

Review

Feedstock-Adaptive Valorisation of Agro-Industrial Organic Waste: Pretreatment, Biological Conversion, and Resource Recovery

Yurii Syromiatnykov ^{1,2,*}  and Marcin Niemiec ^{3,*}

¹ Institute of Soil and Plant Sciences, Faculty of Agriculture and Food Technology, Latvia University of Life Sciences and Technologies (LBTU), LV-3001 Jelgava, Latvia

² Department of Crop Production Systems, Institute of Agrobiotechnologies and Food Safety, Samarkand State University named after Sharof Rashidov, 15 University Boulevard, Samarkand 140104, Uzbekistan

³ Department of Agricultural and Environmental Chemistry, Faculty of Agriculture and Economics, University of Agriculture in Krakow, 31-120 Kraków, Poland

* Correspondence: yurii.syromiatnykov@lbtu.lv (Y.S.); marcin.niemiec@urk.edu.pl (M.N.)

Abstract

Agro-industrial organic wastes differ markedly in moisture, structure, biochemical composition, nutrient value, and contaminant risk, so pretreatment should be selected from the limiting feedstock constraint rather than from a generic technology hierarchy. This critical narrative review examines feedstock-adaptive valorisation routes centred on anaerobic digestion and subsequent recovery of energy, nutrients, carbon-rich fractions, fertilisers, humic-like materials, and plant-biostimulant products. Unlike technology-centred syntheses, the review uses the dominant feedstock constraint as a common comparison axis across pretreatment, biological stability, and product qualification. Mechanical, mechanochemical, cavitation-based, thermal, chemical, and biological treatments are compared in terms of the barrier they remove, their energy and material burden, downstream biological response, and recovered-product quality. The evidence shows that particle-size reduction, solubilisation, or methane yield alone is insufficient for process selection. A minimum-sufficient-pretreatment heuristic is therefore proposed: treatment intensity is increased only until a predeclared conversion or product-quality objective is reached while stability, safety, and positive incremental net benefit are retained. Zero pretreatment remains a valid outcome when concentration, co-digestion, feeding control, biomass retention, or another intervention better addresses the dominant constraint. The framework also highlights the need for harmonised energy reporting, mass balance, continuous validation, and product-specific qualification before laboratory improvements are translated to industrial operation.



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1. Introduction

The starting point for this review is straightforward but often overlooked in technology-centred comparisons: agro-industrial organic waste is not a uniform feedstock. Swine and cattle manure are commonly handled as wet slurries, crop residues are bulky and structurally resistant, food-processing residues can acidify rapidly, and slaughterhouse

streams may contain large protein and lipid fractions. The operating problem therefore changes with the material. Moisture, ash, fibre, lignin, protein and lipid content, C/N ratio, salinity, particle structure, and contaminant load affect storage, pumping, pretreatment, microbial conversion, and, ultimately, whether the recovered material can be used safely in agriculture. Broad circular-bioeconomy reviews describe many conversion pathways, but they are commonly organised around technologies or end products rather than around the feedstock property that actually limits conversion [1–3].

A one-product view of valorisation is equally restrictive. Anaerobic digestion can recover part of the organic carbon as biogas, yet most nitrogen, phosphorus, potassium, mineral matter, and a substantial fraction of the residual organic carbon remain in the digestate. Depending on feedstock history and post-treatment, this stream may be separated, concentrated, stabilised, or used as a precursor for fertiliser, organomineral formulations, humic-like products, and other agricultural inputs. Integrated biorefineries extend the same material logic to volatile fatty acids, microbial biomass, polymers, and other value-added products. The practical question is therefore not which isolated unit operation gives the largest yield, but which sequence preserves the greatest recoverable value without sacrificing process stability or product safety [4–6].

Pretreatment sits at the centre of that decision because it can either remove a genuine rate-limiting barrier or simply add energy, chemicals, and maintenance. Mechanical size reduction can improve access to lignocellulosic surfaces; ultrasound and hydrodynamic cavitation can promote disintegration; hydrothermal treatment can increase solubilisation; and chemical or biological methods can modify lignin and hemicellulose selectively. None of these mechanisms guarantees a net process gain. Severe treatment may increase soluble COD while simultaneously generating inhibitors, increasing salt load, producing side streams that require management, or consuming more energy than the additional methane or product value can recover. Reviews of agricultural-waste biorefineries and coupled digestion-thermochemical systems increasingly recognise these trade-offs, but comparison remains difficult because functional units and system boundaries differ widely [7–9].

Anaerobic digestion is used here as the principal biological conversion route because it is technically established for wet organic residues and produces a secondary stream with clear nutrient-recovery potential. Composting, acidogenic fermentation, microbial upgrading, and thermochemical coupling are included when they extend, compete with, or reshape the digestion pathway. This gives the review a deliberate boundary: it is broader than a methane-focused assessment, but it does not attempt to catalogue every possible waste-conversion technology. The narrower focus makes it possible to examine the pretreatment–microbiology–product-quality interface in enough detail to remain useful for agricultural and agro-industrial waste management.

Recent reviews have mapped emerging pretreatment technologies, integrated anaerobic digestion–pyrolysis routes, combined biogas-digestate valorisation, contaminant control, and separate digestate treatment [10–14]. These syntheses establish the breadth of available unit operations, but they generally treat pretreatment, biological conversion, and digestate management as adjacent subjects. The added value of the present review is the use of the dominant feedstock constraint as a single decision axis across pretreatment, process stability, and recovered-product qualification. The synthesis also formulates a minimum-sufficient-pretreatment rule in which zero pretreatment remains a valid outcome when another intervention addresses the limiting constraint more effectively.

The aim of this review was to determine how feedstock characteristics should guide both the choice and the intensity of pretreatment, the subsequent biological conversion strategy, and the cascaded recovery of energy, nutrients, carbon-rich fractions, and agricultural bioproducts. Three questions are considered together: which material constraint

is being removed, whether the downstream benefit exceeds the treatment burden, and whether the residual or recovered product remains suitable for its intended use. From this synthesis, a minimum-sufficient-pretreatment heuristic is formulated as a testable engineering framework. It is not presented as a universally validated optimisation model; rather, it defines the measurements and decision points that would be needed to validate such a strategy for a specific feedstock and process.

2. Scope and Literature-Selection Approach

This review was conceived as a critical narrative technology review rather than a systematic review or meta-analysis; a PRISMA flow diagram was therefore not used. The literature search was updated through 22 August 2026 and used Crossref metadata, PubMed, Google Scholar, targeted journal and publisher searches, and backward and forward citation tracking. Scopus and Web of Science were not primary search platforms. This is treated as a limitation of coverage, and the manuscript no longer claims that searches in those databases would be immaterial. To reduce the risk of omission, the final verification incorporated recent reviews spanning pretreatment, integrated conversion, biogas-digestate valorisation, contaminant control, and digestate treatment [10–14], followed by checks of their directly relevant primary sources.

The principal publication period was 2010–2026. This interval was chosen to capture the development of contemporary pretreatment, conversion, recovery, and scale-up technologies while still allowing earlier work to be retained where it remained technically important. Several older publications were therefore included because they established mechanisms that continue to underpin current process interpretation, reported benchmark engineering data, or provided continuous and full-scale observations that have not been superseded by more recent studies. In this respect, publication age was treated as an indicator of context rather than as an automatic exclusion criterion.

Searches were organised into reproducible concept blocks. The principal query was (“agro-industrial waste” OR manure OR “livestock waste” OR “slaughterhouse waste” OR “spent mushroom substrate” OR “crop residue” OR “food-processing waste”) AND (“anaerobic digestion” OR co-digestion) AND (pretreat* OR milling OR mechanochemical OR “vortex layer reactor” OR ultrasound OR cavitation OR hydrothermal OR alkaline OR biological). Recovery searches used (digestate OR “anaerobic digestate”) AND (“nutrient recovery” OR fertiliser OR biofertiliser OR biostimulant OR “humic substances” OR biochar). Scale-up searches combined “anaerobic digestion” with (“life-cycle assessment” OR “techno-economic assessment” OR “pilot scale” OR “full scale”). Safety searches added pathogen, “heavy metal”, pharmaceutical, microplastic, antibiotic, or “antimicrobial resistance”. The terms were adapted only for database syntax and spelling variants. Backward and forward citation tracking was then used to identify primary studies supporting quantitative or mechanistic claims.

A paper was retained when it made a clear contribution to at least one of the questions examined in the review. Quantitative process studies were included when the feedstock and operating conditions were described well enough to permit interpretation. Mechanistic studies were retained when they helped explain treatment response, inhibition, or process limitations. Evidence on downstream products was included where it addressed product quality, contaminant transfer, energy balance, life-cycle performance, or techno-economic feasibility. Particular attention was also given to pilot- and full-scale studies because these provide information on operability that cannot be inferred reliably from laboratory experiments alone.

Publications were excluded when the feedstock or treatment conditions were described too poorly to support a defensible interpretation, when the item was a corrigendum

containing no independent scientific evidence, or when the subject fell outside the questions addressed in the review. Evidence from municipal sludge and other non-agricultural matrices was used selectively. Such studies were retained only where they addressed a mechanism or engineering issue that could reasonably be transferred across matrices, for example, pretreatment energy demand, membrane separation, contaminant behaviour, or antimicrobial resistance. Even in these cases, numerical performance values were not treated as directly transferable to agricultural or agro-industrial waste streams.

The same caution was applied to mechanical and agronomic studies conducted outside waste-processing systems. These publications were used only where they offered comparator evidence for general engineering behaviour, equipment design, material transport, wear, or field-validation principles. Their numerical results were not extrapolated to organic-waste treatment. This distinction is important because similarity in a physical or biological mechanism does not imply equivalence in process performance. Feedstock composition, moisture content, rheology, particle structure, microbial activity, and contaminant load can all alter the response of a treatment system.

The final evidence base comprised 208 peer-reviewed publications together with Regulation (EU) 2019/1009. All peer-reviewed publications have a DOI, and bibliographic records were verified against DOI metadata. Because the literature search combined iterative database searching with backward and forward citation tracking, it was not treated as a closed systematic-search dataset for retrospective record-count reconstruction.

