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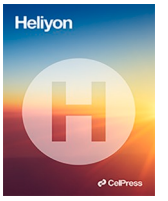

Review article

Valorization of crop residues and animal wastes: Anaerobic co-digestion technology

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ABSTRACT

To switch the over-reliance on fossil-based resources, curb environmental quality deterioration, and promote the use of renewable fuels, much attention has recently been directed toward the implementation of sustainable and environmentally benign 'waste-to-energy' technology exploiting a clean, inexhaustible, carbon-neutral, and renewable energy source, namely agricultural biomass. From this perspective, anaerobic co-digestion (AcoD) technology emerges as a potent and plausible approach to attain sustainable energy development, foster environmental sustainability, and, most importantly, circumvent the key challenges associated with mono-digestion. This review article provides a comprehensive overview of AcoD as a biochemical valorization pathway of crop residues and livestock manure for biogas production. Furthermore, this manuscript aims to assess the different biotic and abiotic parameters affecting co-digestion efficiency and present recent advancements in pretreatment technologies designed to enhance feedstock biodegradability and conversion rate. It can be concluded that the substantial quantities of crop residues and animal waste generated annually from agricultural practices represent valuable bioenergy resources that can contribute to meeting global targets for affordable renewable energy. Nevertheless, extensive and multidisciplinary research is needed to evolve the industrial-scale implementation of AcoD technology of livestock waste and crop residues, particularly when a pretreatment phase is included, and bridge the gap between small-scale studies and real-world applications.

1. Introduction

The exponential growth of the global population, industrialization, hastened urbanization, excessive dependency, and foreseeable exhaustion of fossil fuels (coal, natural gas, and crude oil) are the most disquieting and acute challenges of the modern era [1]. All these factors give rise to widespread issues rooted in waste accumulation, oil price hikes, air pollution, climate change, and global warming due to the emissions of GHGs and the release of sequestered carbon into the atmosphere [2,3]. Furthermore, when considering emissions by fuel type in 2020, fossil fuel consumption accounted for 93.22% of global CO_2 emissions (the percentages are 31.81%,

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Nomenclature

AcoD	Anaerobic co-digestion
AD	Anaerobic digestion
AGU	D-Anhydroglucopyranose units
ASA	Accessible surface area
BMP	Biochemical methane potential
BRF	Brown-rot fungi
C/N ratio	Carbon-to-nitrogen ratio
Ca	Calcium
CH_4	Methane
CI	Crystallinity index
CO	Carbon monoxide
CO_2	Carbone dioxide
Cu	Copper
DP	Degree of polymerization
FTIR	Fourier-Transform Infrared spectroscopy
GHG	Greenhouse gas
GPC	Gel-permeation chromatography
H_2	Hydrogen
H_2O	Water
HCO_3^-	Bicarbonate ions
HRT	Hydraulic retention time
Fe	Iron
K	Potassium
LAB	Lactic acid bacteria
LCB	Lignocellulosic biomass
LCC	Lignin-carbohydrate complex
LHW	Liquid hot water
LiP	Lignin peroxidase
Mn	Manganese
MnP	Manganese peroxidase
N	Nitrogen
NaOH	Sodium hydroxide
NH_3	Ammonia
ODM	Organic dry matter
OHPA	Obligate hydrogen-producing acetogens
OLR	Organic loading rate
P	Phosphorus
S	Sulphur
SE	Steam explosion
SRF	Soft-rot fungi
TAN	Total ammoniacal nitrogen
TS	Total solids
VFAs	Volatile fatty acids
VP	Versatile peroxidase
VS	Volatile solids
WRF	White-rot fungi
WSC	Water-soluble carbohydrates
Zn	Zinc

21.26%, and 40.15% for oil, gas, and coal, respectively) [4]. To alleviate the over-dependence on fossil-based resources and curb the global carbon footprint, it is necessary to hunt for an alternate, renewable, and clean energy source that can significantly contribute to environmental conservation, long-term energy security, and sustainable economic growth [5]. In this perspective, developing and developed countries have realigned the “take, make, and dispose" linear and conventional economic model of production and consumption with a greener, closed-loop, and circular bio-economy model [6,7]. In this model, biomass resources are efficiently and sustainably valorized in multi-output and integrated production chains such as biorefineries, while also fully recycling the waste and encouraging the long-term optimization of biomass value via cascading [8]. The circular bio-economy broadly means the sustainable valorization of renewable biological resources and waste streams into a multitude of useful and value-added products (biopolymers,

food, bio-based chemicals, feed) and bioenergy (power, biofuels, heat) [9,10], while contributing to the Sustainable Development Goals adopted by the United Nations Framework Convention on Climate Change [4]. Among auspicious bioenergy resources, agricultural biomass emerges as a substantial candidate to satisfy future energy demands, disregarding the harmful environmental problems because it is a carbon-neutral, abundant, and inexhaustible feedstock [11,12].

Lignocellulosic agri-waste pertains to second-generation biomass substrates and is typically categorized into crop/plant-based residues (such as wheat straw, corn stover, prunings, sorghum stalks, etc.) and livestock wastes (manure, droppings). Due to their significant annual generation in large quantities, the conversion and proper management of these wastes through the AcoD process represent one of the most promising bioenergy production strategies and potential energy sources for the future [13]. AcoD, or biomethanation process, denotes the concurrent digestion of feedstocks with complementary properties and their decomposition through the syntrophic interaction of various microbial populations under anoxic conditions across four consecutive phases (hydrolysis, acidogenesis, acetogenesis, and methanogenesis) [14]. The expression of the overall bioconversion reaction of biowaste organic fractions is given in **Eq. (1)** [15]:

$$C_cH_hO_oS_s + wH_2O \rightarrow mCH_4 + nNH_3 + SH_2S + (c\text{-}m)\ CO_2 \quad (1)$$

Where, w = 1/4 (4c-h-2o+3n+3s) and m = 1/8 (4c + h-2o-3n-2s). The biodegradable portions of biowastes ($C_cH_hO_oS_s$) are mostly composed of lipids ($C_{12}H_{24}O_6$), carbohydrates ($C_6H_{12}O_6$), and proteins ($C_{13}H_{25}O_7N_3S$) [15,16]. The deconstruction of feedstock and the segregation of cell-wall constituents (holocellulose and lignin) represent critical and intricate steps. This complexity arises from the inherent composition and structural features of the lignocellulosic material, encompassing cellulose crystallinity, polymerization degree, pore volume, degree of lignification, accessible surface area, and particle size. These factors often obstruct the biodegradability of cellulosic and hemicellulosic fractions and impede their accessibility to microbial attack [3,17]. Hence, a pretreatment step is compulsory before the AD process. The primary objective of pretreatment techniques, whether biological (e.g., fungi, ensiling, enzymes, microbial consortium), physical (extrusion, comminution, microwave, cavitation), chemical (acid, alkali, ozonolysis, ionic liquids, organosolv), or combinatorial (LHW, SE, ammonia fiber explosion), is to alleviate the inherent recalcitrance of LCB by increasing the ASA, disrupting the persistent carbohydrate-lignin shields, disintegrating the lignin sheath, and shrinking the crystalline structure of cellulose [18,19]. Thus, improving biogas rate and productivity. This review article is prompted by the urgent need to address contemporary environmental challenges arising from reliance on fossil-based resources, with a specific focus on the efficacy of AcoD as a potent strategy for sustainable bioenergy production. Emphasizing the critical need to transition toward bioenergy derived from renewable sources, particularly lignocellulosic residues and livestock waste, we underscore their potential not only to reduce dependence on exhaustible fossil fuels but also to contribute significantly to the reduction of carbon footprints and GHG emissions. A pivotal aspect of this review involves the identification and analysis of the 13 most prevalent models that quantify the annual potential of crop residues and livestock waste for bioenergy production. These models are meticulously drawn from various literature-based case studies conducted in different countries. Notably, to the best of our knowledge, this review stands as the first to present these models cohesively and collectively in a single review, providing a distinctive contribution to the field. This approach not only provides invaluable insights for farm owners, stakeholders, and researchers but also establishes a robust foundation for assessing total waste quantities when actual data may be lacking. Furthermore, this manuscript comprehensively explores various facets of AcoD, including the intrinsic structural and compositional features of lignocellulosic residues, the different biotic and abiotic parameters impacting the overall co-digestion performance, and the physico-chemical factors influencing pretreatment efficiency. Additionally, we delve into the latest trends, limitations, and noteworthy research advancements in diverse pretreatment methods applied prior to the AcoD process, contributing to a comprehensive understanding of the complexities involved in advancing sustainable bioenergy production from lignocellulosic residues and livestock waste.

2. Agricultural biomass

2.1. Classification

Generally, agricultural wastes encompass the following categories.

1) Crop or plant-based residues can be categorized into two types: primary and secondary. Primary residues, also known as field-based residues, are generated as by-products of crop post-harvesting, including cereal straw, maize stalks, stubble, leaves, tree prunings, and branches. Secondary or process-based residues are collected during industrial crop processing, such as oil extraction and milling, which include maize cobs, bagasse, nut shells, almond husks, olive stones, and coffee pulp [20,21].
2) Livestock wastes predominantly consist of animal manure, slurry, poultry droppings, and bird feathers.
3) Forest residues and wood wastes include sawdust, timber slash, wood chips, and old tree trunks, among others.
4) Vegetable and fruit residues (e.g., lettuce, mango, cabbage, etc.).

2.2. Theoretical estimation of annual potential in selected countries

2.2.1. Crop residues

The gross residue potential, which represents the total annual residue generated by a particular crop, can theoretically be predicted based on various parameters, including the crop's yearly production data, cultivated area, moisture content, crop yield, and the

Table 1
Theoretical potential of field-based residues generated annually in various countries.

