0.26%	210.667 ± 3.581 g/L	3.5 ± 0.04	[63]
OFMSW	461 g/kg	386 g/kg	468 g/kg	na	[76]
Organic waste from household	25.58 wt.%	23.94 wt.%	300.3 g/L	na	[26]
Slaughterhouse liquid waste	15.11% *w*/*w*	14.29% *w*/*w*	na	7.2	[77]
Solid fish waste	25–37%	0.737–0.851 g VS/g dry waste	1.126–1.423 g COD/g dry waste	na	[78]
Solid slaughterhouse wastes	27.9–65.2%	95.2–98.6%	na	na	[79]
Source-separated organic household waste	28–52%	76–94% TS	na	na	[80]
Source-separated organic household waste	24–86% ww	81–94% TS	na	na	[81]
Spent coffee grounds	493 ± 78 g/kg	484 ± 76 g/kg	na	6.2 ± 0.2	[37]
Totally cooked food waste	32.47 (1.41)%	95.28 (3.66)% TS	na	na	[28]
Untreated OFMSW	1.41% *w*/*w*	0.94% *w*/*w*	17.9 g/L	5.2	[77]
Waste coffee grounds	40.6 ± 0.3%	40.0 ± 0.3%	na	na	[31]
Wastes from a pig slaughterhouse	180.0–297.5 g/kg	170.2–256.4 g/kg	na	na	[82]
Wastes of an ice-cream processing plant	9.10 ± 0.36 g/L	9.27 ± 0.53 g/L	221 ± 16 g/L	4.39	[27]
Wastes of manufacturing chicken fat for marinades	289 ± 5 g/L	275 ± 4 g/L	648 ± 119 g/L	5.79	[27]
Wastes of manufacturing cranberry sauce	224 ± 6 g/L	225 ± 6 g/L	436 ± 46 g/L	2.85	[27]
Wastes of meatball fat from frozen food processing	144 ± 24 g/L	135 ± 23 g/L	148 ± 21 g/L	4.42	[27]
Whey from local dairies	6.63–7.44% *w*/*w*	5.64–6.73% *w*/*w*	81.8–105.0 g/L	5.5–5.8	[77]

COD—chemical oxygen demand; FM—fresh matter; na—not available; OFMSW—organic fraction of municipal solid waste; TS—total solids; VS—Volatile solids.

Table 4. Characteristics of energy crop feedstocks for anaerobic digestion.

Feedstocks	TS	VS	COD	pH	Reference
Alfalfa	91%	85.1%	na	na	[83]
Cañadú	917 ± 4 g/kg	862 ± 5 g/kg	981 ± 32 g/O_2 kg	na	[84]
Commercial hybrid cultivar PR87G57 (Nine S. bicolor varieties)	922 ± 4 g/kg	838 ± 5 g/kg	1026 ± 42 g/O_2 kg	na	[84]
Commercial hybrid cultivar PR88Y20 (Nine S. bicolor varieties)	917 ± 5 g/kg	809 ± 11 g/kg	1017 ± 65 g/O_2 kg	na	[84]
Crop waste	104.2 (0.8) g/kg wet	82.7 (0.5) g/kg wet	na	na	[36]
Fresh sugar beets	26.08 (0.38)%	92.11 (1.06)% TS	na	5.93 (0.07)	[85]
Grass	93%	81.0%	na	na	[83]
Maize Silage	31.66 ± 0.32%	95.51 ± 0.53% TS	na	na	[86]
Milho painzo	916 ± 1 g/kg	832 ± 4 g/kg	1062 ± 32 g/O_2 kg	na	[84]
Panizo	934 ± 3 g/kg	859 ± 6 g/kg	1092 ± 24 g/O_2 kg	na	[84]
Public genotype PR898012 (Nine S. bicolor varieties)	924 ± 2 g/kg	817 ± 2 g/kg	980 ± 21 g/O_2 kg	na	[84]
Red Clover	94%	84.2%	na	na	[83]
Reed Silage	62.85 ± 0.99%	91.16 ± 0.27% TS	na	na	[86]

Table 4. *Cont.*

Feedstocks	TS	VS	COD	pH	Reference
Silages of cup plant, Virginia mallow, reed canary grass, tall wheatgrass, wild plant mix, giant knotweed	21.1–39.9% FM	85.1–94.1% TS	na	na	[87]
Switchgrass Shawnee	938.12 (0.54) g/kg	824.31 (3.36) g/kg	na	na	[88]
Trigomillo	927 ± 2 g/kg	852 ± 5 g/kg	1079 ± 27 g/O_2 kg	na	[84]
Wheat straw	na	0.93 ± 0.003 gOM/gDM	na	na	[89]
Wheat straw	895–924 g/kg	821–846 g/kg	1075–1089 g/kg	na	[90]
Wheat straw	922 ± 2 g TS/kg	92% VS/TS	1078 ± 8 g TCOD/kg	na	[91]
Wheat straw	94.0%	86.8% (wet weight)	na	na	[92]
Zahina	916 ± 5 g/kg	829 ± 8 g/kg	1018 ± 26 g/O_2 kg	na	[84]
Zahina gigante	918 ± 4 g/kg	841 ± 5 g/kg	1702 ± 124 g/O_2 kg	na	[84]
Water hyacinth	8.24 ± 0.36%	76.54± 0.30% of TS	na	na	[74]
Blue algae	4.13 ± 0.18%	86.68 ± 1.47% TS	na	na	[47]
Invasive aquatic plants	51.8–148.8 g/kg	37.7–74.2 g/kg	27.8–49.5 g/kg	na	[33]
Paragrass	29.37 ± 0.27% (wet weight)	25.80 ± 0.22% (wet weight)	na	6.67	[92]
Grass silage	292.7 ± 3.4 g/kg	268.4 ± 2.8 g/kg	na	na	[43]

COD—chemical oxygen demand; DM—dried matter; FM—fresh matter; na—not available; OM—organic matter; TS—total solids; VS—Volatile solids.

Table 5. Characteristics of other organic feedstocks for anaerobic digestion.