The strength of individual studies was judged according to the type of conclusion they could reasonably support. Continuous and full-scale measurements were given the greatest weight when assessing operability, auxiliary energy demand, maintenance, long-term stability, and practical scale-up. Controlled comparative experiments were particularly useful for identifying treatment-response relationships under defined conditions. Batch biochemical methane-potential tests were interpreted more cautiously. They provide valuable evidence of biological potential and may help clarify mechanisms, but they do not reproduce the hydraulic, microbial, or operational behaviour of continuous reactors.

Review papers were useful for mapping research directions and identifying relevant primary studies, but quantitative statements were linked to direct experimental or operational evidence whenever this was available. Comparator matrices were handled in the same way. Where evidence from sewage sludge, microalgae, or another material outside the main feedstock scope was used, its role was stated explicitly and the limits of transfer were considered in the interpretation. A mechanism observed in one matrix may help explain why a response occurs in another, but it does not by itself demonstrate that the same conversion efficiency, energy demand, product quality, or scale-up performance will be obtained.

3. Feedstock Heterogeneity as the Starting Point for Process Selection

3.1. *Livestock Manures*

Livestock manures have a common biological origin, but their composition and handling properties differ enough to alter the process bottleneck. Dairy cattle manure often contains slowly degradable plant fibre and bedding and may show only moderate methane productivity unless co-substrates increase the biodegradable organic load. Swine manure is generally more homogeneous and readily pumpable, although dilution can make transport and reactor heating inefficient. Poultry manure is nutrient-dense but can impose severe ammonia, salinity, and solids-handling constraints. Meta-analysis and cold-region studies show that methane yield and energy recovery depend not only on animal species but also on the collection system, storage history, temperature, retention time, and co-digestion strategy [15–18].

These differences lead to different pretreatment priorities. Fibre-rich cattle manure may benefit from moderate mechanical conditioning when physical accessibility limits hydrolysis. A dilute swine-manure stream, by contrast, may gain more from concentration, co-digestion, or improved organic loading than from intensive disintegration. Poultry manure is commonly constrained by nitrogen loading rather than by particle structure. Carbonaceous additives, alkaline operation, and suitable co-substrates can alter microbial performance under ammonia stress, but any benefit has to be weighed against the additional material requirement, cost, and downstream consequences [19–21].

Manure also carries upstream management history into the recovered-product stage. Veterinary compounds may be degraded incompletely, and digestate composition reflects both animal-production practices and digestion conditions. For that reason, a useful feedstock dossier should record veterinary inputs, bedding, wash water, storage duration, solids, nutrient concentration, and other source-specific risks before pretreatment is selected [22–24]. The practical diagnosis is whether performance is primarily limited by physical accessibility, insufficient organic loading, ammonia stress, or product-safety constraints. Each diagnosis points to a different intervention.

3.2. Crop Residues and Spent Mushroom Substrate

Crop residues and spent mushroom substrate are dominated by lignocellulosic structure, in which cellulose and hemicellulose are partly protected by lignin and by the spatial organisation of the cell wall. High volatile-solids content therefore does not mean that the methane potential is readily accessible. Particle size, crystallinity, lignin shielding, low bulk density, water absorption, and slow hydrolysis can all become limiting. Bioaugmentation and biological-hydrolysis studies show that improved access to structural carbohydrates can accelerate conversion, although the response depends on substrate composition and on whether the added hydrolytic function persists under digester conditions [25–28].

Spent mushroom substrate should not be treated as equivalent to untreated straw. Fungal cultivation has already modified part of the lignocellulosic matrix, while residual mycelium, mineral additives, moisture, and the cultivation substrate influence handling and digestion. Co-digestion with manure can improve moisture balance, C/N ratio, inoculation, and buffering. In practice, this means that a moderate mechanical or mechanochemical step may be sufficient for some spent mushroom substrates even where an untreated crop residue would require stronger delignification. Reviews of lignocellulosic digestion consistently report a broad response range rather than a single optimal pretreatment [29–32].

Hydrothermal, organosolv, alkaline, biological, and combined treatments can all improve accessibility, but they do so with different selectivities and burdens. Increasing severity may remove lignin and solubilise hemicellulose, while at the same time producing phenolic or furan-derived inhibitors, losing soluble carbon in wash streams, or creating a chemical-recovery requirement. For lignocellulosic feedstocks, the relevant endpoints are therefore methane productivity, retention-time reduction, recoverable carbon, and net energy, not fibre disruption in isolation [33–36].

3.3. Slaughterhouse and Lipid-Rich Animal-Processing Wastes

Slaughterhouse waste, dissolved-air-flotation sludge, fats, and related animal-processing residues may have high theoretical methane potential because lipids are energy dense. Their practical digestion is limited by a different set of mechanisms: long-chain fatty acids (LCFAs) can accumulate, adsorb to biomass, interfere with mass transfer, and inhibit microbial activity, while protein degradation can increase ammonia exposure. Foaming and biomass coating add further operational problems. A high biochemical methane potential therefore does not justify intensive pretreatment by itself. The rate at which lipids and

proteins are released must remain compatible with the syntrophic pathways needed for LCFA conversion [37–40].

The threshold at which inhibition becomes important is not fixed. It changes with feeding pattern, acclimation, reactor configuration, temperature, and microbial-community structure. Conductive materials and biofilm-supporting surfaces can improve electron transfer or biomass retention, but they do not remove the need for controlled loading. Co-digestion with carbohydrate-rich residues can reduce the effective lipid and nitrogen burden and may improve buffering at the same time [41–43].

This feedstock class exposes a weakness in technology-first pretreatment. Mechanical disruption can accelerate the transfer of fats and proteins into the liquid phase, yet that faster release can intensify the biological limitation rather than solve it. Slaughterhouse waste should therefore be assessed for inhibition before energy is invested in disintegration. In many cases, staged feeding, co-digestion, biomass retention, phase separation, or another loading-control strategy is the more rational first intervention [44–46].

3.4. Food-Processing Wastes and Rapidly Degradable Residues

Food and food-processing wastes are usually more readily biodegradable than ligno-cellulosic residues, but their composition varies widely in water, carbohydrates, proteins, lipids, acidity, and salt. Because hydrolysis can be fast, increasing organic loading too quickly may lead to VFA accumulation and pH decline before methanogenic capacity can respond. Characterisation studies and major reviews therefore place process stability, source contamination, collection logistics, and composition variability at the centre of design [47–50].

Two-stage digestion, controlled feeding, co-digestion, and buffering can decouple rapid acidogenesis from slower methanogenesis. The extra equipment and control effort are justified only when feedstock supply and product value support them. For many pumpable food-processing streams, contaminant removal and consistent homogenisation matter more than fine grinding. A pretreatment developed for fibrous crop residues should therefore not be transferred to food waste unless a physical accessibility limitation has actually been demonstrated [51–53].

Environmental assessments of food-waste digestion also show that source separation, transport, avoided disposal, process energy, and digestate management can change the final result substantially. The feedstock dossier should therefore extend beyond laboratory composition to annual volume, seasonality, packaging contamination, and the realistic destination of residual nutrients [54,55]. Table 1 summarises the initial feedstock classes and deliberately treats digestate as a downstream resource stream rather than as an incoming waste category.

Table 1. Initial agro-industrial feedstock classes, dominant constraints, and rational first interventions.

Initial Feedstock	Typical Characteristics	Dominant Constraint	Rational First Intervention	Primary Evidence Needed
Cattle/dairy manure	Wet slurry; variable bedding and fibre; moderate organic load	Slow fibre hydrolysis; dilution; seasonal variability	Characterise fibre and loading; compare conditioning with co-digestion	BMP plus continuous loading, energy, and digestate quality [16–18]
Swine manure	Pumpable and often dilute; nitrogen and veterinary-residue risks	Low organic loading and/or ammonia rather than particle size	Homogenisation, concentration, co-digestion, or mild treatment	Stable productivity, ammonia, net energy, and product safety [19–22]

Table 1. *Cont.*

Initial Feedstock	Typical Characteristics	Dominant Constraint	Rational First Intervention	Primary Evidence Needed
Poultry manure	High nitrogen, nutrients, salts, and solids	Free ammonia, salinity, and solids handling	Co-digestion, loading control, or ammonia recovery before disintegration	Free ammonia, salinity, acclimation, and nutrient recovery [15–17]
Crop residues	Dry, bulky lignocellulosic material; low bulk density	Lignin shielding, crystallinity, and slow hydrolysis	Size reduction or selective thermal, chemical, or biological treatment	Rate, ultimate yield, carbon recovery, and specific energy [29–32]
Spent mushroom substrate	Partly fungal-modified fibre; variable moisture and mineral additives	Residual recalcitrance and heterogeneous handling	Test mild mechanical/mechanochemical treatment and co-digestion first	Treatment-response curve, loading, mass balance, and energy balance [25–28]
Slaughterhouse/lipid-rich waste	High protein and lipid; high theoretical methane potential	LCFA and ammonia inhibition; foaming; biomass coating	Controlled feeding, co-digestion, biomass retention, or phase separation	LCFA profile, free ammonia, and stability under repeated feeding [37–40]
Food-processing waste	Rapidly degradable; variable acidity, salt, water, and packaging	Fast acidification and contamination rather than structural access	Sorting, homogenisation, staged feeding, buffering, or two-stage digestion	VFA/alkalinity, source variability, logistics, and product outlet [47–50]

Note: Digestate is treated as a downstream resource because it is generated after anaerobic conversion and its properties depend on both the original feedstock and the conversion history.

3.5. From Composition Data to a Process Constraint

Routine proximate analysis is necessary, but it is rarely enough to identify the operating constraint. Two materials with similar TS and VS may behave very differently if one contains readily accessible carbohydrates while the other contains lignin-protected fibre. Likewise, the same total nitrogen concentration can produce different free-ammonia exposure depending on pH and temperature. Structural measurements, particle-size distribution, soluble COD, alkalinity, VFA, lipid and protein fractions, electrical conductivity, and expected nutrient speciation provide the link between composition and mechanism. The analytical set should therefore be proportional to the suspected constraint rather than identical for every waste.

For process selection, it is useful to distinguish three types of constraints. Handling constraints include pumpability, bulk density, grit, packaging, and reliable transport through the active zone. Biological constraints include hydrolysis, rapid acidification, ammonia, long-chain fatty acids, salinity, and trace-nutrient imbalance. Product constraints include nutrient form, stability, contaminants, storage, and application logistics. Several may occur simultaneously, but the primary constraint should be stated before treatment begins. That diagnosis also determines the appropriate control. A milling experiment requires an unmilled control at equal loading; an inhibition-mitigation experiment requires a control that separates the effect of dilution or co-substrate addition from the treatment itself.