Country	Crop	Residue type	Total residues potential per year	Available residues potential (surplus) per year	Theoretical biomass potential estimation	References
Turkey (Hatay)	Wheat Barley Maize Olive	Straw Straw Stalk Cob	–	35,037 (t) 610 77,291 14,492	$\mathbf{(AAR)i = (AAP)_i \times (RPR)_i \times (A)_i}$ $\mathbf{(AAR)_i}$ = Available amount of agricultural residues of ith crop in ton $\mathbf{(AAP)_i}$ = Amount of agricultural product in tons or number of tree for pruning wastes $\mathbf{(RPR)_i}$ = Residue-to product ratio of the ith crop $\mathbf{(A)_i}$ = Availability of residues	[25]
India	Wheat Sugarcane Maize	Straw Chaff Tops & leaves Stover	122,991(Kt) 22,362 40,986 28,396	–	**Residue Production = Residue Yield × Gross Cropped Area**	[22]
Turkey	Wheat Barley Maize Rye Grapes	Straw Straw Stalks Cob Straw Prunings	Avg. : 22,218 (Kt) Avg. : 8491 Avg. : 10107 Avg. : 3356 Avg. : 278 Avg. : 843	–	$\boldsymbol{TBP = \sum_{i=1}^{n} CP(i) \times RPR(i) \times \left[\frac{100 - M(i)}{100}\right]}$ **TBP** = Theoretical biomass potential **CP (i)** = Amount of product produced per year as tons **M (i)** = Relative moisture content in percentage terms	[26]
Greece (Florina)	Wheat Barley Maize Rye Pear Apple	–	7.822 (t) 8.550 34.678 12.501 130 4.325	–	$\boldsymbol{THP - RAP = \sum(CAi \times APi \times RtPi \times Avi)}$ (For cereal crops residues) $\mathbf{THP\_RAPpr = \sum[Production(tn) \times RtPri \times Avi]}$ (For fruit tree pruning) **THP_RAP** = Primary agricultural residues in tonnes $\mathbf{CA_i}$ = Cultivated area of i crop, in decares $\mathbf{AP_i}$ = Agricultural production of i crop, in tonnes per decare $\mathbf{RtP_i}$ = residue to product ratio of i crop $\mathbf{Av_i}$ = Availability of residues for i crop according to current harvesting system	[27]
Pakistan	Wheat Corn Rice	Straw Stover Straw	–	1943 (10^4 Mt) 452 574	**R (Mt) = GY (Mt) × WGR × availability** **R (Mt)** = Crop residue produced in million tonnes (Mt) **GY (Mt)** = total grain yield in (Mt) **WGR** = waste to grain ratio **Availability** = percentage (%) of crop residue potentially available for exploitation as a fuel for electricity generation	[28]
Bangladesh	Wheat Rice Maize	Straw Straw Stalks	–	0.2 (Mt) 29.5 2.8	$\mathbf{GCR_i = Y_i * (RYR)_i}$ $\mathbf{GCR_i}$ = gross crop residue amount generated annually by the crop type i $\mathbf{Y_i}$ = annual crop yield $\mathbf{(RYR)_i}$ = residue-to-yield ratio of crop type i	[29]
Southern Italy (Calabria)	Wheat Barley Maize Citrus Grapevine	Straw Straw Straw – –	135,569 (t) 24,117 14,134 39,822 13,799	–	$\mathbf{R_c = P_c\ RPR}$ (For cereal crops residues) $\mathbf{R_L = A_c\ RAR}$ (For fruit tree pruning) $\mathbf{R_L}$ = potential annual amount of lignocellulosic residues from periodic pruning operations of tree crops **Ac** = cultivated area **RAR** = residue-to-area rate $\mathbf{R_C}$ = straw quantity produced yearly $\mathbf{P_C}$ = crop production referred to grain **RPR** = residue-to-product rate	[30]
Cameroon	Wheat Maize Rice	Straw Stalk Cob Straw	0.7 (Kt) 3144.1 429.2 209.2	–	$\boldsymbol{ARG = \sum(RPR \times AH)}$ **ARG** = amount of a residue generated annually **RPR** = residue production mass ratio of the economic product **AH** = annual harvest mass of the crop or product	[31]

residue-to-product ratio (RPR, defined as the ratio of residue weight generated to the total weight of crops obtained after harvesting) [22]. Table 1 presents the annual potential of agricultural residues in various countries. In general, the potential of crop residues is directly proportional to total crop production. However, this potential varies from crop to crop and from one region to another due to numerous factors, notably the type of crop grown, soil type, local meteorological conditions, and harvesting techniques (whether manual or mechanical) [23]. It is important to note that not all estimated agricultural residues will be collectible and technically useful for bioenergy production. Only a portion, often referred to as the surplus residue potential, can realistically be harnessed due to competing applications such as livestock fodder and bedding, household fuel for cooking and heating, roof thatching, as well as for ecosystem services like soil mulching, conservation of soil fertility, and erosion control [24].

2.2.2. Livestock waste

The quantities of animal residues produced annually by large ruminants (buffaloes, horses, and beef cattle), small ruminants (pigs, sheep, and goats), or poultry (ducks, native chickens, turkeys, broilers, and layers) can generally be estimated using the number of livestock (heads) and the annual average manure generated per head [32] (Table 2). However, the amount of these wastes is heavily contingent on factors such as the type, age, size, and body weight of the animal, the breeding type, and feed intake [31].

3. Composition and structure of lignocellulosic residues

Lignocellulose is a natural, three-dimensional, intricate biocomposite consisting of three major interwoven biopolymers: cellulose $(C_6H_{10}O_5)_n$, hemicellulose $(C_5H_8O_4)_m$, which are high molecular weight polysaccharides, and lignin $[C_9H_{10}O_3(OCH_3)_{0.9–1.7}]_x$, along with a variety of other compounds present in minor amounts, referred to as extraneous materials, such as extractives, lipids, ash, and proteins [18,35]. These polymers are tightly intertwined and bonded through covalent or non-covalent linkages, forming a complex array known as the lignocellulosic hetero-matrix [36,37]. This network exhibits high resistance to depolymerization, explaining the intrinsic recalcitrant nature of biomass [38]. The proportion of cellulose, hemicellulose, and lignin within a particular crop differs depending on the plant species, origin, season, and stage of growth, as well as harvesting and storage processes [39,40].

3.1. Cellulose

Cellulose, the main structural and integral mesogen in the lignocellulose cell wall, typically makes up 40–50% of the lignocellulosic material by dry weight [41]. It is a linear, unbranched, non-meltable, syndiotactic homopolymer consisting of parallel AGU units covalently bonded together through β-(1,4) glycosidic bonds, with an average molecular weight of approximately 100 kDa [42,43]. Consecutive glucose molecules in the cellulose chain, also known as cellobiose, are oriented 180° to each other [43]. It is noteworthy

Table 2
Theoretical potential of livestock waste generated annually in various countries.

Country	Livestock	Total recoverable manure per year	Theoretical biomass potential estimation	References
Bangladesh	Buffaloes Cattle Goats Sheep poultry	0.7 (Mt) 12.3 1.5 0.2 2.1	$\mathbf{LM_j = N_j * (RGR)_i}$ $\mathbf{LM_j}$ = annual manure production of livestock species j $\mathbf{N_j}$ = head count of livestock type j $\mathbf{(RGR)_i}$ = residue generation rate	[29]
Southern Italy (calabria)	Bovine Pig Poultry	196,380 (t) 42,610 16,298	$\mathbf{R_A = N_A\ RPC}$ $\mathbf{R_A}$ = Amount of annual biomass (a blend of wastewater and manure) $\mathbf{N_A}$ = number of animals (capita) **RPC** = produced residue per capita	[30]
Thailand	Beef Dairy Buffaloes Goats Sheep	8760.00 ± 9.68 (10^2 t) 4884.00 ± 7.67 2994.93 ± 2.44 256.93 ± 0.67 46.44 ± 0.18	$\mathbf{RPLM} = \sum_{j=1}^{m} \boldsymbol{Hj} \times \boldsymbol{YDj} \times \boldsymbol{Tj} \times \boldsymbol{Rj}$ **RPLM** = recoverable potential of livestock's manure **Hj** = number of livestock (heads) **Ydj** = the dry matter yield of livestock's manure **Tj** = growth time of livestock (d) **Rj** = recoverable fraction of manure	[33]
South Africa (KwaZulu-Natal + Limpopo)	Cattle Goats Sheep Pigs Chicken	102.93 (t) 6.98 3.47 3.15 0.37	$\mathbf{AM = P_{live} \bullet M \bullet RF}$ **AM** = potential of recoverable animal manure for biomethane energy use $\mathbf{P_{live}}$ = number of animals (heads) **M** = estimated amount of manure per head **RF** = recoverability fraction	[34]
Greece (Florina)	Cattle Pigs Goats and sheeps	121.878 (t) 1.452 102.120	$\mathbf{THP\_Manure = \sum (NHeads_i * MpH_i)}$ **THP_Manure** = theoretical potential of manure $\mathbf{NHeads_i}$ = number of heads for the i type of livestock $\mathbf{MpH_i}$ = amount of manure for the i type of livestock	[27]

that extensive inter- and intramolecular hydrogen bonding among hydroxyl groups (OH), as well as van der Waals bonds in cellulose molecules, create a strong and stable structure referred to as “microfibrils", which, in turn, impart stiffness to cellulose strands [36]. Cellulose chains generally consist of both crystalline (highly ordered) and amorphous (less organized) regions.

3.2. Hemicellulose

As the second most abundant branched and heterogeneous heteropolymer, hemicelluloses, also known as polyoses, are composed of various components, namely uronic acids (D-galacturonic,D-4-*O*-methylgalacturonic, and D-glucuronic acids), acetylated sugars, pentoses (arabinose, xylose), and hexoses (glucose, galactose, and mannose) [44,45]. Unlike the crystalline and tightly packed structure of cellulose, hemicellulose has an amorphous and random nature, along with a low degree of polymerization, ensuring high susceptibility to chemical and thermal hydrolysis. Additionally, hemicellulase enzymes both remove the side chains and attack the backbone, thus releasing oligosaccharides that are further degraded into simple sugars [46].

3.3. Lignin

Lignin is the third most plentiful naturally occurring biopolymer in plant cell walls. It is a non-carbohydrate aromatic, amorphous, hydrophobic, and three-dimensional polyphenolic polymer [47]. Lignin is tightly enmeshed with cellulose and hemicellulose fibers through covalent linking, forming an impermeable barricade called LCC. This structure imparts structural rigidity, impermeability, mechanical strength, and moisture resistance to the lignocellulosic plant cell wall, creating a protective bulwark that obstructs any microbial invasion [48–50]. Lignin is formed by enzymatic dehydrogenative polymerization of three *o*-methoxylated *p*-hydroxyphenyl propanoid units, which exist as lignin monomers (monolignols), including *p*-coumaryl alcohol (*p*-hydroxyphenyl propanol, (H-unit)), sinapyl alcohol (syringyl alcohol, (S-unit)), and coniferyl alcohol (guaiacyl propanol, (G-unit)) [39,50]. These phenolic moieties are held together via diaryl-ether and C–C linkages such as β-5 (phenylcoumaran), β-1 (1,2 diaryl propane), 5-5 (biphenyl and dibenzodioxocin), and β-β (dibenzodioxocin). However, these bonds are believed to be more resilient to chemical degradation due to their “condensed" structure compared to aryl-ether connections, which are described as “non-condensed" or “unstable" [51].

The G:S:H unit ratio and the total lignin content in plants may vary significantly based on the plant species and tissue. For instance, lignin content in softwoods (gymnosperms) is significantly greater than in hardwoods (angiosperms), agricultural residues, and herbaceous species. Furthermore, G-units (G-lignin) dominate in softwood, accounting for around 90% of the total units, while (GSH-lignin) units are mainly present in monocotyledonous plants, and both G- and S-units are found in dicotyledonous plants [52,53].

3.4. Extraneous materials

In addition to the three main pre-described components, lignocellulosic biomass (LCB) contains numerous extra substances referred to as non-structural (non-cell wall) components, most of which occur in low amounts and are termed “extraneous materials". These materials can be categorized into two groups based on their solubility in water and/or neutral organic solvents: extractives or non-extractives [52]. Extractives encompass phenols (tannins, flavonoids, and stilbenes), resins (e.g., alcohols, phytosteroles, and fatty acids), and terpenes (e.g., ketons). They can be extracted using polar solvents (e.g., water, acetone, dichloromethane, and ethanol) or non-polar solvents (benzene, toluene, and hexane) [36,54]. Non-extractives refer to inorganic components, including pectins, proteins, starches, and metal salts (alkaline earth carbonates, oxalates, and silicates) [36].