Feedstocks	TS	VS	COD	pH	Reference
Alcoholic beverage production wastes	6.06–44.1%	5.55–38.3%	na	na	[93]
Bamboo waste	93.3–94.5%	77.3–90.0%	902 g/L	na	[94]
Brewery grain waste	24.2%	23.0%	na	na	[69]
Chicken feather waste	100 ± 0.5%	99 ± 1.4%	1408 ± 59 g/kg	na	[41]
Condensate water from factory	0.018% *w*/*w*	na	4.15 g/L	3.5	[77]
Corn Stover	86.02 ± 0.91%	80.89 ±0.67%	na	na	[34]
Grain mill residues	874–912 g/kg	896–940 g/kg TS	na	4.1–4.5	[30]
Grape Marc	38.7 ± 1.51%	24.1 ± 0.54%	223 ± 16.3 g/L	9.19 ± 0.01	[95]
Grease trap waste	16.28%	13.89%	245.75 g/L	5.23	[65]
Grease waste	673 ± 4.5 g/kg	645 ± 1.5 g/kg	na	na	[96]
Grease waste from a DAF tank from WWTP	505.2 g/kg	468.2 g/kg	648.3 g/kg	na	[29]
Landfill leachate	2.45 (0.05) g/L	2.02 (0.04) g/L	2.52 g/L [1]	7.00 (0.05)	[28]
Low-organic waste of landfills	18–90%, kg/kg waste, ww	7–70%, kg/kg waste, ww	na	na	[97]
Olive oil waste (olive pomace)	331.33 ± 6.81 g/L	305.60 ± 6.18 g/L	na	6.75 ± 0.05	[98]
Rice straw	92.59%	70.37%	na	6.22	[62]
Rice straw	92.6 ± 0.31%	70.4 ± 0.22%	na	6.2 ± 0.02	[63]
Sherry-wine distillery wastewater	1.47 ± 0.11 g/L	1.06 ± 0.09 g/L	24.6 ± 2.2 g/L	6.4 ± 0.2	[59]
Sunflower oil cake	93.0 (±0.1)%	93.0 (±0.1)% (dry basis)	1.24 (±0.02) g O_2/g TS dry basis	na	[99]

Table 5. *Cont.*

Feedstocks	TS	VS	COD	pH	Reference
Two-phase olive mill solid waste	265.0 ± 2.6 g/kg	228.4 ± 2.3 g/kg	331.1 ± 0.7 g O_2/kg	4.9 ± 0.2	[100]
Two-phase olive mill solid waste	265 ± 3 g/kg	228 ± 2 g/kg	331 ± 1 g O_2/kg	4.9 ± 0.2	[101]
Winery solid	87.93%	80.05%	na	4.53	[102]

[1] calculated; COD—chemical oxygen demand; DAF—dissolved air flotation tank; na—not available; TS—total solids; VS—Volatile solids; WWTP—wastewater treatment plant.

The comparison of the several feedstocks becomes difficult due to the variability of the substrates and to the use of different units.

For the sludge, food waste, energy crops, and other organic feedstocks the ranges for each parameter are presented in the lists below.

The sludge feedstocks (Table 2) present the following parameters, expressed with different units:

- TS: 3.67 to 106.1 g/L, 47.3 to 71.2 g/kg, 0.4 to 19.17%;
- VS: 2.04 to 60.1 g/L, 40.5 to 54.9 g/kg, 0.66 to 94.7%;
- COD: 5 to 406 g/L, 83.9 g/kg;
- pH: 5.0 to 7.6.

The food waste feedstocks (Table 3) present the following parameters, expressed with different units:

- TS: 9.10 to 289 g/L; 71.4 to 991.0 g/kg, 0.97 to 89.9%;
- VS: 9.27 to 275 g/L, 51.2 to 988.8 g/kg, 0.94 to 100%;
- COD: 17.9 to 648 g/L, 90.5 to 2880.0 g/kg;
- pH: 2.85 to 7.2.

The energy crop feedstocks (Table 4) present the following parameters, expressed with different units:

- TS: 51.8 to 938.12 g/kg, 4.13 to 94%;
- VS: 37.7 to 862 g/kg, 25.8 to 95.51%;
- COD: 27.8 to 1702 g/kg;
- pH: 5.93 to 6.67.

The other organic feedstocks (Table 5) present the following parameters, expressed with different units:

- TS: 1.47 to 331.33 g/L, 265.0 to 912 g/kg, 0.018 to 100%;
- VS: 1.06 to 305.6 g/L, 228 to 940 g/kg, 5.55 to 99%;
- COD: 2.52 to 902 g/L, 331 to 1408 g/kg;
- pH: 3.5 to 9.19.

pH is the parameter that shows the smallest variation, ranging from 2.85 in food waste feedstocks to 9.19 in other organic feedstocks. According to Cecchi et al. (2002) [103], the AD process is stable in the pH range of 6.5 to 7.5. Therefore, most of the feedstocks need to be neutralized with the addition of a base or an acid or mixed with feedstocks from different sources to achieve the suitable pH.

The TS and VS contents of the feedstocks shown in Tables 1–5 vary significantly, from 0.018 to 100% and from 0.7 to 100%, respectively, when compared to the database presented by Moretta et al. (2022) [2] (TS from 6.02 to 93.45% and VS from 64.69 to 98.65%). This fact can probably be explained by the feedstock's variability, but also by the different analytical methods of determination of solids.

The COD determination of solid or liquid feedstocks with high content of suspended solids can be made by several analytical methods (e.g., open and closed reflux), which may influence the results. In this present review, the feedstocks presented different COD values ranging from 9.43 g/L to 902 g/L and from 27.8 g/kg to 2880.0 g/kg.

The high variable composition of feedstocks in terms of pH, solids, and COD implies several challenges for their anaerobic digestion such as low biodegradability, toxicity, and inhibition.

Uddin and Wright (2021) [104] pointed out that economic viability is a major obstacle for the application of some feedstocks in the AD process.

3. BMP Assay Evolution

3.1. Anaerobic Digestion and BMP Publications

The AD process of different substrates has received increasing attention in the recent years because it can be considered an economical and environmentally friendly technology for treating several organic wastes [1]. In effect, the energy-rich biogas produced by AD can be used as renewable energy, and the digestate can be applied in agriculture.

In the last four decades, BMP assays have been widely applied to estimate the methane yield and the biodegradability of individual organic substrates or those co-digested by the AD process [33,34,55,63,105–108]. In 2012, Raposo et al. (2012) [109] reported that the BMP tests have increased, which is reflected in the numerous research papers. Nevertheless, the groundwork for future studies began as early as 1979, with the study carried out by Owen et al. (1979) [105].

To present the evolution of the number of publications on AD and BMP assays (Figure 3), a search was carried out with the research engine from Online Knowledge Library (B-on) covering non-peer-reviewed as well as peer-reviewed publications from 2008 to 2023 (through mid-March).