Feedstock variability is not background noise; it is part of the process design problem. A system designed around one composite sample may fail when bedding changes seasonally, cleaning-water pulses alter solids concentration, animal diets change, or food-waste packaging varies. Characterisation should therefore report the sampling period and the distribution of key properties, not only a mean. When source variability is of the same order as the expected treatment effect, adaptive blending or a defined operating envelope is more defensible than one fixed treatment intensity.

4. Pretreatment and Process-Intensification Technologies

4.1. Mechanical Size Reduction and Conditioning

Mechanical pretreatment includes cutting, shredding, maceration, refining, hammer milling, ball milling, and related operations. These methods reduce particle size, increase external surface area, and can improve mixing or disrupt fibre structure. Their strongest rationale is found where hydrolysis is limited by physical accessibility rather than by ammonia, salinity, LCFA, or another inhibitory mechanism. Laboratory studies on lignocellulosic and agricultural substrates show that mechanical treatment can accelerate methane formation, but the magnitude of the response depends strongly on the starting structure and the particle-size distribution ultimately achieved [56–59].

Smaller particles are not automatically better. Fine milling consumes electricity, increases wear, can worsen rheology at high solids, and in some cases gives only a modest increase in ultimate methane yield. The useful outcome may instead be a shorter lag phase, greater stable loading, improved pumpability, or lower mixing demand. Mechanical studies are therefore most informative when they report treatment energy together with the downstream operating response on a clearly defined fresh-mass, TS, or VS basis [60–63]. Equipment geometry, throughput, and active-zone configuration should be reported because they affect treatment intensity and reproducibility.

The landscape-management grass comparison by Heller et al. is a useful example because the same general objective produced very different energy outcomes. Cross-flow grinding and ball milling gave positive net balances of 12.3 and 21.4 kWh Mg^{−1} VS, respectively, whereas the tested Grasshopper harvesting/maceration route gave −18.3 kWh Mg^{−1} VS [61]. These values do not establish a universal ranking of equipment. They show instead that feedstock condition, baseline preparation, device efficiency, and the selected process boundary can determine whether mechanical treatment is sufficient, excessive, or energetically unjustified. Broader mechanical-pretreatment studies and reviews reach the same conclusion: the physical effect has to be interpreted together with energy demand and downstream conversion [64–66].

4.2. Mechanochemical Activation and the Vortex Layer Reactor

Mechanochemical activation combines impact, shear, abrasion, and repeated high-frequency contact, producing structural changes that go beyond conventional cutting. In a Vortex Layer Reactor (VLR), ferromagnetic elements move within a rotating electromagnetic field and create an intense interaction zone in which organic suspensions are homogenised and disintegrated. Local hydrodynamic effects may accompany the direct mechanical action. Some publications use the term Vortex Layer Apparatus (VLA) for the same operating principle. In this review, VLR is used consistently because the unit is treated as a continuous process-intensification reactor rather than merely as an apparatus.

The most directly relevant continuous-flow evidence compared swine manure, spent mushroom substrate, and slaughterhouse waste under the same VLR principle. Biogas production increased by 14% for swine manure and by 18% for spent mushroom substrate, whereas slaughterhouse waste showed no positive response. For the slaughterhouse stream, net energy efficiency fell by approximately 9%; the maximum net energy gain among the responsive substrates was 17.6%. The study reported electricity demand of 2.16–3.6 kWh Mg^{−1} of raw material and an approximate normalisation near 9 kWh Mg^{−1} TS [67]. These are study-reported values and should not be treated as universal device constants, because solids content and auxiliary demand remain site-specific.

A separate vortex-layer study showed that recirculation ratio alters the degree of pre-processing and the subsequent bioconversion response [68]. Taken together, the two studies support selective application rather than universal superiority. They also illustrate the minimum-sufficient-treatment principle clearly: treatment should stop at the lowest

intensity that produces a reproducible downstream benefit. Additional recirculation, or treating a feedstock whose dominant limitation is biological inhibition rather than accessibility, adds energy without necessarily changing the limiting mechanism.

4.3. Ultrasound and Hydrodynamic Cavitation

Ultrasound generates acoustic cavitation, microjets, shear, and local pressure changes that can disrupt cells and flocs. Hydrodynamic cavitation produces related collapse phenomena by forcing liquid through a constriction, nozzle, or rotor-stator geometry. Both approaches can increase soluble organic matter and alter viscosity, yet their efficiency depends on solids concentration, device geometry, exposure time, and scale. Evidence from sludge and microalgal systems is useful for understanding the mechanism but should not be transferred numerically to fibrous agricultural residues without validation [69,70].

The full-scale hydrodynamic-cavitation study in an agricultural biogas plant provides a stronger engineering reference point. A specific energy input of 470 kJ kg^{-1} TS was selected for implementation, and the plant recorded an increase in specific methane production of about 10%. Changes in rheology and particle size also contributed to lower energy demand for mixing, heating, and pumping. Increasing cavitation intensity beyond the useful range reduced the energetic attractiveness of the treatment [71].

Ultrasound studies on waste activated sludge show a more complex picture: sonication can increase solubilisation and improve digestion, but the response depends strongly on dose, temperature, solids concentration, and the operating conditions of the downstream reactor [72,73]. For that reason, high-energy laboratory sonication should not be compared directly with fresh-mass VLR values or with full-scale hydrodynamic cavitation unless the same denominator and system boundary can be reconstructed. Cavitation work on reed and dedicated crops further illustrates the transfer problem: a physical response demonstrated for bioethanol production or another substrate cannot be presented as a methane gain for manure without new evidence [74–76].

4.4. Thermal and Hydrothermal Treatment

Thermal and hydrothermal pretreatments transfer heat to the feedstock to promote cell disruption, alter viscosity, and solubilise organic polymers. Temperature, residence time, pressure, solids concentration, and the presence of catalysts or ammonia determine the resulting severity. Mild hydrothermal treatment may improve hydrolysis, whereas severe treatment can generate phenols, furans, Maillard products, or other compounds that reduce digestibility. Studies on microalgae, mixed food and garden waste, microplastic-containing sludge, and Sargassum-based systems show that the useful operating region is a severity window rather than the highest achievable temperature [77–80].

The energy and product consequences are feedstock-specific. Hydrothermal treatment of animal residues can generate hydrochar that alters subsequent digestion, while treatment of antibiotic-production residues may improve biodegradability but also changes the fate of residual compounds. Life-cycle work on food-waste digestion shows that an increase in methane conversion does not automatically translate into a better environmental balance when additional heat and infrastructure are included [81–83]. These examples make heat recovery, liquid by-products, and downstream product quality part of the pretreatment decision rather than secondary considerations.

Severity also affects what is released into the liquid phase. Studies of antibiotic residues and corn stover show that hydrothermal treatment can improve conversion within a useful operating range, while excessive severity may increase recalcitrant or inhibitory products [84,85]. For this reason, a hydrothermal result should be reported together with

carbon allocation, soluble-product composition, and the fate of compounds relevant to the final digestate or recovered product.

4.5. Chemical Pretreatment

Alkaline pretreatment can swell lignocellulosic material, disrupt ester linkages, and redistribute or remove lignin; acid treatment acts more strongly on hemicellulose but can generate inhibitory products and demands corrosion-resistant equipment. For manure and crop residues, chemical treatment should therefore be evaluated against reagent use, salt accumulation, neutralisation, wash-water generation, and the possible loss of soluble carbon. Corn stover, sludge, solid-waste, and Hybrid Pennisetum studies illustrate the alkaline response [86–96], while recent pretreatment reviews provide the broader evidence base for distinguishing acid and alkaline mechanisms [10,11].

Combined alkaline, high-pressure, electrical, or trace-element interventions can increase solubilisation or methanogenic activity, but interpretation becomes difficult when several variables change at the same time. A rise in soluble COD is not automatically beneficial; it can also accelerate VFA accumulation if the methanogenic step cannot use the released carbon. The useful chemical treatment is therefore the one that reduces the structural limitation without merely transferring the burden to salinity, inhibition, corrosion, or downstream product quality [90–93].

Experiments combining alkaline treatment with iron dosing, together with work on melanoidin formation and base-induced delignification, illustrate the two sides of severity. Structural modification can improve access to carbohydrate fractions, yet stronger thermal–alkaline conditions may also create recalcitrant or inhibitory products. The defensible dose is therefore the lowest one that reaches the predefined conversion target while retaining an acceptable material and energy balance and a usable digestate [94–96].

4.6. Biological and Enzymatic Pretreatment

Biological pretreatment uses fungi, hydrolytic bacteria, or enzymes to modify lignin and polysaccharides under comparatively mild conditions. It can reduce chemical demand and limit inhibitor formation, but treatment time, enzyme cost, contamination control, and loss of readily degradable carbon can restrict industrial use. Bioaugmentation and enzyme studies show another important point: the relevant function has to persist under digester conditions. Inoculation alone is not evidence of improved conversion.

These approaches are particularly attractive where a decentralised facility has storage time available but limited thermal or chemical infrastructure. Their performance is better expressed as an increase in methane productivity or conversion per unit treatment time and cost than as percentage lignin removal alone. Hybrid fungal-mechanical or enzymatic-mechanical routes may reduce the severity demanded from either component, but the net benefit still has to be demonstrated for the actual feedstock. Table 2 therefore keeps biological and enzymatic treatment separate from chemical pretreatment rather than collapsing them into one low-severity category.

Table 2. Pretreatment families separated by mechanism, suitable constraint, principal burden, and required endpoint.

Technology	Principal Mechanism	Most Relevant Constraint	Main Burden or Risk	Endpoint Required for Selection
Mechanical size reduction	Particle-size reduction, surface exposure, improved mixing	Bulky or fibre-accessibility-limited material	Electricity, wear, over-milling, altered rheology	Stable productivity or HRT benefit plus net energy [56–66]
Mechanochemical activation/VLR	Repeated impact, shear, abrasion, rapid homogenisation	Responsive heterogeneous slurry or partly altered fibre	Recirculation energy, wear; no benefit when inhibition dominates	Feedstock-specific gas/product response and net gain [67,68]

Table 2. Cont.