4. Valorization of agricultural wastes

4.1. Mono- and co-digestion

The AD process is increasingly emerging as a sustainable and rational biotechnology for biomass handling. However, from both economic and microbiological perspectives, mono-digestion of carbon-rich lignocellulosic residues or N-rich animal manure is not a profitable and viable option due to several substantial factors. These include an imbalance of micro- and macro-nutrients (inappropriate C/N ratio), deficiency of trace metals (e.g., iron, nickel, molybdenum, cobalt, and selenium), process instability, and the production of low-quality mono-digestate, primarily from animal manure. This can result in serious environmental issues such ecotoxicity, phytotoxicity, soil salinity, and heavy metal accumulation [55,56]. Crop residues (C-rich substrates) are distinguished by a high organic loading, a high carbon-to-nitrogen ratio, but a low buffering ability [57]. In contrast, animal manure contains.

1) A wide variety of nutrients, namely (P), (N), (K), and micronutrients, such as (Fe), (Zn), (Mn), and (Cu).
2) High ash and moisture contents.
3) Strong alkaline metals (high buffer capacity), such as (Ca) and magnesium, originating from growth-promoting feed additives.
4) The requisite fermentative and methanogenic microbes for the start-up of the AD process [58,59].

Therefore, lignocellulosic residues can be a prospective co-substrate candidate to offset the carbon shortage of livestock manure and to meet the nutritional needs for microbial growth in the digester [60]. AcoD is considered a practical and potent measure to mitigate the hindrances encountered in mono-digestion by concomitantly digesting two feedstocks with complementary properties and to enhance the economic viability of AcoD facilities due to increased methane yields [61]. Furthermore, AcoD can provide the

following advantages [62–65].

a) Improved stability and nutritional equilibrium in the system due to the stabilized (C/N) ratio and supplementation of trace elements.
b) Dilution of inhibitory compounds (ammonia nitrogen, lignin derivatives, long-chain fatty acids) or toxic compounds (heavy metals) to a safe level below the thresholds.
c) Increased loading of biodegradable organic matter.
d) A safe and nutrient-rich co-digestate for agricultural applications.

When compared to mono-digestion of the same feedstocks, AcoD can improve the biogas yield by 25%–400% [63,66]. Despite the enumerated benefits, AcoD remains an intricate and responsive system whose stability and efficiency depend fundamentally on several factors, including the diversity of microbial populations, their resilience, and interactions in the bioreactor, the co-feedstock composition and properties, and the optimal blending ratio of substrates.

The microbial process of AcoD is characterized by a sequence of consecutive stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Fig. 1): **Hydrolysis phase:** During this stage, extracellular enzymes, including amylase, lipase, cellulase, protease, and pectinase, are secreted by facultative and obligatory anaerobic bacteria. These enzymes break down complex biopolymers such as carbohydrates, proteins, nucleic acids, and lipids into soluble mono- and oligomers, resulting in the formation of simple sugars, amino acids, purines, pyrimidines, and fatty acids [67]. **Acidogenesis/fermentation phase:** During acidogenesis, monomers generated in the hydrolytic phase undergo further decomposition into volatile fatty acids (such as butyrate, acetic acid, isobutyrate, isovalerate, and propionic acid), CO_2, methylamines, hydrogen, lactate, alcohols, and other byproducts. Bacteroidetes, Firmicutes, Chloroflexi, Proteobacteria, and Cloacimonetes are the predominant phyla present in this stage [68]. The specific concentrations and percentages of intermediates generated in this stage may vary depending on the conditions within the digester, and they can have either a positive or negative impact on the overall performance of the biodigestion system [69]. **Acetogenesis/dehydrogenation phase:** This step involves all the reactions leading to the production of acetate. On one hand, homoacetogenic bacteria convert H_2 and CO_2 into acetate through anaerobic respiration. On the other hand, (OHPA) convert alcohols and VFAs into CO_2, acetic acid, and H_2 [70]. **Methanogenic phase:** This final phase occurs under strictly anaerobic conditions. Three types of methanogenic archaea, namely, acetotrophic or acetoclastic, hydrogenophilic or hydrogenotrophic, and methylotrophic, generate methane. Acetoclastic methanogens split acetate into CO_2 and CH_4 (see Eq. (2)) [71]. Hydrogenotrophic methanogens consume 1 mol of CO_2 as a carbon source and 4 mol of H_2 as an electron donor to generate 1 mol of CH_4 (see Eq. (3)) [72]. Meanwhile, methylotrophic methanogens produce methane by the decarboxylation of methylamines, methylsulfides, and methylalcohols [73].

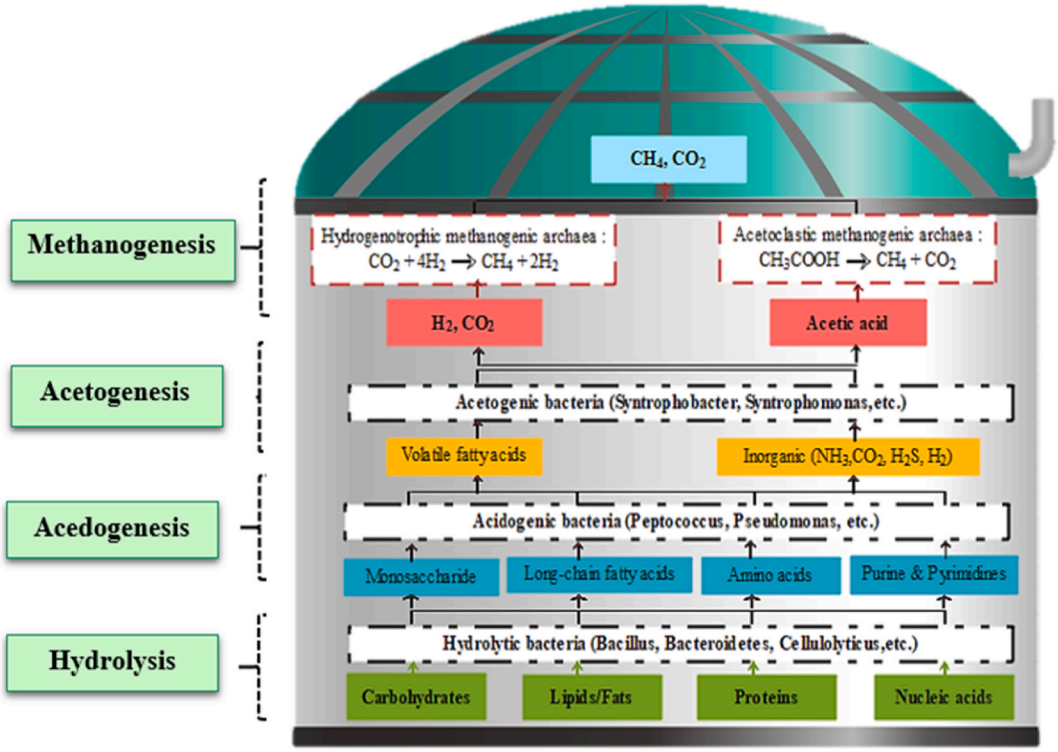

Fig. 1. Degradation steps of lignocellulosic material in the anaerobic digester (modified from Ref. [74].

$$CH_3COOH \rightleftharpoons CH_4 + CO_2, \quad (2)$$

$$\Delta G^0 = -36 \text{ kJ/mol}$$

$$4H_2 + CO_2 \rightleftharpoons CH_4 + 2H_2O, \quad (3)$$

$$\Delta G^0 = -130 \text{ kJ/mol}$$

It is important to emphasize that the structure of the microbial consortium, its diversity (species evenness and richness), as well as the syntrophic relationships between anaerobic bacteria, are critical factors to highly consider when assessing the effectiveness and stability of the AD system. When properly understood, these factors can serve as biomonitoring tools to predict system gaps and failures while sustaining functional stability and ensuring the microbial ecosystem [67,75]. For instance, an ecosystem with high richness and evenness (relative abundance) can be an indicator of stable and well-balanced AD performance [68].

4.2. Main parameters affecting AcoD process

Besides the microbial consortia (biotic factor), abiotic factors (digester parameters) such as the carbon-to-nitrogen ratio, pH, mixing, inhibitors, OLR, alkalinity, temperature regime, HRT, etc., can either positively or negatively influence the overall digestibility and performance of the process. Therefore, to optimize the AcoD process and achieve maximum biogas yields, these parameters must be regularly and accurately tracked and maintained within their optimal levels. Any deviation of a parameter from its optimal range may slow down or even halt microbial growth and, consequently, disrupt the entire process [76].

4.2.1. Temperature regime

The AD process typically operates within three distinct temperature regimes: psychrophilic (<20 °C), mesophilic or cryophilic (25–40 °C), and thermophilic (45–60 °C) [73]. Generally, operating under high-temperature conditions (thermophilic) results in a faster and more productive process compared to mesophilic and psychrophilic conditions. This is attributed to effective pathogen deactivation, shortened retention time, faster reaction kinetics, reduced viscosity, increased solubility of organic fractions, and a higher growth rate of methanogens [60,77]. It has been reported that the biogas production rate under thermophilic conditions is enhanced by 41% and 411% compared to cryophilic and psychrophilic temperatures, respectively [78]. However, thermophilic digestion is highly vulnerable to sudden environmental disturbances and often leads to chronic increases in NH_3 and VFA inhibition [79]. It has been shown that ammonia inhibition in co-digestion can be reduced or avoided by raising the substrates' C/N ratio to an acceptable level, or by optimizing the pH level [80]. Among the temperature ranges, mesophilic digestion is the most widely adopted in full-scale anaerobic digesters, as it is less sensitive to inhibitors and provides better process stability [62]. On the other hand, the psychrophilic system is the most suitable for cold environments and is the most economically sustainable since it requires zero heat energy demand [78]. Psychrophilic (cold-adapted) microorganisms thrive in cold regions or sub-zero temperatures by increasing the unsaturated fatty acids in their membrane lipids, excreting specialized enzymes (trehalose disaccharide), and modifying the structural conformation of their DNA [78,81].

4.2.2. pH level and buffer capacity

The pH level is a crucial operational parameter that significantly influences the overall AcoD system's progress. The optimal pH values fluctuate at each phase of the process. For example, the pH optima for hydrolysis and acidogenesis lie between 5.5 and 6.5, while methanogenesis occurs at pH 6.5–7.2, with an optimum pH of around 7.0 [82]. The ideal pH range to achieve the highest biogas yields in AD is reported to be between 6.8 and 7.2 [63]. However, it should be noted that pH is not a stand-alone parameter, as it depends on the system's buffer capacity, the partial pressure of CO_2, bicarbonate and VFA concentration [83]. Buffer capacity, also known as alkalinity, refers to the equilibrium between CO_2 and HCO_3^- [84]. It is considered a credible monitoring parameter for assessing imbalances and fluctuations within the digester than direct measurement of pH values, as buffering capacity will drastically decline with excessive ammonia and VFAs accumulation before the pH drops [66]. To increase the system's alkalinity and alleviate pH drops, various solutions have been explored in the literature, including: i) the reduction of organic loading rate; ii) the usage of additives like neutralizers (e.g., potassium hydroxide, sodium hydroxide), bicarbonates, zero-valent iron (ZVI), and supplement micronutrients (e.g., trace metals) [85].