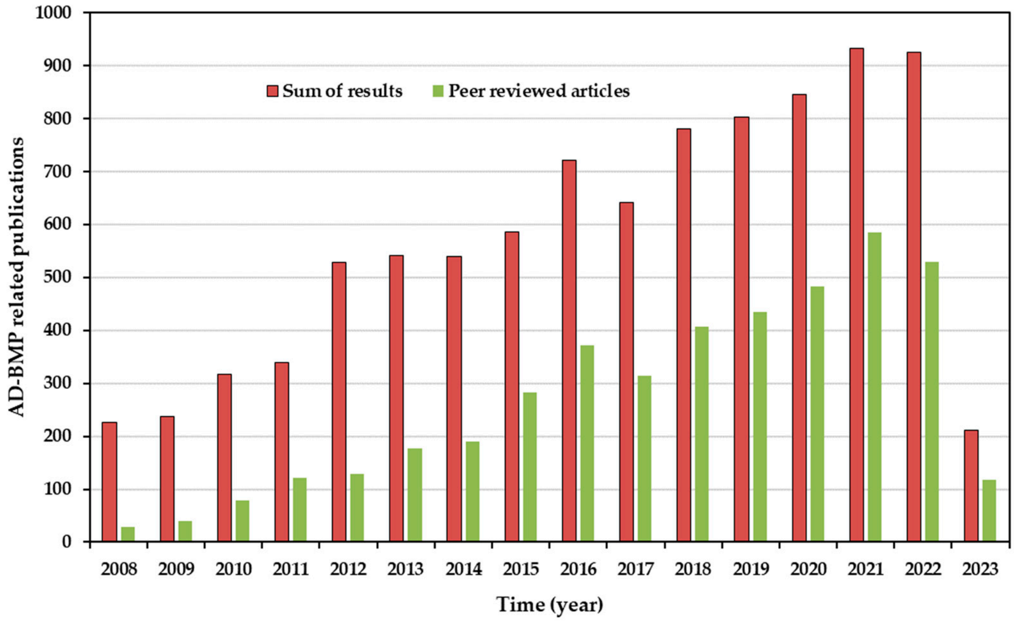

Figure 3. Number of publications with the keywords "anaerobic digestion" and "BMP" from 2008 to 2023.

The interest in BMP assays is evidenced by the number of publications which increased significantly over the years, especially after 2011, with more than 120 papers and a total of around 340 publications, as shown in Figure 3. The highest number of paper publications was reached in 2021, with 585 peer-reviewed papers, and a total of 932 publications. A slight decrease was observed in 2022, probably due to the COVID pandemic. Based on the results for the first three months, it is expected that 2023 will present similar values to those of 2020.

3.2. BMP Methodologies

BMP assays employed to evaluate the suitable feedstocks for the AD process to produce biogas are an essential basis to assess the benefit of AD and to optimize process design. The BMP provides a vital reference index for stable and reliable biogas production [34,110].

Usually, the BMP assays consist of mixing substrate and an inoculum and measuring methane production during a certain time.

The basis for the BMP assays and the model for future studies were laid out by Owen et al. (1979) [105]. This was one of the first BMP studies; it aimed to determine the biodegradability of various organic substrates. The methodology presented by Owen et al. (1979) [105] consists of the incubation of substrate samples with inoculum (20% by volume to defined media) and specific nutrient medium for a certain period of around 30 days. The mixture obtained is placed in 250 mL serum vials and flushed with a mixture of CO_2 and N_2 (30:70 volume ratio) at 0.5 L/min to initiate anaerobic conditions. During the incubation time, the biogas is measured with a volumetric syringe and analysed by gas chromatography (GC) [4].

Hansen et al. (2004) [111] adopted and modified the existing procedures, especially the one proposed by Angelidaki and Ahring (1997) [112], to determine methane potential of more than 100 solid waste samples.

Angelidaki and Sanders (2004) [113] reviewed proposed methods for determining the anaerobic biodegradability of macropollutants. In this study, it was observed that due to the complexity of the anaerobic process, the BMP assays can lead to significant uncertainties. Therefore, it is important that the procedure ensures optimal conditions for anaerobic growth, and that the results are carefully evaluated.

The Association of German Engineers published the first version of the detailed technical guideline VDI 4630 in 2006, presenting rules and specifications for batch and continuous tests. In November 2016, a new version of this standard was published [17].

In 2009, Angelidaki et al. (2009) [1] presented guidelines to define a standard protocol for BMP assays applied to solid organic wastes and energy crops such as the definition of common units to be used in anaerobic assays.

Holliger et al. (2016) [23] reported that the presentations made during a workshop in Leysin, Switzerland, in June 2015 clearly indicated the need to standardize the BMP assays. This paper mentions the need for mandatory elements, e.g., the minimal number of replicates to carry out blank and positive control assays, test duration, and detail the calculation carried out. Some recommendations concerning the inoculum characteristics, substrate preparation, test setup, and data analysis are also offered. Between 2016 and 2017, an inter-laboratory study was carried out to assess the guidelines presented in 2016. The results showed that only 26.8% of 62 BMP assays could be validated considering the reproducibility criteria, which corresponds to a 73.2% of rejected results.

In April 2018, a workshop was held in Freising, Germany to make the BMP assays more reliable and reproducible. A second inter-laboratory study was performed in 2018 to enable the application of the refined validation criteria for BMP assays. The results of this inter-laboratory study showed that the rejected results dropped to 55%.

After all the attempts to create a standard and to develop a guidance on BMP measurement and data processing accessible to the entire scientific community, a website was created: https://www.dbfz.de/en/BMP (accessed on 13 December 2022) [114]. On this website, it is possible to find the required components for any BMP protocol, as well as the validation criteria [115]. Specific method calculations are described for each BMP measurement method: volumetric (document 201) [116], manometric (document 202) [117], gravimetric (document 203) [118], and gas density (document 204) [119].

Despite several attempts to standardize the BMP assay procedure, a recent study mentions that even in the peer-reviewed publications, results are not always used appropriately [120]. In this study, several limitations of BMP assays were presented, such as not providing information about the chronic toxicity of a substance and not allowing to obtain the methane yield and the organic load rate in a continuous system generally used on an industrial scale. Also, the synergies or antagonisms occurring in the co-digestion and the long-term effects of nutrients or trace elements cannot be evaluated because the BMP assay has a different feeding when compared with the continuous process, which allows a typically high amount of inoculum in the batch test.

In 2020, another study carried out by Koch et al. (2020) [3] mentioned the importance of using a positive control in BMP assays.

4. BMP Experimental Conditions and Results

The experimental conditions for BMP assays can be divided into operational conditions and gas measurement systems. Operational conditions include physical and chemical conditions and the inoculum/substrate (I/S) ratio [106,109].

In the BMP assays, there are several physical conditions that affect the results, namely reactor material and capacity (total and working volume), incubation temperature (mesophilic or thermophilic), stirring (manual, automatic, and continuous), and incubation time (pre-incubation and assay duration) [106].

The chemical incubation's conditions, such as headspace gas, pH, alkalinity, and mineral medium, can also affect the results.

The BMP experimental conditions and results for several feedstocks from 2011 to 2023 are presented in Table 6. The experimental conditions considered are: substrate and inoculum sources, reactor capacity (total and working volume), headspace, I/S ratio, temperature, incubation time, and methane production.

The substrate source is mainly contained in real conditions, such as farms, industries, and WWTP. It is important to ensure that the material collected for BMP assays is representative of organic matter to be digested at full scale. Therefore, the sampling procedure is an important step.

Table 6 shows that the inoculum may come from various sources, but mostly from anaerobic digestors in WWTP or animal farms. Holliger et al. (2016) [23] mentioned a quality criterion for inoculum with the following characteristics: $7.0 < pH < 8.5$, VFA < 1.0 gCH_3COOH/L; $NH_4^+ < 2.5$ $gN\text{-}NH_4/L$; and alkalinity > 3 $gCaCO_3/L$.