Technology	Principal Mechanism	Most Relevant Constraint	Main Burden or Risk	Endpoint Required for Selection
Ultrasound	Acoustic cavitation, microjets, cell/floc disruption	Cellular biomass or sludge comparator systems	Potentially high specific energy; scale and dose distribution	Energy-normalised response at representative solids [69,70,72,73]
Hydrodynamic cavitation	Pressure-induced bubble formation and collapse in flowing slurry	Pumpable feed where disintegration and rheology gains coexist	Pump energy, erosion, and strong geometry dependence	Full-process electricity, rheology, and methane response [71,74–76]
Thermal	Heating, cell disruption, viscosity change	Heat-responsive cellular or organic matrices	Heat demand, cooling, recalcitrant products	Recovered heat and usable-energy balance [77–80]
Hydrothermal	Pressurised water, polymer solubilisation, possible hydrochar formation	Lignocellulosic or selected wet residues	Pressure equipment, heat, inhibitors, liquid by-products	Severity window, carbon balance, and contaminant fate [81–85]
Alkaline	Fibre swelling, ester-bond cleavage, delignification	Lignin- or structure-limited residues	Reagent, salt, neutralisation, wash water	Dose–response, soluble-carbon fate, and digestate quality [86–96]
Acid	Hemicellulose hydrolysis and solubilisation	Selected lignocellulosic matrices	Corrosion, neutralisation, furans/phenolics	Inhibitor profile and net carbon recovery [10,11]
Biological/enzymatic	Selective lignin/polysaccharide modification	Storage-compatible, low-severity applications	Time, enzyme cost, contamination, carbon loss	Productivity gain per treatment time and cost [25–28]

Note: No pretreatment family is ranked as universally superior. Selection depends on the dominant limitation, the treatment intensity required to change it, and the same downstream functional objective.

4.7. Comparative Interpretation and Evidence Limits

Pretreatment families are most usefully compared by the function they perform, not by a single scale of severity. Cutting and maceration primarily alter particle handling and external surface area; mechanochemical treatment adds repeated impact and shear; cavitation concentrates disruptive events in a liquid phase; thermal and chemical routes alter solubility and polymer chemistry; and biological routes exchange high instantaneous energy input for time and selectivity [10,11]. Their effects overlap, but they are not interchangeable. A larger soluble-COD increase can help a hydrolysis-limited residue and be harmful to a rapidly acidifying waste.

Device energy is only one part of the process burden. Pretreatment may reduce downstream mixing, heating, pumping, or reactor volume, and those avoided loads should be measured rather than assumed. Conversely, neutralisation, wash water, cooling, dewatering, membrane replacement, grinding-media wear, rejected material, and product conditioning can exceed the nominal energy demand of the core device. Table 3 therefore separates reported treatment input from downstream response and retains the original denominator whenever the reported data do not support a common basis.

Table 3. Representative quantitative energy and performance evidence for pretreatment; original reporting bases are retained where harmonisation would require unsupported assumptions.

Feedstock/Technology	Reported Treatment Input	Basis Used for Comparison	Downstream Response	Scale	Interpretation	Source
Swine manure/VLR	2.16 kWh Mg ^{−1} raw for this substrate	≈9 kWh Mg ^{−1} TS (study-level value)	+14% biogas	Continuous-flow	Positive response; verify site-specific solids and auxiliaries	[67]
Spent mushroom substrate/VLR	2.7 kWh Mg ^{−1} raw for this substrate	≈9 kWh Mg ^{−1} TS (study-level value)	+18% biogas; maximum net energy gain 17.6% among responsive cases	Continuous-flow	Partly fungal-modified fibre responded to mechanochemical treatment	[67]

Table 3. Cont.

Feedstock/ Technology	Reported Treatment Input	Basis Used for Comparison	Downstream Response	Scale	Interpretation	Source
Slaughterhouse waste/VLR	3.6 kWh Mg ⁻¹ raw for this substrate	≈9 kWh Mg ⁻¹ TS (study-level value)	No positive biogas response; net energy efficiency ≈9% lower	Continuous-flow	Disintegration did not resolve the dominant biological limitation	[67]
Agricultural biogas feed/hydrodynamic cavitation	470 kJ kg ⁻¹ TS	≈131 kWh Mg ⁻¹ TS (converted from 470 kJ kg ⁻¹ TS)	≈10% increase in specific methane production; lower mix- ing/heating/pumping demand reported	Full-scale	Higher cavitation input reduced energetic attractiveness	[71]
Landscape grass/cross-flow grinder	Measured for the tested configuration	Net energy balance: +12.3 kWh Mg ⁻¹ VS	Positive combined methane-energy balance	Comparative study	Equipment and initial preparation controlled the result	[61]
Landscape grass/ball mill	Measured for the tested configuration	Net energy balance: +21.4 kWh Mg ⁻¹ VS	Positive combined methane-energy balance	Comparative study	Positive only under the stated feedstock and boundary	[61]
Landscape grass/Grasshopper route	Measured for the tested configuration	Net energy balance: −18.3 kWh Mg ⁻¹ VS	Negative combined balance	Comparative study	A mechanically effective route can remain energetically excessive	[61]
Wastewater algae/mechanical vs. hydrothermal	Study-specific; no common value retained	NR on a reconstructable mass basis	Hydrothermal, but not mechanical, pretreatment improved the reported energy balance	Laboratory	Energy recovery and kinetics, not disintegration alone, determined the outcome	[56]

Note: NR = not reported on a reconstructable common basis in this synthesis. Values are expressed on fresh-mass, TS, or VS bases only where the source data support that denominator. No universal conversion between these bases was imposed; comparisons should be made only within an identical denominator and process boundary.

The scale changes which mechanisms remain useful. Laboratory ultrasound can expose a small, uniform sample to a controlled dose, whereas an agricultural plant has to distribute energy through a variable slurry containing fibres, grit, and sometimes packaging residues. Hydrothermal systems can recover heat at scale, but they require pressure-rated equipment and reliable heat integration. Mild mechanical conditioning may appear less dramatic in a bottle test yet perform more consistently under continuous feeding. The relevant comparison is therefore between complete process configurations that deliver the same functional output, not between isolated devices operated at different solids concentrations or scales [64–66,71–73,81–83]. Figure 1 summarises this feedstock-to-decision sequence.

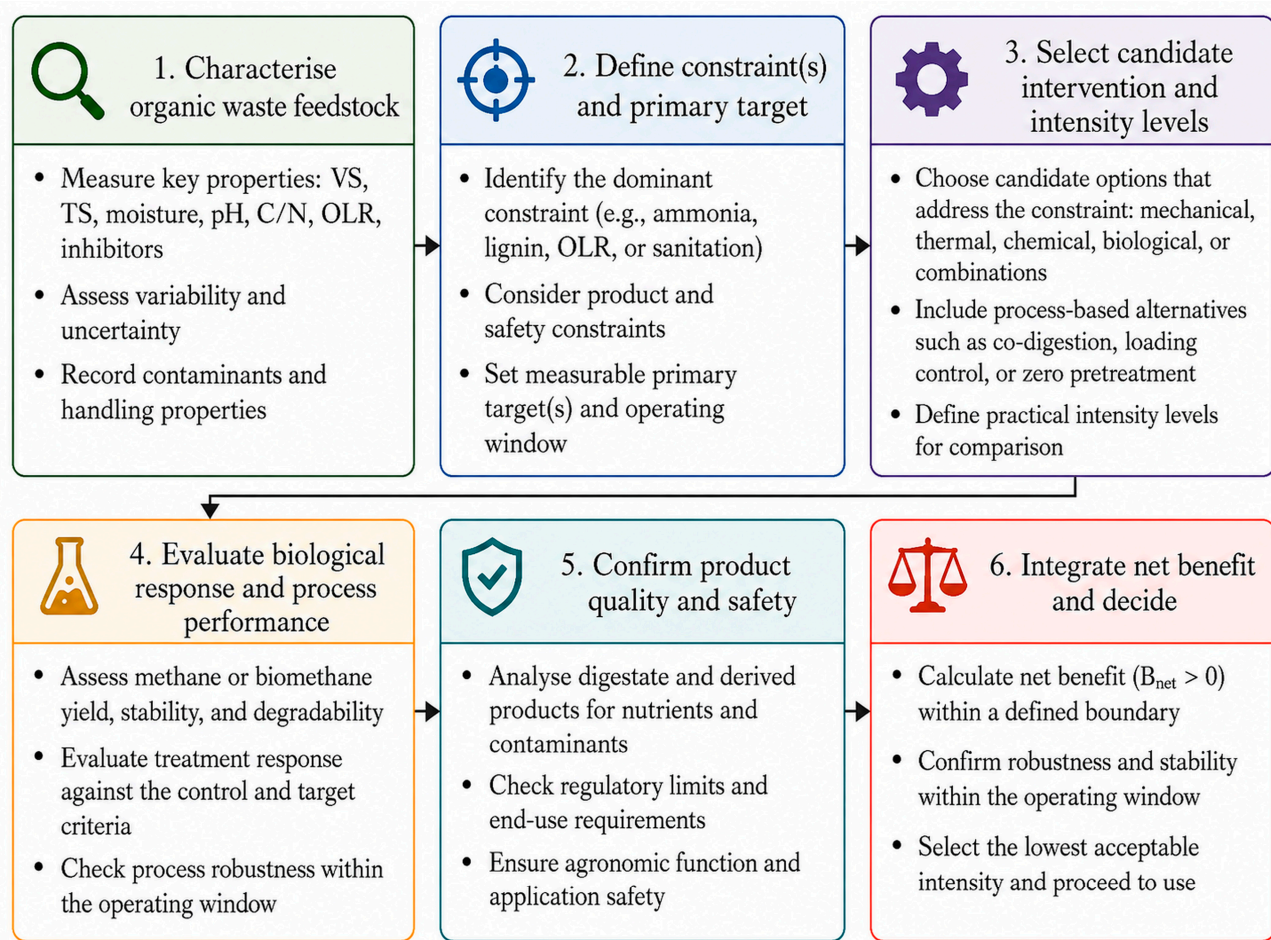


Figure 1. Feedstock-adaptive sequence linking material characterisation to the minimum sufficient pretreatment and the final net-benefit and safety decision.