4.2.3. Carbon to nitrogen ratio

The C/N ratio refers to the proportion of carbon and nitrogen found in organic materials. Carbon acts as the principal energy source for microbial growth, while nitrogen is an important element for (i) the synthesis of amino acids, proteins, nucleic acids, and (ii) the maintenance of neutral pH conditions in the digester due to its neutralizing effect on volatile acids when it is in reduced form [86]. It has been reported that anaerobes consume carbon 25–30 times faster than nitrogen [87]. Thus, for a balanced AD system, the C/N ratio should be maintained between 20 and 30, 16–25, or 20–35, depending on the nature of the feedstock [66,88,89]. A high C/N ratio causes a poor rate of protein solubilization, resulting in low TAN and unionized ammonia/free ammonia concentrations within the digester. Conversely, a low C/N ratio induces high ammonia accumulation and impedes methanogenic activity, resulting in reduced biogas production [62]. Wang et al. [90] reported that C/N ratios of 25 and 30 provide better digester performance due to a stable pH around 7.0 and low TAN and NH_3 concentrations.

4.2.4. Type of substrate

The type of substrate and its biochemical composition are crucial parameters that significantly influence digester performance, biogas yield and composition, substrate biodegradability, stability, and effectiveness of the AcoD process. The type of substrate is a key criterion for selecting the appropriate mixture of co-substrates to be fed into the digester. Protein-rich substrates, mainly derived from animal manure, milk and meat processing industries, and slaughterhouse waste, possess high biological oxygen demand, high nitrogen concentration, and yield a greater methane potential [91,92]. However, the microbial degradation of this substrate type often leads to ammonia buildup, which can impede methanogenic activity at values up to 4 g/l, destabilize the fermentation, and even lead to process failure [93–95]. AcoD with carbohydrate-rich substrates (e.g., lignocellulosic residues, food wastes, and vegetable wastes) is one of the most efficient tactics to prevent such problems. Lipid-rich feedstocks, such as slaughterhouse waste, grease traps, palm oil mill effluents, and olive oil mill effluents, hold a higher theoretical methane potential than carbohydrate-rich and protein-rich substrates [96]. The degradation of lipidic waste produces long-chain fatty acids, which, in high concentrations, may impede methanogenic metabolism, clog the mass transfer between anaerobes and the media, cause sludge washout, and lead to the formation of scum and foam [96,97]. Therefore, the use of these wastes as co-substrates in the AcoD process can significantly alleviate the inhibitory effect of long-chain fatty acids and improve process efficiency.

4.2.5. Organic loading rate (OLR)

OLR is a key operating parameter that must be considered when choosing the system design, including digester size and type. It represents the amount of volatile solids (VS, or organic dry matter – ODM) loaded into the digester per m^3 of working volume per day. OLR can be estimated using **eq. (4)** [98,99]:

$$\mathrm{OLR} = \frac{Q \times VS}{V} \left(\mathrm{Kg\ VS\ m^{-3}\ d^{-1}}\right) \tag{4}$$

Q represents the daily flow rate of feedstock fed into the digester, VS is the influent concentration in volatile solids content (%VS), and V is the reactor operating volume (m^3). A high OLR enriches the bacterial medium within the bioreactor, demands lower energy requirements for heating, decreases the digester's capacity requirements, and yields high biogas production [61]. However, overloading of OLR causes over-accumulation of VFAs and ethanol, as the rate of hydrolysis/acidogenesis overtakes the methanogenesis rate, ultimately causing irreversible acidification of the medium and reactor failure [100]. Additionally, it can hinder heat transfer, create an unbalanced distribution during agitation, and potentially damage circulating pumps when it exceeds their load capacity [101,102]. The co-digestion of pig manure and sugar beet byproduct under mesophilic conditions for 6 days resulted in the highest methane productivity at an OLR of 11.2 gVS/Lreactor d [103].

4.2.6. Hydraulic retention time (HRT)

HRT is a critical parameter that determines the average duration that a substrate remains within the bioreactor, or the time necessary for microorganisms to completely consume and break down the organic matter. It can be calculated using **Eq. (5)**, where V denotes the digester volume (m^3), and Q represents the substrate daily feed rate (m^3/day) [98]:

$$\mathrm{HRT\ (d)} = \frac{V}{Q} \tag{5}$$

The actual residence time will differ from the one defined since the optimal HRT is highly dependent on several factors.

1) Substrate composition: Carbohydrate-rich substrates are readily decomposed by microorganisms, resulting in a relatively reduced HRT, while microorganisms may require much longer time to effectively decompose fiber- and cellulose-rich substrates [15].
2) Operating temperature: For example, the retention time varies between 10 and 40 days in a mesophilic environment, but it is shorter (around 14 days) in thermophilic conditions [82]. It has been reported that retention time decreases with increasing temperature up to 35 °C, and the opposite is true [104].
3) Type of mixing.
4) Climate conditions: In cold-climate countries, the HRT may extend up to 100 days compared to warm climate regions, where the values range typically from 30 to 50 days [104]. Moreover, an extended HRT can cause the death of microorganisms due to nutrient scarcity, and necessitates a large digester volume and high operational expenses. Conversely, short HRT is preferable for industrial-scale applications as it requires a smaller digester volume and entails lower investment costs. However, it may lead to microbial washout, cell intoxication, and the accumulation of VFAs, resulting in reduced biogas production [61,105].

4.2.7. Stirring

Being regarded as a prominent physical parameter, the stirring (mixing) within the digester has a substantial impact on the overall productivity of the biogas plant. Stirring offers several advantages, including [83,106,107]. .

a) Sustains continuous and close contact between the bacteria, nutrients, and the digesting substrate.
b) Liberates easily trapped gas bubbles in the reactor.
c) Evades the formation of scum, sediments, crust, and foam.
d) Dilutes the concentration of any toxic agents contained in the digester.
e) Ensures a homogeneous medium for anaerobic bacteria.

f) Promotes the distribution of heat throughout the entire substrate mass.

The main stirring technologies employed in biogas facilities include mechanical, hydraulic, and pneumatic systems. Of these, mechanical or impeller mixing stands as the prevailing technology in Europe today [108]. Hydraulic mixing is accomplished using pumps, mainly airlift pumps, positioned outside the reactor, which circulate the AcoD slurry. However, the main limitation of this technique is its suitability only for small-scale digesters [109]. Pneumatic mixing utilizes the generated biogas, which is pumped and released at the bottom of the digester, creating a horizontal stirring motion as the gas bubbles ascend to the surface [110]. However, it is not commonly used for agricultural feedstocks and is primarily employed for thin liquid substrates that are less prone to form floating layers [111]. The efficiency and performance of any stirring technology rely on various factors, mainly the duration and rotating speed of agitation, the mixing mode (continuous, intermittent, or minimal mixing), the digester's geometry, and the rheological properties of the sludge [110,112]. It has been reported that high mixing intensity has a detrimental impact on digester performance, as it increases shear stress, disrupts bacterial morphology, especially in the archaea group, breaks the spatial juxtaposition of microorganisms, which restricts syntrophic interactions between species, and consequently results in reduced biogas yields [107,113,114]. Nevertheless, higher agitation speeds are advantageous during reactor start-up, but as the process reaches the methanogenesis phase, it becomes essential to reduce the stirring intensity [115].

5. Bioreactor configurations

In AcoD processes, several bioreactor configurations can be employed based on critical parameters, including the residence time, the amount of biomass to be digested, operating temperature, mixing, continuity/feeding mode (batch versus continuous), number of steps involved (single-stage versus two-stage), dry matter content/total solid concentration (wet versus dry), as well as feedstock properties, solubility, and hydrolysis rates [116].

5.1. Wet vs dry digester

Solid-state or dry digesters are typically used when the TS content of the feedstock exceeds 15% (usually between 20% and 40%). This mainly includes green waste, energy crops (either ensiled or fresh), crop residues, agricultural by-products, household and municipal organic waste. In contrast, wet or liquid-state digesters are designed to process feedstocks with high moisture content (TS<15%), such as sewage sludge, livestock manure, domestic and household wastewater [117,118]. Solid-state anaerobic digestion exhibits the following advantages [119,120]: i) smaller digester volume; ii) minimal energy requirements for stirring and heating; iii) increased methane productivity; iv) easier digestate handling due to its low water content. However, solid-state AcoD often faces challenges, namely, low methane yields and system instability owing to insufficient mass transfer, accumulation of inhibitors, and nutrient imbalance [121].

5.2. Single-stage vs multi-stage digester

In a single-phase system, the microbiological stages of the AcoD pathway occur within a single reactor, while, the two-phase AD takes place in two separate reactors arranged in series, operating under distinct conditions. The first reactor, where hydrolysis, acidogenesis, and acetogenesis are prevalent, is often operated under acidic conditions (pH 5.5–6.5) with a short HRT. The second reactor, where methanogenesis predominates, is maintained at an optimal pH level (between 6 and 8) with a relatively longer HRT (20–30 days) [122,123]. Two-phase AcoD systems are especially recommended for treating feedstocks with high lipid content [124]. The two-stage AcoD process offers several benefits over the single-stage AcoD, namely [125,126].

1) Improved process stability and overall performance due to improved control of the acidification phase in the first tank, preventing overloading and pH shock to the methanogenic bacteria in the second reactor.
2) Ability to handle high OLRs.
3) Efficient destruction of pathogenic microorganisms.
4) Increased degradation, resulting in higher methane production.

Nevertheless, the primary challenges limiting the widespread application of this system include higher maintenance and implementation expenses, the possibility of hydrogen gas buildup in the reactor's acidic phase, and sophisticated parameter control systems [127,128]. In addition to the two-phase system, the multi-stage AcoD process also incorporates a three-stage system. There are distinct concepts for distributing process steps among the three-stage compartments. The first one is to associate hydrolytic and acidogenic bacteria since they often thrive under similar optimal growth conditions and to split up acetogens and methanogens due to their distinct nutritional requirements, growth kinetics, and physiological characteristics, leading to the following sequential process: 1) hydrolysis/acidogenesis, 2) acetogenesis, and 3) methanogenesis [129,130]. For the second concept, the four digestion phases are divided as follows: 1) hydrolysis, 2) acidogenesis/acetogenesis, and 3) methanogenesis. This separation arises from variations in the required mixing intensity for each stage. Intense mixing is beneficial for improving hydrolysis but is counterproductive in the case of methanogenesis. Acidogenic and acetogenic chambers, on the other hand, require moderate mixing [131]. As microbial metabolism is non-homogeneous in reality, the separation order of digestion phases for the third concept is, 1) hydrolysis/acidogenesis, 2) acidogenesis/acetogenesis, and 3) acetogenesis/methanogenesis [129,132]. The three-stage AcoD system is designed to independently

control the operational parameters in each reactor chamber, as it lowers the retention time, accelerates the stabilization rate, and can remove VS with an efficiency of up to 83.5% [126].

5.3. Batch vs continuous digester

Generally, anaerobic digesters are classified into continuous and batch reactors based on the substrate feeding mode. In batch-mode digesters, the reactor is fed once with raw substrate for a specific time period, inoculated with anaerobic sludge from another digester and completely evacuated after complete degradation. In contrast, in continuous digesters, the raw material is continuously and evenly fed, either mechanically or by the force of the newly loaded feedstock, pushing out the already digested substrate [111,133]. Continuous digesters include plug-flow systems, expanded granular sludge blankets, continuous stirred tank reactors, up-flow anaerobic sludge blanket reactors, internal circulation reactors, while batch AcoD processes employ hybrid and sequential batch reactors [134]. The application of a continuous-feed digester is often preferred over a batch digester for several reasons [132,135].