Pretreatments applied to the substrates can include pH adjustment, blending, thermal treatment at different temperatures (20 to 200 °C), and chemical treatment (acid, base, enzyme, and ozone). The most usual pretreatments are thermal and chemical with a base (NaOH).

In general, the reactor material for BMP tests is glass bottles [111], but other materials can be found, such as heavy-duty polypropylene [87].

Concerning the reactor capacity, the total volume ranges from 60 mL [57] to above 3000 mL [51] with different working volumes even for a similar reactor volume. For example, Raposo et al. (2011) [106] reported a total volume of 1000 mL with working volumes of 200, 700, and 750 mL. Usually, the reactor volume depends on the substrate homogeneity [1]. Holliger et al. (2016) [23] reported that the reactor can be smaller for homogenous substrates (≈100 mL), large volumes are adequate for heterogenous substrates (500 to 2000 mL) and the working volume ranges from 400 to 500 mL.

According to Holliger et al. (2016) [23], the headspace depends on biogas measurement method (volumetric or manometric), ranging from 500 to 1000 mL. In the present review, it was found that the headspace ranges from 10 mL [84] to 1400 mL [67].

The temperature incubation for BMP tests is mesophilic and thermophilic. The mesophilic temperature ranges from 35 °C to 39 °C, but the most used values are 35 °C and 37 °C. In Table 6, only one study used an incubation temperature of 14 °C [42]. The thermophilic temperature ranges from 45 °C to 65 °C, with 55 °C being the most frequent value. Holliger et al. (2016) [23] mentioned that typical incubation temperatures are 37 °C and 55 °C, with a maximum variation of ±2 °C.

Regarding the I/S ratio, a very large range is presented, expressed with different basis, like g VSS/g COD, g VS/g CODsoluble+colloidal, g VS/g CODtotal, g SS/mg COD, and g COD/g VSS. The I/S ratio presents values such as 0.5, 1, 1.33, 2 and 4.00 g VS/g VS, the 2 gVS/gVS being the most used. Holliger et al. (2016) [23] recommended the I/S ratio between two and four, VS-based.

In the present review, the incubation times ranges from 7 days [98] to 216 days [42], but a higher range (7 to 365 days) was referred to by Raposo et al. (2012) [109]. The most used incubation time is around 30 days. According to Holliger et al. (2021) [115,121], BMP incubation time is achieved when daily methane production during three consecutive days is less than 1% of the accumulated volume of methane after the subtraction of the inoculum biogas production.

There are several methods to measure biogas production in the BMP assays. In recent years, some commercial automated systems have been developed, typically volumetric, with less labour but with high initial costs. However, recent studies [35,36] used the manual methods based on volumetric and manometric principles. There are three main methods for volume determination: pressure transducer, volume displacement and syringe. Usually, the biogas methane contents are determined by gas chromatography (GC) with a thermal conductivity detector (TCD).

The results of BMP assays are presented with the specific methane production of the feedstocks assessed. To achieve these results, it is necessary to perform several calculations, like the volume of methane produced at standard temperature and pressure conditions (1 atm and 273.15 K), but there are some studies with different temperatures, like the one presented by Suhr et al. (2015) [46] in which the authors use a temperature of 20 °C. To obtain the correct results, it is necessary to discount the biogas production of the inoculum (blank tests) from the substrate biogas production.

Non-standard procedures continued to be applied for BMP tests up to 2023, resulting in the lack of comparable values due to different experimental conditions, procedures, and equipment. The same was found by Filer et al. (2019) [122]. However, an effort has been made to minimize or even eliminate systematic errors, with a positive evolution in the last 10 years.

Table 6. BMP experimental conditions for different anaerobic feedstocks and methane production.

Reference	Substrate Source	Inoculum Source	Pretreatment	Total and Working Volume (mL)	Headspace (mL)	I/S	T (°C)	Incubation Time (d)	Gas Measurement	Methane Production
[33]	Raw manures, food residues, invasive aquatic plants, others (switchgrass, corn silage, corn leachate, mouthwash, suspended FOG and settled FOG).	Farm-based completely mixed AR	Mixed and blended	250 (na)	na	>0.5 gVS/gVS	35	40	Pressure transducers. GC-TCD	106.5–648.5 mL CH_4/g VS_{add}
[30]	Silage and hay, animal slurry, agro-industrial waste.	AR of a WWTP	na	575 (200)	375	150 mL/0.3 g TS	36	42–78	Pressure transducer. GC	286–319 L CH_4/kgVS_{add}; 238–317 L CH_4/kgVS_{add} 272–714 L CH_4/kgVS_{add}
[123]	MSW, raw wastes (papers, vegetables and a waste built by mixing some of the simple wastes) and lignocellulosic green wastes.	Active anaerobic sludge	na	600 (na)	na	0.5 gVS/gVS	35	35	Every 2 days with Micro-GC	MSW: 87–355 mL CH_4/g VS; Raw: 20–400 mL CH_4/g VS
[124]	Thickened sludge samples from WWTP.	Digested sludge from digester-WWTP	na	1000 (na)	na	100 g/500 g	MC	21	Liquid displacement. GC-TCD	25–456.3 mL CH_4/g ODM
[50]	Aerobic Granular sludge.	na	Thermal (60–210 °C)	570 (400)	170	1 g VS/gVS	35	26	Pressure transducer and GC	169–404 mL-CH_4/g-VS_{fed}
[67]	Wastes from agro-food industries (dairy, cider production, cattle farming).	Anaerobic sludge from a municipal WWTP	na	2000 (600)	1400	0.67, 1, 1.33, 2 and 4.00 gVS/gVS	35	55	Pressure transmitter. GC-TCD	202–549 mL STP CH_4/gVS waste