5. Pretreatment–Bioconversion Interactions

5.1. Hydrolysis, Loading, and Volatile Fatty Acids

Hydrolysis is often rate-limiting for particulate and lignocellulosic substrates. Pretreatment can increase the soluble fraction and the contact area available to hydrolytic microorganisms, but the whole digestion chain has to remain balanced. Rapid release of sugars, amino acids, and fatty acids is useful only when acidogenic intermediates are consumed at a comparable rate. Micro-aeration, controlled loading, and staged operation have been used to limit VFA accumulation under high organic loading [97–100].

Total VFA concentration by itself is an incomplete stability indicator. Acetate, propionate, and butyrate differ in their kinetic and thermodynamic significance, and alkalinity determines how strongly a rise in acids affects pH. Continuous interpretation is therefore stronger when the VFA profile is considered together with alkalinity, gas composition, methane productivity, and organic loading. A high soluble-COD value immediately after pretreatment may indicate improved accessibility, but it may also be the first sign that substrate release has outrun downstream conversion [101–103].

Hydrolytic bioaugmentation, electrical-alkaline treatment, and broader process optimisation can accelerate conversion, yet performance should be reported through production rate and stable loading as well as through ultimate yield. A shorter hydraulic retention time is beneficial only if conversion efficiency and microbial retention remain adequate. Where solids retention time is coupled to HRT, excessive shortening increases washout risk;

biomass-retaining configurations such as attached-growth reactors or membrane systems require SRT to be evaluated separately [104–106].

5.2. Inhibition Mechanisms

Inhibition thresholds are strongly matrix-dependent. Phenolic compounds can interfere with anaerobic conversion, while ammonia toxicity depends on total ammoniacal nitrogen, pH, temperature, ionic strength, and microbial acclimation because the free-ammonia fraction is generally more inhibitory than ammonium. Reviews consequently report broad threshold ranges rather than one universally applicable concentration. Progressive loading tests and acclimation data are more informative than importing a single literature limit into a different feedstock [107–110].

Salinity and sulphide introduce another set of interactions. Sulfate, ammonium, ionic strength, and salt concentration can act together, and the dominant methanogenic pathway may shift under stress. Recent work on concentrated wastewater and nitrogen-rich feedstocks shows that combined inhibition cannot be interpreted reliably from one ion concentration alone [111–113]. Pretreatment that increases ionic strength or releases additional nitrogen should therefore be assessed against the operating envelope of the microbial community rather than against a generic threshold.

For nitrogen-rich streams, ammonia stripping and related removal strategies can be more effective than further disintegration when ammonia is the binding constraint [114–116]. Long-chain fatty acid (LCFA) inhibition follows a different mechanism: LCFAs adsorb to biomass, interfere with mass transfer and cellular function, and depend on syntrophic conversion before methanogenesis proceeds. Strategies developed for ammonia cannot be assumed to solve LCFA accumulation [117]. For lipid-rich feedstocks, loading pattern, biomass retention, and acclimation should therefore be considered before stronger pretreatment is selected.

5.3. Co-Digestion as a Corrective Strategy

Co-digestion can correct several feedstock imbalances at once. A suitable mixture can adjust C/N ratio, moisture, alkalinity, trace nutrients, and overall degradability while diluting inhibitory fractions. Studies combining cattle or dairy manure with food waste, crop residues, slaughterhouse waste, or fruit and vegetable residues report improved methane productivity and stability when the mixture is matched to microbial capacity rather than chosen solely from theoretical methane potential [118–121].

The word synergy should be used cautiously. A mixture is not synergistic simply because it produces more gas than a low-yield component. A defensible test compares the observed response with the weighted response expected from the individual substrates under equivalent loading, inoculum, and retention conditions. Semi-continuous evidence and mixed-feed studies, including agricultural and municipal organic wastes co-digested with dairy manure, are more useful than a single batch BMP when the question is whether the mixture remains stable under repeated feeding [122–124].

Co-digestion also changes the digestate. Nutrient concentration, salinity, organic stability, and contaminant dilution or redistribution determine whether the final material remains suitable for agricultural use. The best mixture is therefore not necessarily the one that maximises methane yield. In many practical systems, a slightly lower gas response may be preferable if it provides a more stable reactor and a better-quality downstream product [125,126].

5.4. Response Variables and Causal Interpretation

Pretreatment studies frequently report soluble COD, cumulative biogas, or methane yield as if these variables were interchangeable, but they answer different questions. Soluble COD measures release into the liquid phase; it does not prove that the released material

is biodegraded. Ultimate methane yield measures recoverable energy under the test conditions; it does not describe reaction rate or reactor capacity. Volumetric methane productivity reflects rate and loading but can mask falling conversion efficiency. A causal interpretation therefore needs a response chain from physical or chemical change, through hydrolysis and intermediates, to stable methane formation and the quality of the residual stream.

The baseline matters just as much as the treated condition. A large percentage gain over a poorly mixed or under-loaded control can exaggerate the apparent value of pretreatment, whereas a small gain over an already optimised control may still be industrially useful if it reduces viscosity or permits higher loading. Inoculum-to-substrate ratio, initial particle preparation, temperature, mixing, and adaptation history should be reported because they can amplify or suppress the observed treatment effect. Continuous validation is then needed to determine whether the response persists after the microbial community has adapted.

Mass balance is the most reliable safeguard against misleading performance claims. Carbon entering with the feed should be reconciled with methane, carbon dioxide, dissolved organics, biomass, and residual solids; nitrogen and phosphorus should be followed into liquid, solid, gaseous, and recovered-product streams. More methane is not a complete conversion gain if a substantial soluble-carbon loss in wash water is ignored. The same principle applies to contaminants: a lower concentration can reflect dilution or repartitioning rather than destruction.

6. Biological Conversion and Cascaded Resource Recovery

Anaerobic digestion remains the central biological route in this review because wet and mixed organic streams can be treated without an energy-intensive drying step. Hydrolysis, acidogenesis, acetogenesis, and methanogenesis convert part of the organic carbon into methane and carbon dioxide, whereas nutrients, minerals, microbial biomass, and recalcitrant organic matter remain largely in the digestate. Pretreatment can therefore alter two outputs simultaneously: gas productivity and the composition, dewaterability, stability, and agronomic value of the residual stream.

A cascaded process should begin by defining the intended outputs before the reactor and pretreatment are fixed. Energy-oriented plants may prioritise methane productivity and biomethane upgrading. Nutrient-oriented systems may accept a lower gas yield if the liquid fraction is suitable for nitrogen or phosphorus recovery. Carbon-oriented routes may stabilise or further transform the separated solids. The accounting has to prevent double counting: carbon converted to methane cannot simultaneously be credited as retained soil carbon.

Alternative biological routes broaden the product portfolio. Acidogenic fermentation can recover volatile fatty acids; selected agro-industrial streams can support microbial biomass, bacterial cellulose, polyhydroxyalkanoates, yeast products, or other biorefinery outputs. Composting can stabilise solids before land use, and pyrolysis of separated solids can be coupled with digestion when energy, nutrient, and contaminant balances justify the additional step. These options are treated as complementary branches of the wider biorefinery literature rather than as direct substitutes for anaerobic digestion in every wet feedstock.

The order of the cascade is constrained by material compatibility. Easily degradable carbon converted to methane is no longer available for a later fermentation product, and severe chemical pretreatment may compromise biological upgrading or agricultural use. Conversely, removing an inhibitory lipid fraction or recovering ammonia before or through a sidestream process may improve digestion and create a useful co-product. Figure 2 therefore shows a deliberate material sequence: feedstock, feedstock-specific pretreatment

where justified, anaerobic digestion as the principal biological route, biogas plus digestate, fractionation, recovered products, and agronomic verification.

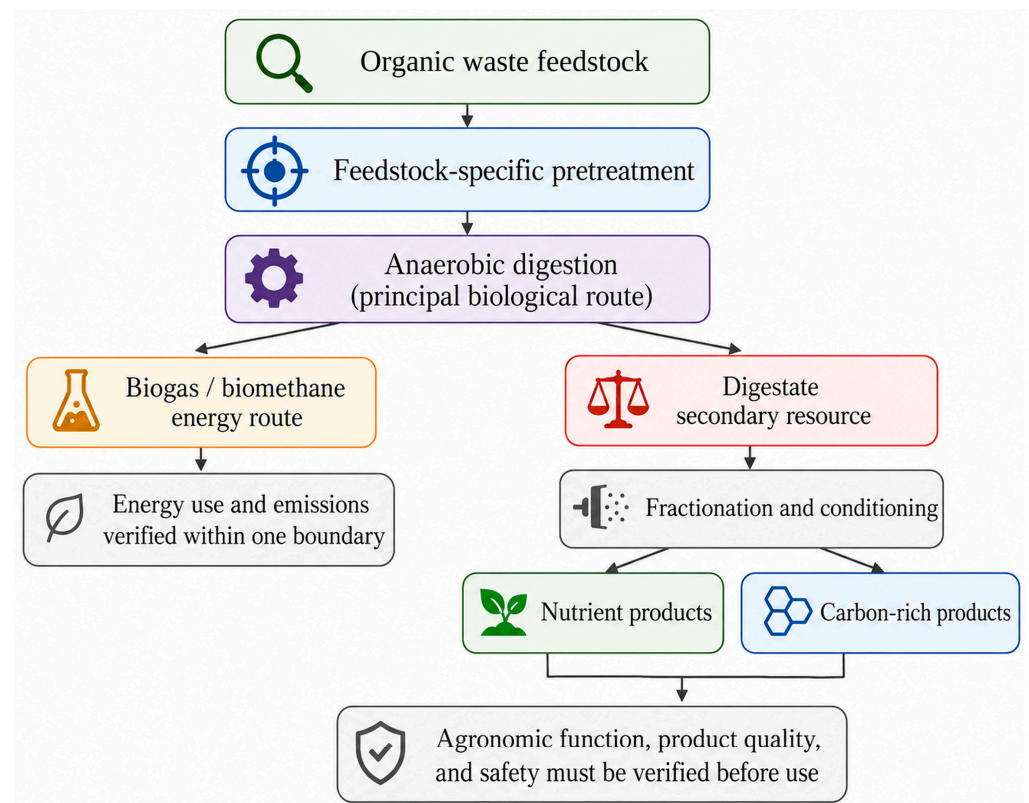


Figure 2. Material-flow sequence for cascaded valorisation. Anaerobic digestion is shown as the principal biological conversion route; digestate is generated downstream of digestion and is subsequently separated and conditioned for nutrient- or carbon-rich products.