1) It requires a smaller reactor space.
2) It promotes a high microbial growth rate and a high resistance of bacteria to environmental fluctuations.
3) It offers better operational stability.
4) It ensures good contact and efficient mass transfer between biomass and bacteria.
5) It produces a constant biogas rate due to the regular substrate input.

However, this method presents certain drawbacks, including high energy expenditures, accumulation of VFAs and scum formation at high OLR, washout of active biomass at low HRT, and the technical challenges associated with the loading pump [136,137]. On the other hand, batch digesters also offer several benefits, such as technical simplicity, low capital cost, minimal maintenance requirements, no need for stirring or pumping, high operational flexibility, better biomass retention, and low parasitic energy loss [98,126]. Nevertheless, the batch-feeding mode may face challenges, including clogging and poor process stability at high OLR, suboptimal biogas yield, and the need for a large land area.

6. Physical and chemical factors affecting pretreatment efficiency

Biomass recalcitrance to enzymatic digestibility and microbial degradation, as well as the effectiveness of pretreatment, are directly influenced by the inherent structural and compositional properties of native lignocellulosic materials. These properties can be broadly classified into two groups [138]: i) Physical direct factors, primarily porosity and accessible surface area. ii) Indirect factors, which include specific surface area, pore volume, chemical compositions of cellulose, hemicellulose, and acetyl groups, DP, and cellulose crystallinity.

6.1. Cellulose crystallinity (CrI)

The crystallinity of cellulose has long been recognized as a crucial structural parameter that hinders the enzymatic hydrolysis rate [139]. This factor plays a significant role in enzyme accessibility to cellulose, thereby influencing the efficiency of adsorbed cellulase [140]. However, reduced crystallinity efficiently improves cellulase adsorption, the hydrolysis rate of lignocellulosic biomass, and biogas yield. The cellulose crystallinity is characterized by (CI), which represents the percentage of the cellulose crystalline fraction [141]. This parameter can be determined using various methods, including infrared spectroscopy, X-ray diffraction (often called the Segal method), solid-state ^{13}C nuclear magnetic resonance, and FTIR [142]. It can also be calculated using the formula provided below [143]:

$$\mathrm{CrI}\ (\%) = \frac{I002 - \mathrm{Iam}}{\mathrm{I002}} \times 100$$

I_{002} represents the diffraction peak intensity at $2\theta \approx 22.5^{\circ}$, and $I_{amorphous}$ refers to the intensity of amorphous zone diffraction at $2\theta \approx 18^{\circ}$.

The recognized mechanism for enzymatic cellulose degradation involves the synergistic action of a wide array of cellulolytic enzymes, commonly referred to as 'cellulases'. These enzymes are secreted by aerobic and anaerobic bacteria, as well as eukaryotes (fungi), to break down β-1,4-glycosidic bonds in cellulose [144]. Cellulases, O-glucoside hydrolases (GH), are considered biocatalysts consisting mainly of three endo- and exo-acting enzymes [145].

1) Endoglucanases (EGL, EC 3.2.1.4) act by randomly cleaving intramolecular β-1,4-glycosidic linkages in amorphous cellulose filaments, thereby creating new chain ends that are subsequently targeted by exoglucanases.
2) Exoglucanases, or cellobiohydrolases (CBH, EC 3.2.1.91, EC 3.2.1.74), attack the cellulose chain ends released by endoglucanases to produce predominantly cellobiose molecules as the main product.
3) β –glucosidases, also known as cellobiases (BGL, EC 3.2.1.21), split cellobiose into free glucose units.

6.2. Degree of polymerization (DP)

The term “degree of polymerization" denotes the average number (n) of glycosidic rings in the cellulose molecule chain, typically ranging from 10,000 glucopyranose units in woody biomass to 15,000 in native cotton [49]. Similar to cellulose crystallinity, the polymerization degree is also a crucial factor influencing LCB recalcitrance. Long cellulose chains (high DP) are characterized by a relatively high number of hydrogen bonds, which restrict the enzyme’s access to cellulose surfaces and result in difficult hydrolysis. On the other hand, smaller chains (low DP) have a weaker hydrogen-bonding system, thus enhancing enzymatic degradation by allowing cellulose to be more amenable to enzyme access [146]. The most frequently employed techniques for measuring cellulose DP include GPC, which is a well-established technology for analyzing both the number- and weight-average DP and providing in-depth information about the nature and length of the cellulose chains [147,148].

6.3. Accessible surface area, porosity, and particle size

The accessible surface area is one of the key factors that influence the rate of enzymatic and bacterial hydrolysis. Nonetheless, ASA is not an independent factor, since it is closely related to substrate specific surface area, pore size, particle size, and pore volume [143]. Cellulose fibers encompass two distinct surface areas: outer and inner. The outer surface area pertains to the particle’s shape, size, length, and width. Conversely, the inner surface (pore surface) is linked to the capillary structure of lignocelluloses and the number of feedstock cracks and pores [149]. Several studies have demonstrated that particle size reduction through mechanical deconstruction (ball milling, extrusion, grinding) could increase the external surface area, or SSA. This, in turn, unlocks the compact structure of LCB and improves cellulose-enzymes affinity, thus accelerating the hydrolysis rate [146,150]. Yu et al. [151] pointed out that mechanical pulverization of corn stover disrupted its compact structure and effectively reduced particle size, thereby improving enzymatic hydrolysis. Similarly, Lu et al. [152] reported that ball milling reduced the particle size, leading to a direct increase in the external surface area of cellulose and ASA, and making the cellulose more reactive and accessible.

7. Pretreatment approaches of agricultural biomass

The pretreatment of residues is a crucial step for enhancing substrate digestibility within the bioreactor, ensuring effective solubilization of holocellulosic and lignin components, and ultimately improving biogas production [153]. Generally, the primary objective of pretreatment is to mitigate the inherent recalcitrance of LCB by.

a) Disrupting the persistant carbohydrate-lignin shields that impede enzymatic and microbial access to holocellulosic components [141].
b) Shrinking the crystalline structure and DP of cellulose, and breaking the lignin seal/sheath.
c) Increasing the ASA for microbial and enzymatic attack or increasing the substrate’s porosity [138].

These modifications contribute to the acceleration of the hydrolysis (rate-limiting) phase of the AcoD process, as hydrolytic microorganisms take an extended period to enzymatically break down the biomass in the absence of pretreatment [154]. In a large-scale AcoD plant, this implies that the retention time of digesters could be reduced, which may enhance both the anaerobic digestion rate and extent, ultimately leading to increased methane production [155]. The pretreatment technology must satisfy the following criteria to be effective and economically feasible.

- Low operational cost and capital investment
- Minimum production of toxic and inhibitory by-products that could hinder the growth of fermentative microorganisms and could negatively affect the downstream bioprocess of biogas generation. This includes weak acids (levulinic acid, formic acid), phenols (alcohols, ketones), and furan derivatives (furfural, 5-hydroxymethyl furfural) [156].
- Minimal energy expenditure.
- Avoidance of the total destruction of hemicellulose and cellulose fractions [149].

Pretreatment technologies are broadly categorized into biological (fungi, enzymes, ensiling, microbial consortium), physical (e.g., comminution, microwave, ultrasound), chemical (alkali, ozonolysis, acid, oxidation), and combinatorial (steam explosion, ammonia fiber/freeze explosion) pretreatments [19].

7.1. Biological pretreatments

Among various technologies, biological pretreatment is gaining increasing significance from both environmental and economic standpoints. It is an eco-friendly, green, and sustainable technology that requires low capital cost, no chemical inputs, and minimum energy expenditure [157]. This approach encompasses fungal, enzymatic, ensiling, and microbial consortium pretreatment.

7.1.1. Fungi

The fungal pretreatment method employs on wood-decaying or xylophagous fungi, which are sorted into three categories: brown-rot fungi (BRF), soft-rot fungi (SRF), and white-rot fungi (WRF) [36]. WRF are accredited as the most proficient basidiomycetes for

biomass bio-digestibility due to their high selectivity and affinity for lignin depolymerization over holocellulose, as well as their powerful delignification enzymatic system [158,159]. To trigger lignin decay, WRF such as Ceriporiopsis subvermispora, Cyathus stercoreus, Pycnoporus cinnarbarinus, Pleurotus ostreatus, Clostridium butyricum, Phanerochaete chrysosporium, Aspergillus oryza, Dichomitus squalens, Coriolus versicolor, secrete a ligninolytic system encompassing two major classes of enzymes: i) lignin-oxidizing enzymes, and ii) lignin-degrading auxiliary or accessory enzymes [160]. The first class comprises extracellular oxidative enzymes (or oxidoreductases), namely manganese peroxidase (hydrogen-peroxide dependent oxidoreductases, MnP), versatile peroxidase (VP), lignin peroxidase or 'ligninase' (LiP), and laccase or phenol oxidases (benzenediol: oxygen oxidoreductases, EC 1.10.3.2) [161].

i. As a heme-containing glycoprotein, MnP (EC 1.11.1.13) facilitates the oxidation of phenolic lignin compounds, ultimately resulting in the release of CO_2 [162].
ii. LiP (EC 1.11.1.14) hydrolyzes mostly non-phenolic methoxyl-substituted lignin moieties (>90% of the polymer) in the presence of H_2O_2 [40].
iii. VP (EC 1.11.1.16) combines the catalytic characteristics of MnP and LiP, enabling the oxidation of both phenolic and non-phenolic aromatic compounds [163].
iv. Laccases are blue, multi-copper oxidoreductases (BMCO) that catalyze the oxidation of a broad range of non-phenolic compounds, as well as phenolic ones, through the reduction of O_2 to H_2O [161,163].

The second class of auxiliary enzymes includes benzoquinone reductase, glyoxal oxidase, galactose oxidase, veratryl alcohol oxidase, vanillyl alcohol oxidase, and glucose oxidase. These enzymes function as supporting enzymes to mitigate specifically the methoxy radicals generated by laccase, LiP, and MnP [46,164]. In contrast to WRF, BRF (Laetiporus portentosus, Gloeophyllum trabeum, Fomitopsis pinicola, Tyromyces balsemeus), as well as SRF (Deuteromycetes, Ascomycetes), are more efficient in depolymerizing cellulose and hemicellulose fractions, while subtly altering the lignin structure [157]. Moisture content, fungal strain, aeration (O_2 concentration), pretreatment incubation conditions (pH, temperature, and time), and nutrient supplementation (N, Cu^{2+}, and Mn^{2+}) are considered the most critical process parameters affecting fungal growth and metabolism during pretreatment [165–167]. Several laboratory tests assessed the effect of fungal pretreatment on the advancement of methane yield. Villa Gomez et al. [168] demonstrated that pretreating bean straw through solid-state fermentation using Pleurotus ostreatus (WRF) for 28 days at 30 mg fungus/g straw significantly enhanced the total methane yield. This improvement was attributed to cellulose solubilization, and maximum hemicellulose (44%) and lignin (18%) decay, compared to untreated BS. Furthermore, the influence of pretreatment on rice straw using three fungal strains—Pleurotus ostreatus (PO), Phanerochaete chrysosporium (PC), and Ganoderma lucidum (GL) was studied over a period of 5 weeks [159]. The results indicated that the three WRFs significantly degraded rice straw, but with varying rates and efficiencies. The methane yields of (PC), (PO), and (GL) were 2.22, 1.64, and 1.88-fold higher, respectively, than the untreated sample. The improvement in methane yield is attributed to the disentangling of cellulosic fibers and the breakage of lignin microfibrils in rice straw through extracellular enzymes secreted by PC, PO and GL. These findings indicate that implementing this study on a full-scale, with a total production of 769.65 MT of rice straw, has the potential to increase biomethane by 140 billion m^3 [159].