Table 6. *Cont.*

Reference	Substrate Source	Inoculum Source	Pretreatment	Total and Working Volume (mL)	Headspace (mL)	I/S	T (°C)	Incubation Time (d)	Gas Measurement	Methane Production
[54]	Primary sludge of WWPT and OFMSW.	Primary mesophilic AR at a WWTP, Mesophilic AR treating SSO, Mesophilic AR treating primary and secondary wastewater	na	260 (200)	60	0.25, 0.5, 1, 2 and 4 g VSS/g COD	37	App. 28	Glass syringes 5–100 mL. GC-TCD	Primary sludge: 221–283 mL CH_4/g VSSsub; OFMSW: 440–1400 mL CH_4/g VSSsub
[125]	Herbaceous plants and no herbaceous.	Biogas plant 37 °C (80% animal slurry + 20% organic industrial waste)	na	na (1000)	na	3:1 TS	37	App. 60	VDI and GC-TCD	104–388.9 CH_4 N L/kg VS
[99]	Sunflower oil cake sample from factory.	Granular sludge from an industrial AR 35 °C	Chemical and Thermochemical (75 °C)	na (250)	na	2 gVS/2.5 gCOD	35	7 to 10	Liquid displace (2 N NaOH)	0–273 mL CH_4/gCOD$_{add}$
[41]	Chicken feather waste and poultry litter from industry.	Anaerobic suspend sludge-municipal AR. Anaerobic granular sludge-brewery industry	Thermochemical (20–90 °C)	na (50)	na	0.66, 0.71, 0.76 and 1.32 g VS/g VS	37 and 65 (BA)	80	GC-FID	45–123 L CH_4/kg VS$_{add}$
[78]	Solid fish waste-tuna, sardine, mackerel, and needle fish.	Suspended sludge–urban WWTP. Granular sludge-brewery industry	na	na (na)	na	0.15–0.91 g VS/g VS	37	60–80	Pressure transducer. GC-FID	0.04–0.35 L CH_4/g VS$_{add}$;
[60]	Thickened primary and secondary sludge from a municipal activated sludge facility.	Anaerobic Granular sludge from an UASB treating industrial waste	na	250 (150)	100	1/1, 1/3 and 1/8	35	21	Glass sy-ringes. GC-TCD	21.93–76.27 mL CH_4/g VS$_{added}$

Table 6. *Cont.*

Reference	Substrate Source	Inoculum Source	Pretreatment	Total and Working Volume (mL)	Headspace (mL)	I/S	T (°C)	Incubation Time (d)	Gas Measurement	Methane Production
[75]	Greaves and rinds from a meat-processing plant.	Granular sludge from a brewery WWTP	NaOH, NaOH+ temperature, NaOH+ autoclave, temperature, enzyme and autoclave +enzyme (25–121 °C)	160 (na)	80	4 g VS/g CODsoluble + colloidal and 1.3–3.3 g VS/g CODtotal; untreated: 4 g VS/g CODtotal	37	50–110	GC	305–919 LCH_4 STP/kgVSsub
[25]	Dry (non-treated) and steam-exploded wheat straw, cattle manure from a farm.	Mesophilic biogas plant with SSMHW and grass silage	na	1120 (700)	420	2 gVS/gVS	na	25 and 60	GC	0.15–0.33 N L CH_4/g VS
[51]	Dewatered sludge from a WWTP.	Digested sludge from mesophilic AR-WWTP	Mild thermal (50–120 °C)	na (3000)	na	0.0014–0.022 gSS/mg COD	na	30	Liquid displacement. GC	67.7–144.7 $mLCH_4$/g VS_{add} (20 d)
[43]	Grass silage;,fresh slurry-dairy farm.	2 digesters (FW and mix of poultry/CM)	na	500 (400)	100	2:1	37	30	Liquid displacement	239–400 L CH_4/kg VS
[47]	Blue algae and swine manure.	Swine manure. Granular sludge	na	500 (400)	100	0.5, 1.0, 2.0 and 3.0 gVS/gVS	35	22	Alkali solution and gas flow meter. GC-TCD	32.8–212.7 mL CH_4/g VS
[82]	Wastes from a pig slaughterhouse.	Inoculum from a farm-scale biogas plant that digests piggery slurry	na	160 (60)	100	0.67, 1, 2 and 10 gVS/gVS	38	76	Liquide displacement (acidified brine solution). GC-TCD	0.357–1.076 N m^3/kg-VS_{added}
[94]	Bamboo waste from a chopstick production factory.	Anaerobic sludge from a mesophilic AR feed with dewatered sewage sludge from WWTP	Acid, alkaline, enzyme and alkaline aided enzyme	na (na)	na	2	37	30–33	Automatic equipment	25–303.3 mL CH_4/g VS

Table 6. *Cont.*

Reference	Substrate Source	Inoculum Source	Pretreatment	Total and Working Volume (mL)	Headspace (mL)	I/S	T (°C)	Incubation Time (d)	Gas Measurement	Methane Production
[29]	Biological sludge thickened—WWTP, OFMSW—synthetic mixture of foods, MSW sorted from WWTP, grease waste from DAF-WWTP, spent grain from brewery industry, CM from slaughterhouse.	WWTP mesophilic digested sludge	Thermal hydrolysis (120–170 °C)	300 (na)	na	1:1 gVS/gVS	35	App. 40	Pressure meter. GC	184–524 $mLCH_4/gVS_{in}$
[45]	Pig slurry.	Pilot sludge digester anaerobic treating activated sludge	Thermal steam (120–180 °C)	300 (110)	190 [1]	2 gVS/VS	35.1	App. 40	Manually by a pressure transmitter. GC-TCD	159–329 mL CH_4/gVS_{fed}
[11]	FW and straw shredded to a small size.	Anaerobic granular sludge-UASB reactor treating starch processing wastewater at 35 °C	na	1000 (600)	400	600 mL/12 g VS	35	8	Liquid displacement. GC-TCD	0.157–0.392 m^3 CH_4/kg VS
[46]	Solid waste produced in RAS.	Digested CM	na	540 (200)	340	4, 8 and 16 g/g [1]	35	24	GC	318 ± 29 mL CH_4/gTVS
[92]	Variety of paragrass samples.	Mesophilic anaerobic sludge from a domestic WWTP.	na	100 (60)	40	1 g VS/g VS	32–35	80	Glass syringes. GC-TCD	277 and 316 NmL/g VS
[52]	Grass silage, dairy slurry.	Pre-incubation at 40 °C for 3 d	na	500 (400)	100	2:1 gVS/gVS	37	30	Liquid displacement (3 M NaOH) GC-TCD	239–400 NL CH_4/g VS
[58]	Secondary sewage sludge—WWTP.	Anaerobically digested sludge–mesophilic AR fed with mixed sludge from the local WWTP	Thermal hydrolysis and advanced thermal hydrolysis (H_2O_2) (90–170 °C)	160 (na)	60	2	35	28	Periodically with a manual pressure transmitter and GC-TCD	227–327 $mLCH_4/gVS_{fed}$