Digestate is treated here as a secondary resource stream rather than an initial feedstock category. That distinction matters because digestate properties are created by the original feedstock together with the conversion history. Its downstream route can only be selected rationally after those upstream decisions are known. The same digestate label can therefore describe materials with very different ammonium content, organic stability, salinity, contaminant profile, and dewaterability.

Product portfolios also need to be tested for compatibility rather than assembled as a list of theoretical outputs. A methane-maximising route removes biodegradable carbon that could otherwise support fermentation, whereas a VFA platform deliberately prevents complete methanogenesis. Phosphorus precipitation may improve a fertiliser product while consuming reagents and altering the remaining liquid. Pyrolysis can stabilise part of the separated solid but changes nitrogen availability and concentrates non-volatile minerals. Cascade design therefore requires explicit allocation of carbon, nutrients, water, energy, and contaminants at every branch.

Location and market demand can change the preferred sequence as much as reactor performance. A farm with nearby land may obtain more value from a hygienically safe digestate and avoided mineral-fertiliser purchase than from an energy-intensive nutrient concentrate. A centralised plant that must transport dilute material over long distances may benefit from separation and concentration despite higher electricity use. A technically recoverable co-product has little value if it lacks a specification, storage stability, or a realistic buyer. Engineering optimisation should therefore follow each product to its delivery point rather than stopping at the reactor outlet.

Integration can nevertheless create genuine synergies. Heat from combined heat and power may support hygienisation or moderate thermal treatment; recovered ammonia can become a concentrated nitrogen product; a separated digestate solid can serve as a precursor to carbon-rich material after appropriate processing; and dewatering can reduce the volume transported off-site. Such benefits should be credited only when the associated unit operation and end use are inside the system boundary and supported by a mass or energy balance [4–6,127–129].

7. Digestate Fractionation and Nutrient Recovery

7.1. Solid–Liquid Separation

Solid–liquid separation is the first practical branching point after anaerobic digestion because it determines how water, suspended solids, organic carbon, ammonium, and particulate phosphorus are distributed between subsequent routes. Digestate-valorisation and membrane-recovery studies show that front-end solids management strongly affects transport, fouling, nutrient concentration, and the suitability of later recovery steps [130–132]. A high solids-capture efficiency may reduce the mass transported with the liquid fraction while leaving most ammonium in solution. The separator should therefore be selected for the intended downstream product, not for an abstract goal of maximum solids removal.

The liquid fraction can be used locally under controlled fertigation conditions, concentrated, or treated for nutrient recovery. The solid fraction can be composted, dried, formulated, or converted to a carbon-rich material. Every option changes energy use, emissions, and contaminant distribution. Separation does not destroy a contaminant; it moves it. Nitrogen, phosphorus, carbon, salts, metals, water, and source-relevant micropollutants should therefore remain in the mass balance until the final destination of both fractions is known.

7.2. Membrane and Electrochemical Recovery

Pressure-driven membranes, membrane contactors, vacuum membrane distillation, electrodialysis, and combined membrane trains can recover water and concentrate ammonium or other ionic nutrients. Their advantages are modularity and product concentration; their recurrent limitations are fouling, cleaning demand, membrane replacement, concentrate management, and electricity use. Digestate solids content strongly affects flux and the amount of pretreatment required before a membrane step [133–136].

Ammonia recovery through membrane contactors or distillation can produce ammonium salts or concentrated nitrogen streams when product purity and contaminant limits are met. Evidence from chicken-manure digestate demonstrates the direct relevance of membrane-contactor recovery to agro-industrial streams [132,133,137]. If integrated upstream or as a sidestream, nitrogen removal may also relieve ammonia stress in the digester. That benefit is not free: pH adjustment, heat, vacuum, gas transfer, membrane wetting, and cleaning can become important burdens. Recovery percentage should therefore be reported together with nitrogen mass balance, product purity, and energy per unit of recovered nitrogen.

7.3. Phosphorus Recovery and Combined Routes

Struvite precipitation can recover phosphorus together with ammonium as a solid mineral product, but purity depends on magnesium supply, pH, competing ions, suspended solids, and trace contaminants. Phosphorus may also remain associated with the separated solid fraction or be concentrated after thermal and membrane treatment. Technology reviews make clear that no single nutrient-recovery route is optimal for every digestate composition [138,139].

Hydrothermal carbonisation and membrane treatment can be coupled with digestion to partition carbon and nutrients, but the resulting liquid by-products still require management. Systematic and full-scale evaluations show that nutrient recovery becomes economically credible only when transport savings, product value, avoided treatment, and local fertiliser demand are included alongside recovery efficiency [140,141]. Table 4 therefore distinguishes the recovered fraction, the transfer risk, and the evidence needed before the output is treated as an agricultural product.

Table 4. Digestate fractionation and nutrient-recovery routes, with the recovered fraction, principal burden, and qualification requirements.

Route	Principal Recovered Fraction	Potential Benefit	Main Burden/Transfer Risk	Qualification Before Use
Mechanical solid–liquid separation	Fibre/carbon-rich solid; ammonium-rich liquid	Reduced transport volume and route-specific handling	Contaminants are redistributed, not destroyed	Mass balance, dewaterability, nutrients, salts, and contaminants [127,130,140]
Pressure-driven membranes	Concentrated nutrient stream and treated water	Modular concentration and water recovery	Fouling, cleaning, electricity, concentrate management	Flux stability, nutrient recovery, rejection, and cleaning residuals [131,133–135]
Membrane contactor/vacuum distillation	Ammonia or ammonium salt	Concentrated nitrogen product; possible inhibition relief	pH, heat or vacuum, membrane wetting, product purity	N mass balance, purity, energy, and contaminant testing [132,133,137]
Electrodialysis	Ionic nutrient concentrate	Selective concentration with modular operation	Electricity, scaling, membrane fouling	Long-term flux, ion selectivity, cleaning, and concentrate outlet [136]
Struvite precipitation	Solid phosphorus-ammonium mineral	Transportable nutrient product	Mg/reagent demand, pH, co-precipitation, impurities	Recovery yield, purity, metals/organics, and agronomic availability [139]
Hydrothermal carbonisation/carbon route	Carbon-rich solid plus nutrient-bearing liquid	Carbon stabilisation where demonstrated; possible energy product	Heat, pressure, liquid by-product, mineral concentration	Carbon/nutrient allocation, leaching, liquid treatment, and end use [128,129,138,141]

Note: Recovery percentage should always be reported together with product composition and the fate of the unrecovered fraction.

7.4. Selecting a Recovery Train

A recovery train should begin with digestate composition and destination. High ammonium concentration combined with a realistic market for concentrated nitrogen can favour stripping or membrane-contacting routes. A liquid stream with recoverable dissolved phosphorus and controllable pH may justify precipitation. High suspended-solids content often requires robust mechanical separation before membranes. Conversely, very dilute streams can make impressive recovery percentages economically weak because a large volume of water has to be processed for a small mass of nutrient.

Recovery efficiency and product purity can move in opposite directions. Maximising phosphorus capture may co-precipitate organic matter or metals, while aggressive concentration can increase salinity and enrich persistent contaminants. Reporting recovery as a percentage is therefore incomplete without the composition of the product and the fate of the unrecovered fraction. Cleaning residuals and rejects also remain part of the material balance; concentration does not make them disappear.

Sequential recovery steps are justified only when each stage creates measurable value. Mechanical separation may reduce membrane fouling; ammonia removal may relieve inhibition or create a saleable nitrogen product; phosphorus recovery may reduce discharge risk; and final water treatment may enable reuse. These benefits remain conditional on

energy, reagents, maintenance, recovery yield, quality, and a verified outlet. A modular train is useful only if it can absorb feedstock variability without shifting the burden into an unmarketable concentrate.

8. Agricultural Products: Fertilisers, Humic-like Materials, and Microbial Preparations

8.1. Digestate Fertilisers and Soil Amendments

The fertiliser value of digestate depends on plant-available nutrients, organic-matter stability, pH, salinity, ammonium concentration, and application method. Total nutrient content alone says little about agronomic efficiency. Nitrogen availability may be high, yet surface application can increase ammonia loss; phosphorus response depends on chemical form and soil properties; repeated application can also accumulate salts or metals. Reviews and field studies generally report positive crop responses when application rate is matched to crop demand and soil conditions [142–145].

Comparisons with mineral fertiliser are most meaningful when treatments deliver equivalent available nutrient rates rather than equal fresh mass. Stabilised digestate and recovered ammonium sulphate can replace part of synthetic fertiliser demand, but transport, timing, odour, and ammonia emissions influence the environmental result. Horticultural and field studies likewise show that untreated and conditioned digestates can differ in both nutrient release and crop response [146–149]. More broadly, fertilisation-system experiments in white cabbage show that nutrient management itself can shift soil agrochemical indicators and productivity [150]. This is relevant to recovered-product trials because background fertilisation must be controlled before a yield response can be attributed to the tested material.

The distinction between fertiliser and plant biostimulant should remain explicit. If a digestate increases yield because it supplies nitrogen, its primary function is fertilisation. Under Regulation (EU) 2019/1009, a plant biostimulant acts by stimulating plant nutrition processes independently of its nutrient content, with claims related to nutrient-use efficiency, tolerance to abiotic stress, quality traits, or availability of confined nutrients [151]. Related digestate and biofertiliser studies reinforce why the intended claim determines the experimental control and evidence required [152–154]. For EU fertilising products, claim substantiation must also be considered together with the applicable product-function category, component-material category, contaminant, labelling, and conformity-assessment requirements.

8.2. Humic-like Substances and Non-Microbial Plant Biostimulants

Digestate and compost can contain recalcitrant organic fractions and humic-like substances, but the expression “humified fraction” should be used with care. Anaerobic digestion is not equivalent to soil humification, and the composition of extracted material changes with feedstock, digestion history, and post-treatment. Extraction yield, molecular-size distribution, functional groups, ash, salinity, and residual contaminants should therefore be characterised before biological activity is inferred.