7.1.2. Ensiling

Ensiling is a promising biochemical technology and a logical choice to ensure the availability of seasonal biomass throughout the year, as it preserves up to 90% of a plant's energy [169]. This approach is based on solid-state lactic acid fermentation and serves both as a farm-scale storage procedure for wet or partially dry biomass under an anoxic or microaerophilic environment and as a biological pretreatment method prior to AcoD [170,171]. The ensiling process occurs in four successive phases with competitive microorganisms, namely LAB, fungi (molds and yeasts), endospore-forming bacteria (bacilli and clostridia), and coliform bacteria [172]. The first phase is an aerobic phase that begins right after filling and sealing the silo. During this phase, the biomass and, more importantly, the aerobic bacteria continue respiration by consuming sugars and generating water and carbon dioxide until the trapped oxygen is fully exhausted [173]. The next step is the fermentation phase, during which homofermentative LAB ferment WSC into lactic acid. Heterofermentative LAB, on the other hand, produce ethanol, lactic acid, and acetic acid, thereby reducing the pH value to approximately 4.0. This low pH inhibits the growth of deleterious yeasts and microbes [172,174]. By maintaining anaerobic conditions at a low pH, the majority of microorganisms in the second phase gradually decrease, resulting in stable silage dominated by lactic acid bacteria until the feed-out period or aerobic spoilage phase. During this phase, when the ensiled biomass is reintroduced to an aerobic environment, previously inhibited fungi and bacteria are reactivated [175,176]. To improve the stability of the ensiling process stability and minimize dry matter and energy losses from the original material [173], utilizing additives proves to be a resourceful strategy, especially for crop biomass with a low content of fermentable carbohydrates [177]. Additives can be categorized into two types: chemical and biological. Chemical-based additives, known as fermentation inhibitors, include inorganic acids, organic acids (formic acid, benzoic acids, formaldehyde), and a mixture of sodium nitrite and hexamethylenetetramine. These additives act as antimicrobial and fungistatic agents, restricting the growth of yeasts and undesirable microbiota, such as putrefactive and butyric acid bacteria [177,178]. Biological additives or fermentation stimulants (e.g., LAB inoculants, cellulolytic, and hemicellulolytic enzymes) are more commonly utilized in agricultural ensiling compared to chemical additives due to their non-corrosion nature and safe handling [173]. Various lab-scale experiments were conducted to evaluate the influence of ensiling on CH_4 production. Janke et al. [179] showed that ensiling sugarcane stalks and sugarcane trash without additives increased methane potential by 24.0% and 23.4%, respectively. However, the addition of a combination of sugarcane molasses with a commercial silage inoculant (Lactobacillus plantarum and Pediococcus pentosaseous) to sugarcane trash enhanced methane potential by 51.4%. This improvement can be attributed to the formation of

organic acids (acetate and lactate) that contribute to reducing the silage pH level [179].

7.1.3. Enzymatic pretreatment

Enzymatic pretreatment has proven to be a promising approach for optimizing AcoD performance, boosting biomethane yield, and reducing feedstock viscosity. This process involves the use of crude, purified, or semi-purified enzymes, predominantly derived from hydrolytic or ligninolytic enzymes such as cellulases, endo-xylanases, α-amylases, pectinases, β-glucosidases, cellobiases, laccases, peroxidases, and dextranases [36,39,180]. Koupaie et al. [181] stated that enzymatic pretreatment can potentially enhance methane yield by 15–35%. However, the efficiency of enzymatic degradation on biomass hinges on various factors, including the composition of the feedstock, enzyme concentration, incubation time, mixing method and speed, pH level, and temperature [182]. Pérez-Rodríguez et al. [183] found that enzymatic pretreatment of corn cob with an enzyme cocktail derived from Aspergillus terreus for 7 days improved the methane yield by 14%.

7.1.4. Microbial consortium (MCons)

MCons pretreatment approach requires the utilization of mixed microbes, commonly selected from ecological niches such as soil, herbivore's gut, livestock dung, and biogas slurry, where they form communities or 'guilds' that synergistically degrade complex substrates [184]. The use of microbial consortia, whether natural or synthetic (genetically engineered), represents a more effective and successful alternative for degrading lignocelluloses compared to single strains, as monocultures are more sensitive to environmental fluctuations and have limited metabolic and degrading capabilities [185,186]. Zhong et al. [187] showed that pretreatment of pig manure and rice straw with cellulolytic microflora, including Clostridium, Petrobacter, Defluviitalea, and Paenibacillus, enhanced the cumulative methane production by 45% and reduced the lag phase from 2.43 to 1.79 days. Furthermore, Zhong et al. [188] demonstrated that biological pretreatment of corn straw using a freeze-dried powder containing pure strains of yeast and cellulolytic bacteria at 20 °C for 15 days improved biogas and methane yields by 33.07% and 75.57%, respectively, compared to untreated straw.

7.1.5. Microaeration pretreatment

Microaeration, also known as oxygenation, moderate oxygenation, limited aeration, or micro-oxygenation [189], is a promising technology that can be applied as a pretreatment prior to AcoD, during digestion, and as an upgrading method for biogas desulphurization [190]. Microaeration pretreatment involves the creation of a system with a low dissolved oxygen concentration that overlaps aerobic and anaerobic environments, by injecting small doses of oxygen or air (generally between 0.1 and 1 mg/L) into the pretreatment tank [191]. This technology promotes the diversity and growth of facultative hydrolytic bacteria and acidogens, thereby accelerating the rate of the hydrolysis step and enhancing VFA production. Additionally, it scavenges hydrogen sulfide, contributes to improved system stability, and can increase biogas volume by 30%–216% [192,193]. However, when microaeration is employed as a pre-treatment approach, the residual oxygen remaining in the mixture to be introduced into the biodigester can inhibit microbial activity and growth if it is present in excess [193]. Xu et al. [194] showed that mesophilic microaerobic pretreatment of corn straw with an oxygen rate of 5 ml/g $VS_{substrate}$ increased methane yield by 17.35% compared to untreated straw. This improvement resulted from a decrease in its degree of crystallinity and the hydrolysis of holocellulose and lignin. Cao et al. [195] investigated the impact of microaeration on mono-digestion of swine manure and co-digestion of swine manure with corn silage for VFA production. They observed that microaeration increased VFAs concentration by 20.3% and shortened the time required to reach the maximum from 18 days to 10 days. To date, the literature predominantly emphasizes the effects of microaeration when implemented within anaerobic bioreactors. However, investigations into microaeration as a pretreatment method are largely confined to laboratory-scale studies, with minimal research conducted on microaeration pretreatment preceding the co-digestion of livestock waste and crop residues. Several crucial parameters must be taken into account during microaeration, including.

i) Microaeration intensity (oxygen dosing rate). According to César et al. [193], the most suitable units for measuring this parameter are volume of air/mass of (TS) min for dry and semi-dry AD, and volume of air/volume of liquid min for substrates with TS $< 10\%$.
ii) Dosing frequency, which can be one time, continuous, or intermittent [196].
iii) HRT in the pretreatment reactor, which can vary between 1 and 6 days for liquid waste (e.g., blackwater, sewage, mixed sludge, industrial wastewater, etc.) and 14 days for solid waste (lignocellulosic waste, municipal solid waste, etc.) [193].

7.2. Physical pretreatments

Physical pretreatment serves as the initial step in the feedstock conditioning chain following harvesting. It is typically conducted before any other biological or chemical treatment approaches to reduce the particle size of the substrate and facilitate its treatment and handling. Milling or grinding, extrusion, steam-explosion (autohydrolysis), irradiation (e.g., ultrasonication, microwave), LHW are commonly employed physical techniques to pretreat agricultural wastes for anaerobic digestion.

7.2.1. Mechanical pulverization/comminution

The primary objective of communition is to alter the biomass shape and particle size. This alteration directly increases the bulk density and total ASA of the feedstock, reduces the extent of cellulose polymerization degree, and facilitates the solubilization of fermentable components [197]. These parameters contribute to the advancement of feedstock biodegradability and the acceleration of hydrolysis and acidogenesis steps [198]. Mechanical pretreatment includes grinding or milling (ball-milling/beating, knife milling,

rod-millling, vibro energy grinding, hammer milling, attrition/disk milling), chipping, briquetting, and shredding [199]. The choice of the appropriate grinding equipment depends on the biomass moisture content. For instance, attrition, two-roll, knife, and hammer mills are generally employed only for comminuting dry feedstocks (moisture content >10–15%). On the other hand, extrusion and colloid milling are applicable for wet biomass comminution (moisture content >15–20%). Both wet and dry substrates can be pretreated by ball and vibro energy ball mills [200]. Among other alternative methods, mechanical pretreatment is considered the most suitable technique for preprocessing agricultural wastes on an industrial scale. However, the high energy requirement remains the major shortcoming of this technology [201]. The required energy for comminution varies depending on the type of device used, lignocellulose properties (moisture content, bulk density, composition, ductility, and strength), as well as the initial and final particle sizes [141]. Moreover, it has been reported that excessive particle size reduction may lead to the buildup of VFAs in the digester, resulting in reduced biogas production [198,202].

7.2.2. Liquid hot water

LHW-based biomass pretreatment, also termed hydrothermolysis, aquasolv, aqueous fractionation, and uncatalyzed solvolysis [203], is widely accepted as a green, cost-effective, pollutant/chemical-free, and environmentally benign processing technology. During this process, biomass is heated using hot water as the main reaction medium at high temperatures (ranging from 140 to 220 °C) and pressures (1–5 MPa) to keep water in the liquid phase [204]. These conditions lead to the dissociation of water into hydroxide ions (OH^-) and hydronium ions (H_3O^+). The generated H_3O^+ facilitates the detachment of uronic acid and O-acetyl substitutions in hemicellulose, thereby forming organic acids responsible for the depolymerization of hemicellulose into monosaccharides. Subsequently, these monosaccharides can be further broken down into aldehydes (furfural from pentoses and 5-Hydroxymethyl furfural from hexoses), which exhibit inhibitory effects on microorganisms [141]. To prevent the formation of inhibitors throughout the process, the pH should be kept at a neutral level through the incorporation of alkalis (e.g., sodium hydroxide) [44,205].