Table 6. *Cont.*

Reference	Substrate Source	Inoculum Source	Pretreatment	Total and Working Volume (mL)	Headspace (mL)	I/S	T (°C)	Incubation Time (d)	Gas Measurement	Methane Production
[126]	Composite slurry samples.	Digestate from an AR treating SSOFMSW, manure and industrial waste	na	1000 (na)	700 [1]	2/1 VS	37	35	Gas tight syringe and GC-TCD	445–568 m^3 N CH_4/ton VS introduced
[56]	WWTP that treats pulp and paper industry wastewater.	Mesophilic digested municipal sewage sludge WWTP and digestate from a CSTR	Thermal (80–134 °C)	120 (na)	60	2 VS/VS	35	35	Water displacement and GC-FID	40–160 NL CH_4/kg VS
[42]	Unseparated manure and separated manure.	Mesophilic digester treating the separated cow manure	na	250 (na)	120	1 VS unseparated manure; 2 VS separated manure	14 and 24	216	Glass syringe (50 mL). GC-FID	107–479 $mLCH_4$/g VS_{added}
[53]	Pharmaceutical sludge from a pharmaceutical factory	Inoculum sludge-digester from faecal sludge	na	1000 (na)	na	0, 0.65, 2.58 and 10.32 TS	37	App. 55	Water displacement and Biogas Analyser (daily)	6.98–499.46 mL biogas/g TS pharmaceutical sludge
[44]	Dairy manure, solid fraction, liquid fraction (LF).	Screened LF digested at 50 °C	na	500 (na)	na	1 gVS/gVS	35 (manure + LF). 50 (SF)	80	Pressure measurement and GC-TCD	298 L CH_4/kgVS, 265 L CH_4/kgVS, 343 L CH_4/kgVS.
[98]	Olive pomace	Dairy manure	NaOH, Salts, US, US + salts	250 (na)	na	na	30	App. 60	Liquid displacement. GC	2–193 L CH_4/kgVS0
[32]	Commercial food waste (FW), dairy manure (DM) slurry.	Post solid separated effluent –Mesophilic anaerobic digestion with co-digested DM with assorted FW	na	500 (300 to 400)	100 to 200	2 gVS/gVS	37	33	Continuously (Bioprocess Control) and GC-TCD	165–496 mL CH_4/g VS_{add}
[68]	Hay (control and standard substrate), peel, stalk, flesh and unpeeled banana.	na	na	2000 (na)	na	0.7 VS	37	35	Volumetric method. Methane analyser + infrared sensor	0.256–0.367 m^3 CH_4/kg VS
[81]	Source-separated organic household waste.	Collected from a WWTP	na	1000 (na)	Adjusted to 70%	2 gVS/gVS	37	45	GC-FID	202–572 mL CH_4/g $VS_{subtrate}$

Table 6. *Cont.*

Reference	Substrate Source	Inoculum Source	Pretreatment	Total and Working Volume (mL)	Headspace (mL)	I/S	T (°C)	Incubation Time (d)	Gas Measurement	Methane Production
[62]	TWAS from wastewater treatment plant and RS.	WWTP	Thermal and thermo-NaOH for TWAS (70–90 °C). NaOH and H_2O_2 for RS	250 (na)	70	0.5 TS	37	50	Liquid displacement. GC-TCD	184.63–401.89 mL_{biogas}/gVS_{added}
[70]	Food waste from a canteen.	Anaerobic sludge-up-flow AR of a paper mill	Storage as a pretreatment. FW separately stored for 0–12 d	1000 (na)	na	2:1 VS	35	21/60	Liquid Displacement (3 mol/L NaOH).	311–571 mL CH_4/g-VS_{added}; 285–696 mL CH_4/g-VS_{added}
[101]	Two-phase OMSW or alperujo.	Full-scale mesophilic AR treating brewery wastewater	Steam-explosion (200 °C). Afterwards a LF and a SF obtained	na (250)	na	2 VS	35	23	Liquid displacement (3N NaOH)	(LF) 589 ± 42 mL CH_4/g VS_{added}; (SF) 263 ± 1 mL CH_4/g VS_{added}; (Untreated) 366 ± 4 mL CH_4/g VS_{added}
[100]	The two-phase OMSW used was collected from the Experimental Olive Oil Factory	Industrial AR treating brewery wastewater 35 °C	Thermal (100–180 °C)	na (250)	na	2 VS	35	Period of c.a. 20	Liquid displacement (3N NaOH)	373–392 mL CH_4/g VS_{added}
[74]	Water hyacinth (WH) was harvested, fruit and vegetable waste (FVW) from typical market.	Mesophilic anaerobic sewage sludge—UASB treating domestic wastewater	na	500 (na)	100	na	37	60	Liquid displacement. GC-TCD	0.114 m^3 biogas/kg VS_{added} (WH); 0.141 m^3 biogas/kgVS_{added} (WH + FVW)
[57]	DAF sludge and WAS collected from refinery	Mesophilic AR at a municipal WWTP	Ozonation in a bubble column setup	60 (na)	na	DAF 2–100 gVS/gVSDAF; 5 gVS/gVSWAS	MC	30–50	na	80–160 $L_{biogas}/kgCOD_{added}$
[79]	Selected solid waste fractions from cattle, pig, and chicken slaughtering facilities.	Granular mesophilic inoculum from a mesophilic UASB reactor treating dairy processing waste	Pasteurisation	1000 (na)	100	2 VS	36–39	30–50	Liquid displacement (alkaline solution)	465.34–515.47 $mLCH_4$/gVS (UP); 501.13–650.92 $mLCH_4$/gVS (P)
[55]	Primary sludge from WWTP, fruit and vegetable waste.	Fresh cow manure, activated sludge from WWTP, excess sludge from WWTP	Drying and Grinding	500 (400)	100	2.0	37	30	Liquid dis-placement (3M NaOH)	0–295 L/g VS $_{added}$

Table 6. *Cont.*

Reference	Substrate Source	Inoculum Source	Pretreatment	Total and Working Volume (mL)	Headspace (mL)	I/S	T (°C)	Incubation Time (d)	Gas Measurement	Methane Production
[34]	Corn stover from cornfield, fresh dairy manure from a cooperative, fresh goat manure from agricultural university, fresh swine manure from industry.	From mesophilic biogas digester	Crushing, sieving and drying	500 (350)	150	1	37	30	Drainage method	176.95–332.19 mL/gVS
[61]	Thickened sludge from a WWTP.	Anaerobically digested sludge	Thermal Hydrolysis (TH)	135 (100)	35	2 g VS/g VS	35.0	28	Liquid dis-placement	TH: 305–359 mL biogas/gVS Raw substrate: 226 ± 39 mL biogas/gVS
[77]	Cheese whey (CW) samples from dairy industry, slaughterhouse liquid waste (SLW), condensate water from factory (CWT) OFMSW.	Granular sludge from UASB bioreactor from WWTP	Percolation bed for OFMSW	500 (na)	na	2 gVS/gVS For condensate water, 0.52 (tCOD)	35	25	(NaOH+ tymolphtalein).	CW: 22.8–36.3 L CH_4/kg COD $_{add}$ SLW: 74.8 L CH_4/kg COD $_{add}$ CWT: 147.5 L CH_4/kg COD $_{add}$ OFMSW: 218.9–221.8 L CH_4/kg COD $_{add}$
[9]	Food waste.	AR for WWTP and enriched with pig manure suspension	Blending and grinding	500 (400)	100	1	35	32	GC	38.56–65.91 NmLCH_4/g TVS
[35]	Fruit and vegetable waste, dry cow manure, liquid poultry manure.	Sludge from AR	na	250 (120)	130	1	37	50	Liquid displacement (NaOH 10%, *w*/*v*)	315–650 mLCH_4/g VS
[36]	Fruit + vegetable waste from market, crop (corn stalks, wheat straw) from research farm, fresh buffalo manure from research farm.	AR of poultry manure at 35 °C	Disinfection, removal of un-biodegradable matter, concentration of organic matter, and feed preparation	1000 (500)	500	2	35	60	Liqui displacement (NaOH). Portable biogas analyser	191–155 mL CH_4/g VS