Humic and humic-like fractions can influence root architecture, nutrient acquisition, iron chelation, and stress response. Evidence from leonardite, soil-derived humic substances, compost, and waste-derived formulations supports genuine biological activity, but the effective dose and response are highly source-dependent [155–158]. Humic materials can also enter product chains outside plant production. Sodium humate derived from freshwater sapropel has been evaluated as a feed additive in broiler chickens [159] and Holstein heifers [160]. These studies are relevant here only as evidence of a distinct cross-sector outlet for humic materials; animal-performance endpoints do not substantiate

plant-biostimulant efficacy. A non-microbial plant biostimulant does not require viable microorganisms; it requires a reproducible functional effect consistent with the claimed plant category and distinguishable from simple nutrient supply.

Waste-derived extracts require an additional safety step because salts, metals, or organic contaminants may be concentrated together with the target organic fraction. Compost-derived humic studies illustrate both the potential for biostimulant activity and the strong dependence on source material and molecular composition [161–163]. Product development should therefore combine chemical characterisation, dose–response testing, nutrient-matched controls, and greenhouse or field validation rather than inferring efficacy from chemical resemblance to humic substances alone.

8.3. Microbial Biofertilisers and Inoculants

Microbial biofertilisers, microbial plant biostimulants, and inoculants form a distinct product group. Their performance depends on identified viable microorganisms, a defined dose, storage stability, compatibility with the carrier, and biosafety. The mere presence of microbial biomass in digestate does not turn the material into a microbial preparation.

Plant-growth-promoting bacteria can influence nutrient transformations, phytohormone balance, root development, and rhizosphere community structure. The outcome, however, depends on strain identity, soil, crop, formulation, and environmental conditions [164–166]. A soybean field study combining seed inoculation with Rizoline-R and foliar KALNINI application illustrates an additional experimental issue: microbial and non-microbial inputs should be separated by appropriate controls if their individual contributions are to be interpreted [167]. Waste-derived carriers may be useful in such formulations, but they should not introduce unwanted organisms, excessive salts, or contaminants that undermine the intended biological function.

A credible microbial-product dossier should therefore include taxonomic identity, strain-level information where required, viable-cell count through the declared shelf life, formulation composition, evidence of the claimed function, and an agronomic trial with an appropriate nutrient control. Biosafety assessment should address relevant pathogenicity and antimicrobial-resistance concerns. The foundational literature on plant-growth-promoting bacteria [168] is used here only to define the microbial-function concept; more recent inoculant studies [169,170] support the practical distinction between a defined microbial function and the general nutrient effect of an organic amendment. Table 5 summarises the evidence requirements for fertiliser, soil-amendment, non-microbial biostimulant, microbial inoculant, and microbial-biostimulant claims.

Table 5. Agricultural product categories and the minimum evidence required to support each type of claim.

Product Category	Primary Function	Minimum Evidence	Not Sufficient by Itself	Critical Quality Controls
Digestate fertiliser	Supply plant-available nutrients	Available nutrient rate, crop response, application and emission assessment	Total NPK or equal fresh-mass comparison	Nutrient forms, salinity, hygiene, and contaminants [142–145]
Soil amendment	Improve soil physical or organic-matter properties	Stability, carbon character, soil response, and safe dose	Short-term yield increase driven mainly by mineral N	Stability, salts, metals, and persistent contaminants [143–149]
Non-microbial plant biostimulant/humic-like product	Stimulate plant nutrition processes independently of nutrient content	Nutrient-matched dose response; claim-relevant endpoint; greenhouse/field validation	Humic-like chemistry or any measurable plant response	Source consistency, dose, salinity, and contaminants [155–158,161–163]

Table 5. Cont.

Product Category	Primary Function	Minimum Evidence	Not Sufficient by Itself	Critical Quality Controls
Microbial biofertiliser/inoculant	Deliver viable functional microorganisms affecting nutrient supply or plant performance	Strain identity, viable count through shelf life, function, and agronomic trial	Microbial biomass naturally present in digestate	Identity, viability, carrier, biosafety, and AMR [164–166,168–170]
Microbial plant biostimulant	Microbial stimulation of plant nutrition processes under the claimed category	Identified microorganism(s), viable formulation, nutrient controls, claim-specific response	Fertiliser effect or rhizosphere detection alone	Identity, viability, efficacy, safety, and storage stability [151,164–166,168–170]

Note: The plant-biostimulant distinction follows Regulation (EU) 2019/1009. For EU fertilising products, the applicable PFC, CMC, contaminant, labelling, and conformity-assessment requirements must also be satisfied.

8.4. Evidence Hierarchy for Agricultural Claims

Agricultural product development is strongest when evidence accumulates in stages. Chemical and microbiological characterisation establishes what the material contains. Germination tests or short bioassays can identify acute phytotoxicity and a plausible dose range. Controlled pot experiments then help separate nutrient supply from other plant responses, while multi-environment field trials test whether the effect survives realistic soil, climate, and management variation. Tillage, irrigation, fertilisation, and soil physical condition should therefore be standardised or reported as field covariates. No single evidence tier replaces the others. A positive germination response does not establish yield benefit, and a field response without a nutrient-matched control cannot distinguish fertilisation from biostimulation.

Dose is especially important for concentrated or extracted products. Humic-like fractions may show nonlinear responses, while salinity, ammonium, or residual organic acids can become inhibitory at higher application rates. Microbial formulations can lose viability during storage or fail to establish in a competitive rhizosphere. Reporting only the best-performing treatment therefore overstates robustness. Trials should include an untreated control, a nutrient-equivalent control when relevant, more than one dose, and a reference product when the intended claim makes that comparison meaningful.

The evidence also has to be translated into manufacturing specifications. Feedstock acceptance limits, separation or extraction conditions, marker properties, viable count where applicable, contaminant limits, shelf life, and application rate should be defined. Batches that fall outside those limits require requalification. This prevents a general observation about digestate, compost, or humic substances from being converted directly into a commercial claim and keeps the product category aligned with the evidence summarised in Table 5 [146–148,155–157,164–166].

9. Product Safety, Contaminants, and Traceability

9.1. Pathogens, Metals, and Organic Micropollutants

Product safety cannot be inferred from nutrient content or from the fact that anaerobic digestion has been completed. Humic materials can alter metal mobility, as demonstrated by soil-washing studies using humic substances from manure or sewage-sludge compost [171,172]. Food-waste digestate has also been shown to contain organic micropollutants, heavy metals, and pathogens, with concentrations and survival affected by feedstock and treatment history [173]. Microplastics require separate attention because they can act as surfaces and vectors for other contaminants and microbial communities [174]. These findings argue for source-specific qualification rather than a blanket assumption that digestion produces a safe agricultural product.

Metals are not destroyed during digestion or downstream separation; they are redistributed between liquid, solid, precipitated, adsorbed, and carbonised fractions. Digestate-derived biochar can remove metals and antibiotics from liquid streams, co-pyrolysis can change metal immobilisation, and dissolved organic matter can alter metal behaviour in soil [175–177]. These processes may reduce mobility in one compartment while concentrating mass in another. Total concentration should therefore be complemented by fractionation or leaching tests where the end use makes mobility relevant.

Nutrient precipitation can also transfer pharmaceuticals or metals into a recovered solid. Sequential-extraction work on digestate sludge demonstrates how strongly metals can partition among operationally defined fractions, while closed-loop adsorption strategies using digestate-derived carbon materials show that the spent sorbent itself becomes part of the final material balance [178–180]. A safety assessment is complete only when each contaminant is followed to the final destination of every product and reject stream.

9.2. Microplastics and Antimicrobial Resistance

Microplastics can modify microbial attachment, extracellular polymeric substances, and the fate of antibiotic-resistance genes. Studies using polyethylene or PET particles in sludge and dairy-waste digestion have reported changes in resistance-gene abundance, mobile genetic elements, and microbial-community structure [181–184]. These effects cannot be transferred numerically to every agricultural digestate, but they establish a credible mechanism that deserves attention where plastic contamination is expected.

Operating conditions can modify the same risk. Organic loading, digestate recirculation, temperature, and thermal or hydrothermal treatment influence the occurrence of antibiotics, resistance genes, and mobile genetic elements in different waste matrices [185–188]. Monitoring should therefore be risk-based rather than identical for every product. A manure stream with substantial veterinary-antibiotic exposure and a food-waste stream dominated by packaging contamination do not require the same analytical emphasis.

Traceability is the practical control that connects those risks to process decisions. Each batch should be linked to feedstock origin, storage history, pretreatment, digestion conditions, separation, and chemical additions. With that record, a source-controlled digestate can be evaluated on its actual risk profile rather than being treated as equivalent to a heterogeneous mixed-organic stream. The same traceability also makes it possible to investigate an off-specification batch and determine whether the cause arose upstream, during conversion, or during product conditioning.

9.3. Risk-Based Product Qualification

Applying exactly the same analytical panel to every recovered product is inefficient and can still miss source-specific hazards. Qualification should combine a core set of parameters—identity, nutrient composition, pH, salinity, stability, hygiene, and declared use—with a feedstock-specific risk screen. Veterinary inputs justify targeted antibiotic and resistance monitoring; mixed food waste justifies packaging-derived plastic screening; mineral additives and industrial co-substrates justify focused analysis of metals or organic contaminants.

Process claims and product claims also need to be kept separate. Thermophilic operation or hygienisation may reduce selected pathogens, but validation requires documented time-temperature exposure and organism-specific evidence. Separation can lower a contaminant concentration in one fraction while concentrating it in the other. Pyrolysis may immobilise some metals yet concentrate their total mass in an ash-rich solid. The full material pathway therefore has to be documented before a fraction is described as safe for soil or crop contact [173–177].

A risk-management plan should state what happens when a batch fails. Depending on the failure, the options may include reprocessing, scientifically and legally justified blending, diversion to a less exposed use, or disposal. Repeated application deserves particular attention because salts, metals, persistent organics, microplastics, and resistance determinants may accumulate even when a single application appears acceptable. Test frequency should therefore reflect feedstock variability, exposure, legal requirements, and uncertainty in the evidence base.

10. Energy Efficiency, Life-Cycle Performance, and Economics

Energy reporting remains one of the least harmonised parts of the pretreatment literature. Values are expressed per cubic metre of slurry, per megagram of fresh feedstock, per megagram of TS or VS, per kilogram of TSS, or per reactor volume. Moisture content alone can change the apparent ranking by orders of magnitude. For continuous mechanical systems, throughput and material transport through the active zone also affect the denominator and the apparent specific energy. A valid comparison therefore requires a declared functional basis and a process boundary that includes, where relevant, pumping, recirculation, heating, mixing, chemical preparation, separation, and product conditioning.