7.2.3. Steam explosion (auto-hydrolysis, SE)

The SE process is defined as a thermo-mechano-chemical industrially scalable pretreatment approach that induces the disruption of lignocellulose fiber bundles through steam heating (thermo), shear forces (mechano, owing to pressure drop and moisture expansion), and auto-hydrolysis of acetyl groups in hemicellulose (chemical) [206,207]. More specifically, the feedstock is heated with saturated steam at a relatively high pressure (1–3.5 MPa) and elevated temperature (ranging from 180 to 260 °C) for a short reaction time (several seconds up to a few minutes). Subsequently, it undergoes explosive decompression due to the swift reduction of pressure to atmospheric level [12,208], resulting in the disaggregation of the lignin-carbohydrate complex, an increase in the substrate's porosity, and partial depolymerization of lignin via the homolytic split of β-*O*-4 ether linkages [209]. These modifications were found to influence the rate of biomass degradation during the anaerobic process, digestion retention time, and the specific yields of biogas and methane [210].

7.2.4. Extrusion

The extrusion process, as a thermo-mechanical method, has proven to be a more efficient and advantageous pretreatment method compared to mechanical comminution [48]. In this process, the moistened biomass material is subjected to heating, mixing, and high mechanical shearing, driven by the rotation speed of the extruder screw blades. This results in the disintegration of cellulose and lignin fibers, the lysing of plant cells, a higher water-holding capacity, and larger specific areas, which facilitate digestion within the fermenter [183,211]. For the efficient enhancement of biogas production, extrusion can be applied in combination with chemical pretreatment. For instance, Zhang et al. [212] stated that pretreatment of rice straw firstly by extrusion (physicochemical pretreatment) and secondly by sodium hydroxide (chemical pretreatment) at 35 °C for 48h, increased its methane production by 54.0% and improved the efficiency of energy recovery from 38.9% to 59.9%.

7.2.5. Ultrasound/Sonication

The ultrasonication pretreatment of biomass is an emerging mechanical and green technology based on the acoustic cavitation principle, defined as the spontaneous formation, expansion, and ultimate implosive collapse of microbubbles generated by supplying high-frequency (>20 KHz) ultrasonic radiation [213,214]. The violent collapse generates shock waves at a temperature of approximately 5000 K and a pressure exceeding 50 MPa, resulting in both physical and chemical effects within the liquid medium [5,215]. The physical (mechanoacoustic) effect is a hydromechanical shear force that leads to the formation of intense convection and turbulence [216]. Meanwhile, the chemical (sonochemical) effect generates short-life-time oxidizing radicals, such as H∘ and OH∘, through the dissociation of vapor molecules trapped in the microbubbles [217,218]. The combination of these two effects reduces cellulose crystallinity and DP, breaks glycosidic linkages in the LCB network, and splits lignin and polysaccharide portions of biomass by cleaving β-*O*-4 and α-*O*-4 bonds in lignin [5,219].

7.3. Chemical pretreatments

7.3.1. Acid pretreatment

Acid hydrolysis has proven to be a potent chemical pretreatment approach for LCB. The major reaction occurring during this process is the solubilization of hemicellulose, particularly xylan, along with partial lignin and cellulose depolymerization through the cleavage of van der Waals forces, covalent, and hydrogen bonds, in the biomass [220,221]. The pretreatment, employing either organic acids (maleic acid, oxalic acid, and formic acid) or mineral acids (boric acid, nitric acid, phosphoric acid, hydrochloric acid,

and sulfuric acid), can be carried out using dilute acid (e.g. 0.1%) at elevated temperatures (>200 °C) or concentrated acid (30–70%) at low temperatures (<50 °C) [5,19], Nevertheless, concentrated acid is less commonly used for biomass pretreatment due to its toxicity, corrosiveness of reaction vessels, requirement of specialized non-metallic containers, and the generation of fermentation inhibitors such as phenolic acids and aldehydes [48].

7.3.2. Alkali pretreatment

Contrary to other chemical pretreatment techniques, alkali pretreatment is generally performed under mild reaction conditions (ambient temperature and pressure) without the need for complicated reactors, which makes it one of the most cost-effective and reliable pretreatment methods for on-farm application [207]. However, the need for a washing and neutralization step of the pretreated biomass prior to AD process remains one of the main downsides of this method [222]. NaOH, urea, KOH, Ba $(OH)_2$, ammonia solution, and lime are among the most widely employed alkali reagents for LCB pretreatment [223]. The major reactions occurring during this pretreatment involve intermolecular saponification and the cleavage of aryl-ether, C–C, and ester bonds between carbohydrate polymers and lignin, resulting in biomass swelling, increase of porosity and internal surface area, and cellulose decrystallization [224]. Alkaline pretreatment is considered highly effective for biomass with low lignin content, specifically herbaceous plants and agricultural leftovers [225]. Jaffar et al. [153] conducted a comparative analysis on the effects of rice straw pretreatment, especially on its biodegradability during AD and the fertilizing value of the resulting digestate, using varying concentrations of KOH (1%, 3%, 6%, 9%). The results revealed that a 6% concentration of KOH yielded the most significant impact on the biogas production, showing a notable improvement of 45%. Additionally, the digestate residue exhibited higher fertilizer values for phosphorus (6.6%), calcium (22%), magnesium (16%), and potassium (138%) compared to untreated digestate. Furthermore, Mancini et al. [226] reported that alkaline (NaOH) pretreatment of wheat straw led to a significant improvement in biomethane production kinetics, resulting in a 15% increase in cumulative biomethane production compared to alternative pretreatments (organosolv and organic solvent N-methylmorpholine N-oxide).

8. Discussion and future recommendations

The integration of livestock waste and crop residues as feedstocks for anaerobic co-digestion holds significant promise in the realms of renewable energy, waste management, and agricultural sustainability, particularly in rural areas. This approach stands out as a technology that is aligned with the principles of the circular economy, converting livestock waste and crop residues from liabilities to valuable assets, with positive implications for both environmental stewardship and agricultural economy. As can be seen from the literature, a considerable amount of research has delved into estimating the annual potential of crop residues and livestock manure generated for bioenergy production, using various theoretical models. This estimation is indeed a critical aspect when assessing the feasibility of implementing AcoD plants. However, theoretical estimations often rely on generalized data that may overlook regional or seasonal variations, leading to inaccuracies in the results (i.e., underestimation or overestimation of the actual potential). Hence, integrating more localized and context-specific data, considering factors such as weather conditions, soil quality, waste management practices, and accounting for local variations, can contribute to more accurate assessments that reflect the actual potential of livestock waste or crop residues in a particular region or farm. The implementation and development of small- and large-scale agricultural plants, while offering significant benefits in terms of waste management and renewable energy production, is subject to various technical, economic, political, and social constraints. On the technical front, the primary challenge lies in the variability of feedstock quality and composition. Animal manure and crop residues can exhibit fluctuations in their organic content, nutrient composition, and moisture levels due to seasonal changes, crop type variations, soil conditions, livestock diet and management, and farming practices, making it challenging to maintain optimal AcoD conditions and achieve a consistent balance for microbial activity. Thus, sophisticated monitoring and control systems are required to optimize operating conditions and prevent process upsets. Furthermore, the chemical

Table 3
Pros and cons of pretreatment techniques [36,70,220,230,231].

Pretreatment method	Pros	Cons
Biological	• Eco-friendly and sustainable process •No release of inhibitory compounds due to mild operation conditions (atmospheric pressure, low temperatures) •Low capital and operating cost requirements •Less energy and chemical requirement •Degradation of both hemicellulose and lignin	•Long incubation time •Slow rate of delignification •It requires careful control operation conditions •Lower reaction rates •large space requirement
Physical	•Low environmental impacts •Increase of the ASA •Easy handling especially for mechanical pretreatment •No need for chemical catalysts	•High power, energy and water expenditure •High maintenance cost
Chemical	•Effective solubilization due to reduced cellulose crystallinity and DP •It increases accessibility to cellulose •faster rates and better efficiencies	•High amount and cost of reagents •It mandates expensive corrosion-resistant materials due to caustic properties of chemicals especially acids •Requirement of neutralization and detoxification steps •Release of fermentation inhibitors during the process

Table 4
Effects of biological, chemical and physical pretreatments on biogas yield and composition of crop residues and animal waste.

Pretreatment methods	Feedstock	Process conditions		Outcomes	References
		Pretreatment	AD		
Fungi	Wheat straw	Ligninolytic fungi, batch, 28 °C for 7 days	Thermophilic condition (50 °C), 6 weeks	•48.2% decrease of lignin content •enhancement of biogas yield 5 times compared with untreated WS •407.1% increase in methane yield	[232]
	Bean straw	WRF Pleurotus ostreatus, 30 °C (1, 10 and 30 mg fungus/g straw) for 14, 21 and 28 days	Batch, 30 °C	•Maximum lignin (18%) and hemicellulose (44%) degradation at 30 mg fungus/g straw •Highest total methane yield (38 CH4/g VS loaded)	[168]
	Rice straw	WRF Pleurotus ostreatus (PO), Phanerochaete chrysosporium (PC), Ganoderma lucidum (GL), 30 °C, 5 weeks	–	•2.22-fold increase in methane yield with (PC) pretreated RS. •(GL) and (PO) resulted in 1.88-fold, 1.64-fold increase in methane yield, respectively.	[159]
	Corn stover	Phanerochaete chrysosporium, SSF at 28 °C, 30 days	Batch, 37 °C, 30 days	•Highest methane yield (265 mL/g VS) compared to untreated (215.5 mL/g VS); 49.5 mL/g VS increased biomethane	[233]
	Dairy cattle manure	Pleurotus ostreatus, 28 °C, 14 days Pleurotus ostreatus, 2–17 °C min and 10–31 °C max, 2 months	37 °C	7% increase in methane production 111% increase in methane production	[234]
Ensiling	Mixture of wheat straw + Sugar beet leaves (after chopping)	Lab-scale: storage in vacuum bags in a barn (5–15 °C) for two months and subsequently at room temperature (20 ± 0.5 °C) for 7 months.	Batch, 37 °C, 61 days	•BMP increase ranged from 19 to 34%	[171]
		Pilot-scale: storage in silos with volume of 2.6 m^3 for approx. 6 months (177–189 days)	Batch, 38–39 °C, 58 days	•BMP increase ranged from 18 to 32%	
	Sugarcane stalks (SCS) Sugarcane trash (SCT)	Storage in vacuumed and double sealed silos in the dark under ambient temperature of 20–25 °C for 70 days	38 °C, 30 days	•Increase in methane potential by 24.0% (without additives) •Increase in methane potential by 23.4% (without additives) •Addition of sugarcane molasses + commercial silage inoculant resulted in 51.4% higher methane potential than ensiled SCT (without additives)	[179]
	Mixture of fresh cattle manure + wheat straw	3.5 L airtight round plastic storage drums, 25 ± 2 °C for 120 days	Batch, 35 °C	•Co-ensiling led to 67% methane potential losses (without additives) •Limitation of energy losses to 25% after formic acid addition •Full preservation of methane potential after glucose addition	[235]
Enzymatic	Chicken manure	Mixture of commercial enzymes (Onozuka R-10 and Macerozyme R-10), 40 °C, 24h	Batch, 37 °C for 21 days	•35% increase in biogas production compared to the control without enzymatic pre-treatment	[236]
	Corn stover	Laccase (LA) and peroxidases (manganese peroxidase + versatile peroxidase), 30 °C for 0, 6, 12 and 24 h	–	•25% increase in biomethane production after 24 h (with laccase) •17% increase in biomethane production after 6h (with peroxidases) •Treatment with both enzyme groups increased biomethane production with 16% and 14% after respectively 6 and 24 h of treatment	[237]
Microbial consortia	Rice straw (RS) + Pig manure (PM)	Cellulolytic microflora (Clostridium, Petrobacter, Defluviitalea, and Paenibacillus), 55 °C, 30 h	35 °C, 15–20 days	•62.20, 59.58, and 33.77% decrease in the content of cellulose, hemicellulose, and lignin, respectively •45% icrease in the cumulative methane production of RS and PM (342.35 ml (g-VS)$^{-1}$) compared to untreated (236.03 ml·(g-VS)$^{-1}$)	[187]
	Corn straw (CS)	Mixed microbes: Phanerochaete chrysosporium, Coriolus versicolor,	35 °C, 30 days	•131.6% increase in methane yield	[238]