Table 6. *Cont.*

Reference	Substrate Source	Inoculum Source	Pretreatment	Total and Working Volume (mL)	Headspace (mL)	I/S	T (°C)	Incubation Time (d)	Gas Measurement	Methane Production
[48]	Swine manure, crude glycerol used was a by-product of the biodiesel production from butchery waste.	2 bench-scale digesters operated with swine manure (37 °C)	na	320 (na)	na	4:1 2:1 1:1	37	30	GC-TCD	544 ± 29 mL CH_4/g VS
[39]	Waste cooking oil (palm and sunflower oils) (WCO), fresh pig slurry from farm (PS), phosphate-based basal medium recommended for the growth of Methanosarcinaspp (HM).	Digestate of pig slurry	Cooking oil 400 rpm (10 min)	na (118.5)	na	0.34 and 0.44	35	≈84	Syringe method. GC-TCD	WCO + HM-922 (17.9) NmL CH_4/gVS WCO + PS-811 (26.5) NmL CH_4/gVS PS-333 (12.5) NmL CH_4/gVS
[37]	Spent coffee grounds from canteen, liquid pig manure from a farm.	AR of the sewage treatment plant	na	120 (na)	na	1:1 and 1:2	37	≈70	GC-TCD	1:1-323 ± 29 mL/g VS 1:2-357 ± 34 mL/g VS
[40]	Pig slurry from a farm.	Agro-industrial waste biogas plant	na	560 (448)	112	2.8 (T1) and 1.6 (T2) g COD/g VSS	35	50	Manometric method. GC	T1—0.25 ± 0.05 L CH_4/g VS_{add} T2—0.21 ± 0.02 L CH_4/g VS_{add}
[65]	Waste activated sludge from WWTP, grease trap waste, wastewater treatment sludge from WWTP, meat processing waste.	Effluent from AR of WWTP	na	500 (na)	na	4:1 gVs/gVS	37	35	Water displacement. (20 g/L KOH)	121–980 mLCH_4/gVS

Table 6. *Cont.*

Reference	Substrate Source	Inoculum Source	Pretreatment	Total and Working Volume (mL)	Headspace (mL)	I/S	T (°C)	Incubation Time (d)	Gas Measurement	Methane Production
[28]	Sewage sludge from WWTP, FW1—cooked food waste, FW2—cooked food waste (80%) + raw vegetables (20%).	Mesophilic inoculum from WWTP, thermophilic inoculum from a lab scale semi-continuous reactor	Sludge–thermal or ultrasonic Food waste crushed + water	na (na)	na	0.5, 1, 2, and 3 gVS/gVS	37	9	Water displacement (3M NaOH)	195.2–516.34 $NLCH_4/kgVS_{loaded}$
[66]	Slaughterhouse waste from a pig and bovine slaughterhouse, waste mixed sludge from a WWTP.	Sampled directly from the digester from a WWTP	na	500 (400)	100	1:3 gVS/gVS	37	28	Water displacement system. Biogas analyser.	TS 4%—434.8–736.4 NL/kgVS TS 7%—647.7–674.1 NL/kgVS
[59]	Municipal sewage sludge from WWTP, Sherry-wine distillery from wastewater plant.	Effluent from laboratory-scale mesophilic AR	pH adjustment	250 (130)	120	60% (*v*/*v*) of substrate, and 40% (*v*/*v*) of inoculum	55	25	GC-TCD	175–302 $NLCH_4/kgV_{Sinitial}$
[49]	Sewage sludge from WWTP (OS, AS and DS).	Without using any external anaerobic inoculum	na	250 (150)	100	na	37	74	Liquid-displacement system (12% NaOH)	OS—86 ± 1 mL CH_4/g VS DS—125–135 mL CH_4/g VS AS—165 ± 1 mL CH_4/g VS
[72]	Food waste (FW), human faeces, toilet paper + water (TP).	Anaerobic digestate from an anaerobic digestion plant	Blender, mixed and diluted	120 (80)	40	na	35	40	GC-TCD	0.348 (TP)-0.619 (FW) L/g VS fed
[87]	Silages of cup plant, Virginia mallow, reed canary grass, tall wheatgrass, wild plant mix, giant knotweed.	From MWTP mesophilic AR	na	2000 (1600)	400	25 g VS/10 gVS	37	42	VDI Volumetric drum-type gas meter Infra-red sensor	132.08–389.49 $L_N/\cdot kgVS$
[85]	Fresh sugar beet from a farm.	Digested cattle slurry and maize silage pulp from agricultural biogas plant	Several times and method of storage	na (na)	na	According to [17]	39	21–26	DIN 38414-S.8 Gas analyser	135.84–148.23 $mL\cdot biogas/g_{fresh\ matter}$

Table 6. *Cont.*

Reference	Substrate Source	Inoculum Source	Pretreatment	Total and Working Volume (mL)	Headspace (mL)	I/S	T (°C)	Incubation Time (d)	Gas Measurement	Methane Production
[83]	Perennial plants from embankments of river: grass, alfalfa, red clover, mixtures.	Biogas plant which used swine and cattle manure	Dried, crushed and milled	1000 (160)	964	na	55	18	Pressure sensor. GC-TCD	190.9–403.2 $mLCH_4/gVS$ 188.2–268.8 $mLCH_4/gVS$ 236.6–276.9 $mLCH_4/gVS$ 177.4–336.0 $mLCH_4/gVS$
[84]	Sorghum bicolor varieties.	Anaerobic sludge from a full-scale up-flow sludge blanket reactor	na	250 (240)	10	0.5 gVS/gVS	35	≈31	Liquid displacement (2 N NaOH)	287–413 NL CH_4/kg VS
[103]	Abattoir solid (AS), winery solid (WS), cow blood.	Fresh zebra dung + rumen content	AS—minced, sterilized and thermally irradiated. WS—sundried and milled pH adjustment	500 (400) and 1000 (900)	100 100	0.5–2 gVS/gVS	38	34	Gas bag (3 N NaOH+ phenolphthalein). Portable Biogas analyser	6.29–369.56 $NmLCH_4/gVS_{added}$
[96]	Dried spent grape marc, cheddar cheese whey.	Sludge from a laboratory-scale digester of composition 3/1 grape marc and cheese whey	na	310 (100)	210	1/9, 3/7, and 5/5	45	58	Liquid displacement. Gas analyser	3.73–5.94 NL CH_4/kgVS
[97]	Gummy vitamin waste, grease waste, food waste, un-separated dairy manure.	AR effluent from a farm	na	300 (na)	na	1:1 gVS/gVS	35	67	Glass syringe (50 mL). GC-TCD	0–374 $NmLCH_4$/g VSsub
[94]	Wastes from alcoholic beverage.	Anaerobic effluent from a lab-scale digester treating liquid dairy manure and food waste	na	250 (na)	na	2 gVS/gVS	38	na	Manometric method. GC-TCD	148–727 L_NCH_4/kg VS

[1] calculated; AR—anaerobic reactor; AS—activated sludge; CSTR—continuous stirred tank reactor; DAF—dissolved air flotation tank; DS—dehydrated sludge; FID—flame ionization detector; FOG—fat, oil and grease; MHW—municipal household waste; OFMSW—organic fraction of municipal solid waste; OMSW—olive mill solid waste; RS—rice straw; SS—source separated; SSO—source separated organic; TWAS—thickened waste activated sludge; UASB—Up-flow Anaerobic Sludge Blanket; US—ultrasonic; WAS—waste activated sludge; WH—water hyacinth.