(*continued on next page*)

Table 4 (*continued*)

Pretreatment methods	Feedstock	Process conditions		Outcomes	References
		Pretreatment	AD		
		Trichoderma viride, Aspergillus niger, Gloeophyllum trabeum, Bacillus circulans, Pseudomonas aeruginosa and Streptomyces badius; 30 °C for 14 days			
Mechanical pretrearment	giant reed stems (Arundo Donax)	Two stages (hammer mill + pin mill) dry milling device with working capacity up to 1,2 t h^{-1}	38 °C; 28 days	137.7% gain in the cumulative methane production	[239]
	Wheat straw			49.1% gain in the cumulative methane production	
	Wheat straw	Knife mill; particle size reduction to 2 mm	40 °C, 60 days	83.5% improvement in methane yield	[240]
	Barley straw	Knife mill; particle size reduction to 5 mm		54.2% improvement in methane yield	
	Horse manure	Prototype ball mill, rotational speeds (6,10,14 rpm)	37 °C, 35 days	Increase in specific methane yield by 37.3 %	[241]
	Cattle manure	Mobile and fixed hammer mills; wet sieving	35 °C	Increase in methane production rate by 15% and 27% for mobile hammer mill and fixed hammer mill, respectively	[242]
Steam explosion	Rice straw	200 °C; 120 s	38 °C; 21 days	• 51 % increase in biogas production • 13.72 % and 16.79% increases in degradation rates of cellulose and hemicellulose, respectively, as compared to untreated straw	[243]
	Corn stover	160 °C; 2 min	mesophilic conditions (37.5 °C); 49 days	22 % improvement in methane yield	[17]
	Pig manure	170 °C; 30 min	35.1 °C	206.9 % improvement in methane yield	[244]
Liquid hot water	Wheat straw	175 °C for 30 min; pressure: 0.4–2.5 MPa	Mesophilic conditions 36 °C	62.9 % increase in methane yield	[245]
Extrusion	Rice straw	twin-screw extruder (55 kW, 120 rpm)	mesophilic temperature 37 °C; 60–90 days	72.2 % increase in methane yield	[246]
Acid pretreatment	Wheat straw	Dilute H_2SO_4 (1%), 121 °C, 10–120 min	mesophilic, 37 °C, 30 days	16% increase in methane yield	[247]
	Corn straw	Dilute H_2SO_4, HCl, CH_3COOH and H_2O_2 (1,2,3,4%), 25 °C, 7 days	mesophilic, 37 °C, 35 days	115.4% increase in methane yield with 3% H_2O_2	[248]
	Cow manure	Peracetic acid (0.01–0.10 g/g VS), 6 and 12h	38 °C, 45 days	39.1% increase in biogas production	[249]
	Dairy cow manure	HCl (2%), 37 °C, 12h	37 °C	Increase in methane potential by 20.6%	[250]
Alkali pretreatment	Wheat straw	Ammonia (2,4,6%), 35 °C, 7 days	35 °C, 60 days	52% increase in methane yield	[251]
	Rice straw	Ammonia (2,4,6%), 30 °C, 7 days	35 °C, 55 days	28.55% increase in methane yield at 4% ammonia concentration	[252]
	Cow manure	Calcium oxide (0.05–0.15 g gTS-1); 6–12h	38 °C	26 % increase in biogas production	[249]
	Dairy cow manure	NaOH (10%), 100 °C, 5min	37 °C	Increase in methane potential by 23.6%	[250]
	Chicken litter	NaOH (5%), 120 °C, 90 min	37 °C	Up to 50% improvement in biogas production	[253]

properties (pH, moisture level, carbohydrates, toxicity, crude protein, etc.) and physical composition (particle size, density, porosity, etc.) of the input substrates should be thoroughly studied before the AcoD process since they have a significant impact on the overall performance of the conversion process, the quality of the end products (biogas and digestate), and, more importantly, the microbial ecosystems in the bio-digester. Specifically, the analytical characterization of feedstocks prior to the process enables the selection of appropriate co-digestion conjugates and the determination of the optimal mixing ratio of raw materials. The microbiological aspect is also one of the most important factors influencing both digestion stability and efficiency. Although there are several studies in the literature describing the microbial pathways of the co-digestion process, there is still a deficiency in the thorough understanding of the biochemistry and microbial ecology in anaerobic digesters when it comes to the co-digestion of various livestock wastes and ligno-cellulosic residues. Therefore, more in-depth research should be conducted to develop instructive approaches that may reveal a complete and accurate identification of all microbial species involved in the process, their complex structure, their relationship with feedstock compositions, their metabolic capacity, and their quantitative and qualitative relationships to the functional performance of the digester. Moreover, it is instrumental to deploy innovative technologies (artificiel intelligence, kinetic modeling, machine learning)

and robotic tools (device mobiles, smart cameras) that can predict the dynamical behavior of substrates during conversion and monitor fluctuations in the microbial environment and their impact on other operational parameters. The inclusion of a pretreatment stage is beneficial for improving co-digestion efficiency and optimizing biogas yield by breaking down the recalcitrant structure of the feedstock and rendering polysaccharides more accessible to enzymes and microorganisms. Physical pretreatments are widely applied on an industrial scale and pose no inherent risk of forming inhibitory compounds. However, they are high-severity methods and involve high power consumption and maintenance costs, making the entire process expensive. Chemical pretreatment processes reduce cellulose crystallinity and moderately boost biogas production, but they require expensive reagents, generate caustic intermediary products, and necessitate large quantities of water for washing the substrates before introducing them into the reactor. On the other hand, biological pretreatment is one of the most promising technologies for enhancing biogas production efficiency, as it is environmentally friendly, requires milder reaction conditions, and produces fewer side-stream products. However, its application to animal manure is underexplored in the literature. Compared to single treatments, multi-stage (combined) pretreatment techniques significantly increase biomethane production, decrease pretreatment severity, and reduce the formation of inhibitory compounds. Table 3 recapitulates the advantages and drawbacks of the aforementioned pretreatments. Nevertheless, comparing pretreatment technologies with each other proves challenging due to the non-standardized conditions such as the substrate's composition and structure, pretreatment operational conditions, and AcoD process types. The effect of pretreatment on both the innate composition of the feedstock and methane yield improvement has been extensively investigated in the literature at bench-scale utilizing BMP assays **(Table 4)**. While this methodology proves effective in determining optimal pretreatment conditions and assessing substrate degradation rates, as well as the ultimate methane yield, it remains challenging to extrapolate laboratory test findings and improvements to industrial-scale, continuously loaded AcoD systems. Hence, the implementation of specific pretreatment techniques for agricultural residues at the industrial level often constitutes a significant impediment [227]. Moreover, for both energetic viability and economic profitability, the input for pretreatments (in terms of extra energy and cost requirements) must be significantly lower than the output energy (biogas, heat) and economic gain (represented by methane yield increase) [40,228]. The effectiveness and selection of the appropriate pretreatment method for lignocellulosic biomass or livestock residues are influenced by several crucial parameters, including the physico-chemical properties of the substrate, pretreatment complexity, pre-treatment operating conditions, the formation of inhibitory products, economic and energy costs, environmental impacts, cost considerations (cost of chemicals, thermal/-electrical energy input, cost of biological agents), and the methane improvement achieved [229]. Therefore, an extended and interdisciplinary investigation into techno-economic and life cycle assessments, energy and exergy analysis, and exergo-environmental evaluation is necessary to scrutinize the environmental sustainability, economic and energy feasibility of pretreatment methods on an industrial scale. This will help bridge the gap between laboratory findings and the real-world application of pretreatment technologies.

From an economic standpoint, it is necessary to consider the collection sites for residual biomass, the transportation to the processing plant, and the subsequent storage. Furthermore, upfront capital expenditure for digester infrastructure and ongoing operational costs, including maintenance, raw material procurement, and monitoring, contribute to the economic challenges. The economic feasibility of AcoD facilities is also closely tied to governmental policies, incentives, and subsidies. Government policies play a crucial role in shaping the regulatory framework, incentives, and support mechanisms that can either promote or hinder the implementation of AcoD technology involving crop residues and livestock waste by agricultural enterprises and waste management facilities. Governments can establish grant programs, subsidies, feed-in tariffs, and tax credits for entities investing in the waste management and conversion sectors. These financial incentives stimulate the uptake of sustainable practices, bolster the economic feasibility of the biogas plant, offset the upfront capital costs and operational expenses associated with setting up the co-digestion facility, and encourage farm owners to invest, particularly in remote areas where animal waste and crop residues are generated in substantial quantities. China, Denmark, and Italy promote the production of biogas and biomethane from agricultural waste. In the United Kingdom, AD facilities primarily employ municipal biowaste, sewage sludge, and wastewater as main feedstocks. This is attributed to regulations that restrict the utilization of energy crops as raw materials to a maximum of 50% [254]. To achieve lasting change, awareness campaigns and educational programs are also essential. Local farmers must be apprised about the environmental and health implications of inadequate waste management and empowered with knowledge about sustainable alternatives. This can instill a sense of responsibility and ownership in adopting more eco-friendly practices.

9. Conclusion

The valorization of lignocellulosic field-based residues and livestock waste as significant bioenergy resources through anaerobic co-digestion represents a potent waste management technology and a greener bioenergy production route. This approach contributes to long-term energy security, reduces GHG emissions, and mitigates environmental and health threats exacerbated by conventional agricultural waste disposal practices such as open-air burning, landfills, and random piling. It also enhances biological and physico-chemical soil quality through the land application of digestate as a bio-fertilizer and soil conditioner. Additionally, the synergistic effect of co-digesting these substrates offers better process stability, adjusts nutrient imbalances, improves buffering ability, stabilizes the C/N ratio, dilutes toxicity from inhibitory compounds, and ultimately increases CH_4 yield. Looking forward, ongoing research, technological innovations in reactor design, process monitoring, and feedstock pretreatment methods, as well as comprehensive assessments encompassing technical, economic, and environmental aspects, hold the potential to further optimize process parameters, increase biogas yields, and improve digestion stability and efficiency.

Data availability statement

No data was used for the research described in the article.

CRediT authorship contribution statement

Imane Adnane: Writing – review & editing, Writing – original draft, Visualization, Conceptualization. **Hamza Taoumi:** Writing – review & editing, Writing – original draft, Visualization, Conceptualization. **Karim Elouahabi:** Writing – review & editing, Writing – original draft, Conceptualization. **Khadija Lahrech:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Conceptualization. **Abdellah Oulmekki:** Writing – review & editing, Conceptualization, Writing – original draft.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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