The results of the BMP tests presented in Table 6 concerning methane production revealed significantly discrepant values which are very difficult to compare. Angelidaki et al. (2009) [1] and Raposo et al. (2012) [109] reached similar conclusions. Considering all feedstocks analysed in the present work, the methane production ranges from 0 to 980 L of CH_4/kg of VS added and 440 to 1400 mL of CH_4 per g VSS added. Therefore, it is extremely difficult to achieve any correlation between these values and the experimental conditions, but the different units used also create a barrier for result comparison (e.g., mL CH_4/g VS_{added} and L_{biogas}/$kgCOD_{added}$,). To alleviate this issue, Holliger et al. (2021b) [121] defended the use of units $NLCH_4$/kgVS, which represent the volume of dry methane gas produced per mass of VS of the substrate added.

Usually, the experimental methane production obtained in BMP tests can be compared with the theorical methane production obtained by several methods that are presented in the next chapter.

5. Models to Predict Methane Production in BMP Assays

In the BMP assays, the methane productivity of a specific substrate can be obtained theoretically [76]. There are several models to perform the theoretical approach that can be classified into three types: the model based on the substrate chemical composition, which implies the use of empirical relationships, the model based on the COD concentration and the model based on the fractions of organic composition (carbohydrates, lipids, and proteins) [33,106,127–129]. The three models' equations can be found in Ali et al. (2018) [127]. However, an adjustment is necessary because all organic matter is considered biodegradable. Therefore, the biodegradability obtained from the experimental assays must be used [76]. Another drawback is that the accuracy of each method depends on the data of substrate composition; consequently, the theorical value of BMP assays is often higher than the experimental one [33,129]. Nevertheless, the methane potential obtained by the BMP test is an important parameter used in several models applied to estimate the cumulative methane production [127].

The variation of biogas production over time can be denominated by biogas production kinetics. There are many models to predict the cumulative methane production, the most used being the following: Gompertz, logistic, first-order, Richards, transfer, artificial Neuron, Cone, and Fitzhugh [10,127,128].

A study carried out by Ali et al. (2018) [127] presented the description of the models, their advantages, and disadvantages. Due to the several review studies [127,130] concerning the kinetics models in the present work, a survey was carried out on the application of these type of models to different feedstocks for anaerobic digestion, which is presented in Table 7.

Table 7. Kinetics models for BMP assays with different feedstocks.

Feedstocks	Substrate	Models Applied	Best Model	R^2	Reference
Animal manure	Dairy manure Horse manure Goat manure Chicken manure Swine manure	First-order Modified Gompertz Chen and Hashimoto	First-order	0.996–0.998	[10]
	Cattle slaughterhouse Agricultural	Cone First-order Modified Gompertz Dual pooled first-order	Cone	>0.985	[131]
	Chicken manure Cow dung	Modified Gompertz First-order	Modified Gompertz	0.955–0.981	[132]
	Poultry litter chicken and quail	First-order Modified logistic Modified Gompertz	Modified Gompertz	0.98–1.00	[133]

Table 7. *Cont.*

Feedstocks	Substrate	Models Applied	Best Model	R^2	Reference
Sludge	Domestic primary sewage sludge and food waste	Modified Gompertz	Modified Gompertz	na	[134]
	Biological sludge	First-order Modified Gompertz	First-order	0.98–1.00	[76]
Food waste	Cooked food waste Fruit waste Vegetable waste Uncooked food waste Paper waste Garden waste Textile waste	Modified Gompertz First-order	Modified Gompertz	0.96–0.98	[128]
	Orange and banana peels	Modified Gompertz Logistic First-order Richards Transfert	Modified Gompertz	na	[130]
	Palm fruits	First-order Modified Gompertz Surface-based	Modified Gompertz	0.998–0.999	[135]
	Food waste Chicken dung	Modified Gompertz Logistic, First-order Monod.	Modified Gompertz	0.8588–0.9208	[136]
	Organic faction of MSW	First-order Modified Gompertz	Modified Gompertz	1.00	[76]
	Bread waste Fish waste	Modified Gompertz First order	Modified Gompertz	0.947–0.985	[69]
Energy crops	Grass	Logistic Modified Gompertz Transfer	Transfer	0.997–0.998	[137]
	Grass	First order Modified Gompertz Logistics function	Modified Gompertz	na	[138]
	Grass Alfalfa Red Clover	Modified Gompertz First order Cone	Cone	na	[83]
Other organic	Vinasse	Modified Gompertz Logistic Transference	Modified Gompertz	0.948–0.999	[139]
	Brewery grain waste	Modified Gompertz First order	Modified Gompertz	0.959	[69]

na—not available.

As can be seen in Table 7, for the different feedstock groups presented, the model that best fit the experimental results of cumulative methane production is the modified Gompertz, although some of the other models present similar results, namely the first-order model.

The importance of the modified Gompertz model is reinforced by the fact that it was the only model applied to all the substrates referenced in Table 7.

6. Conclusions

This literature review shows that the anaerobic digestion process continues to be applied worldwide to several feedstocks and mixtures of them, with methane production enabling the generation of renewable energy and the organic valorization by the digestate.

There is a wide range of anaerobic feedstocks that can be classified into five groups: animal manure, food wastes, sludge, energy crops, and other organic wastes. The feedstocks are usually characterized by TS, VS, COD, and pH.

The BMP assays are an essential method to evaluate different substrates for anaerobic digestion, with wide-reaching application in the last four decades. The number of publications related to BMP assays has significantly increased, especially after 2011, until 2021, having stabilized in the last two years.

This present review demonstrated that despite the various attempts to standardize the BMP tests and the positive evolution, there are still some gaps that make it difficult to compare the obtained results in terms of the specific methane production, and consequently it is necessary to continue the investigation into this issue. Due to the growing demand for energy from renewable sources, the need to sustainably manage the biowaste production, and the results of recent years regarding the industrial application of anaerobic digestion, it is expected that scientific research will continue with the application of BMP tests with increasingly automatic, fast, and standardized methods.

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