The VLR study reported 2.16–3.6 kWh Mg^{−1} on a fresh-mass basis and approximately 9 kWh Mg^{−1} TS for the investigated substrates. The full-scale hydrodynamic-cavitation example used 470 kJ kg^{−1} TS, equivalent to approximately 131 kWh Mg^{−1} TS. Ultrasound studies can require far higher specific inputs, particularly in sludge treatment, but the resulting numbers should not be interpreted as a universal technology ranking. Feedstock, solids concentration, scale, objective, and downstream response differ. Table 3 uses the examples to demonstrate the denominator problem rather than to imply that one device is always superior.

LCA conclusions are equally sensitive to boundaries and avoided burdens. Relevant terms include avoided uncontrolled methane emissions, displacement of fossil energy, displacement of mineral fertiliser, transport of wet digestate, ammonia and nitrous oxide emissions after land application, infrastructure, and reject management. Farm-scale manure digestion and agricultural greenhouse CO₂ utilisation at anaerobic-digestion-based biomethane plants can reach different conclusions when electricity mix, useful heat recovery, and the credit assigned to digestate are changed [189–192].

Pretreatment should therefore be evaluated within the same boundary as the rest of the process. Hydrothermally treated straw or sludge may convert more rapidly while requiring additional heat and capital. Nutrient recovery may reduce transport and create fertiliser value, yet membrane replacement, cleaning chemicals, concentrate management, and product conditioning remain burdens. Full-scale recovery assessments show that local logistics and product value can matter as much as technical recovery efficiency [193–196].

A techno-economic analysis should state throughput, capital cost, energy and chemical prices, maintenance, product revenue, capacity factor, and uncertainty. Comparative assessments of high-solids digestion, agricultural residues, and manure-based co-digestion show that technical success does not guarantee economic viability at a given scale [197–200]. The appropriate decision metric is therefore a net process benefit under stated assumptions, not the largest percentage increase in methane.

Energy normalisation should begin with measured throughput and solids content. Converting kWh m^{−3} to a mass basis requires slurry density and TS; converting fresh-mass values to TS or VS requires the corresponding solids fractions. If those terms are unavailable, the reported value should remain on its original basis and be labelled as not directly comparable. Methane gains should likewise be converted with a declared methane

heating value and should distinguish gross gas energy from usable electricity, heat, or upgraded biomethane. This prevents unwarranted accuracy in cross-study rankings.

Uncertainty becomes especially important when the claimed net gain is small. Feedstock composition, control yield, energy price, heat recovery, equipment life, capacity factor, and product value can each reverse the preferred option. A practical assessment should show a base case, plausible ranges, and the variables to which the decision is most sensitive. For facilities dependent on seasonal residues or external co-substrates, scenario analysis is particularly important. A modest but robust positive balance may be preferable to a larger average gain that becomes negative under common operating conditions.

Environmental and economic boundaries should also be aligned. If the LCA credits avoided mineral-fertiliser production, the TEA should include the real cost of conditioning, certification, storage, and market access for the recovered product. If the TEA relies on a gate fee, the LCA should represent the displaced management route consistently. Agonomic credits depend on the declared management baseline, including tillage, irrigation, fertilisation, and soil condition. Credits for soil carbon, avoided methane, or recovered nutrients should therefore be conservative and time-explicit. The same benefit should not be counted twice in different parts of the assessment [189–192].

11. Minimum Sufficient Pretreatment: A Testable Decision Heuristic

11.1. Definition and Decision Sequence

Minimum sufficient pretreatment is defined here as the lowest non-zero treatment intensity—or zero pretreatment where appropriate—that achieves the predeclared downstream objective while maintaining process stability, product quality, safety, and a positive net benefit. The framework is a decision heuristic derived from the evidence reviewed above, not a universally validated model. Its purpose is to replace the vague instruction to “optimise pretreatment” with a sequence of measurements and decisions that can be tested experimentally.

The first step is feedstock characterisation. Moisture, TS, VS, ash, fibre or lignin where relevant, protein, lipid, C/N ratio, nutrient content, salinity, source-specific contaminants, and physical handling properties should be measured. The second step is diagnosis: structural recalcitrance, poor homogenisation, rapid acidification, ammonia, LCFA, salinity, contaminant risk, low organic loading, or insufficient product value may be the dominant constraint. Pretreatment is selected only if it addresses that constraint more directly than an alternative process intervention.

The third step is to define the intended product portfolio and functional unit. A methane-oriented plant should consider methane productivity and net usable energy; a nutrient-recovery system should include recovered nutrient mass, purity, and avoided transport; a humic-like product route should include extraction yield, chemical quality, dose–response, and plant-function evidence. The fourth step establishes the experimental comparison. For initial screening, a control plus at least three non-zero treatment levels is recommended so that curvature or an early plateau can be observed; additional levels may be required when the response is nonlinear or experimental uncertainty is large.

11.2. Quantitative Sufficiency Criteria

Pretreatment is insufficient when the predeclared response remains below its target or when the chosen mechanism does not address the actual limitation. It is sufficient when the target is met, mandatory stability and product constraints are satisfied, and the incremental benefit remains positive. It becomes excessive when greater intensity gives no practically meaningful improvement, reduces net benefit, destabilises conversion, or pushes

the product outside specification. The useful operating window is therefore bounded by both a lower performance requirement and an upper burden or risk limit.

For an energy-oriented system, a primary criterion is positive incremental net usable energy after pretreatment and auxiliary demand are included. Methane productivity and required HRT can be secondary criteria. A shorter HRT counts as a benefit only if VS destruction, methane concentration, and stability remain acceptable; in biomass-retaining reactors, SRT must be checked separately rather than inferred from HRT. For product-oriented systems, nutrient recovery, purity, salinity, hygiene, phytotoxicity, and verified plant response may be more important than gas yield.

For comparative screening, the economic part of the decision can be expressed as the monetised net-benefit relationship in Equation (1). Every term must use the same time horizon, throughput, currency year, and process boundary. Environmental indicators that have not been monetised should remain separate constraints or LCA endpoints rather than being added directly to monetary terms.

$$B_{net} = V_{\Delta E} + V_{\Delta P} + C_{avoided} - C_{pretreatment} - C_{auxiliary} - C_{conditioning} \quad (1)$$

where B_{net} is the incremental monetised net benefit relative to the untreated or zero-pretreatment reference; $V_{\Delta E}$ is the monetary value of additional usable recovered energy; $V_{\Delta P}$ is the incremental value of recovered or upgraded products; $C_{avoided}$ is a monetised avoided cost or credit included only when it is not already captured in $V_{\Delta P}$; $C_{pretreatment}$ is the direct pretreatment cost; $C_{auxiliary}$ covers pumping, heating, recirculation, mixing, and other auxiliary services; and $C_{conditioning}$ covers separation, stabilisation, cleaning, certification-related conditioning where relevant, and product preparation.

A candidate setting is acceptable only when $B_{net} > 0$ and all mandatory process-stability, safety, and product-quality requirements are met. Environmental performance should be reported in parallel through appropriate LCA indicators unless a transparent monetisation method is used. Double counting also has to be prevented: for example, the sale value of a recovered fertiliser and an avoided mineral-fertiliser cost should not both be credited if they represent the same benefit. Table 6 translates the heuristic into measurable indicators without treating any single percentage threshold as universally valid.

Table 6. Operational template for identifying insufficient, sufficient, and excessive pretreatment.

Decision Domain	Predeclared Indicator	Screening Criterion	Insufficient	Excessive/Reject
Primary conversion target	Methane productivity, HRT/SRT where relevant, VS destruction, or product yield	Predeclared minimum relevant effect with confidence/uncertainty criterion; no universal percentage threshold	Lower confidence bound or practical effect fails the target	Additional intensity gives no defensible gain beyond uncertainty
Incremental energy	Usable additional energy relative to pretreatment plus auxiliary demand	Positive incremental net usable energy on the same basis; an energy ratio >1 is meaningful only when numerator and denominator share the same boundary	Conversion target or energy criterion not met	Marginal energy burden equals or exceeds the marginal gain

Table 6. Cont.

Decision Domain	Predeclared Indicator	Screening Criterion	Insufficient	Excessive/Reject
Stability	VFA profile, alkalinity, methane concentration, ammonia, loading, SRT where relevant	Indicators remain inside the predeclared stable operating envelope	Target not reached despite stable operation	Target reached only with persistent instability or washout risk
Product performance	Recovery yield, purity, nutrient availability, verified function	Meets the predeclared specification and intended-use test	Below recovery or efficacy specification	Conversion gain is accompanied by an off-specification product
Safety	Pathogens, metals, organics, salinity, AMR/microplastics where relevant	Compliance with applicable legal limits and risk-based acceptance criteria	Failure cannot be compensated by energy or revenue	Any failed mandatory safety criterion requires rejection, reprocessing, or rerouting
Plateau/lowest acceptable intensity	Marginal change from intensity i to $i + 1$	Choose i when all criteria pass and the next-step benefit is not practically distinguishable from uncertainty or added burden	i does not satisfy all mandatory criteria	$i + 1$ adds burden without a defensible incremental benefit

Note: Screening criteria are project-specific and should be predeclared with a justified minimum relevant effect and uncertainty range. $B_{net} > 0$ is required for the monetised decision metric, while legal, safety, and product-quality constraints remain mandatory and cannot be offset by an energy or revenue gain.

11.3. Application to Contrasting Feedstocks

For a fibrous crop residue, slow hydrolysis may be the initial limitation. Candidate treatment levels can then be compared by particle-size distribution, initial methane-production rate, volumetric productivity, VS destruction, energy input, and net balance. The sufficient condition is the first level that reaches the pre-declared productivity or retention-time objective with positive net benefit. Additional milling that produces a change smaller than experimental uncertainty or practical significance is excessive.

For dilute swine manure, low organic loading may be more important than particle accessibility. Concentration or co-digestion should therefore be compared with mechanical treatment rather than assuming that greater disintegration is desirable. Spent mushroom substrate presents a different case: fungal cultivation has already modified the fibre, so moderate mechanochemical activation may be sufficient. The VLR results are consistent with this interpretation, but they do not establish a universal intensity threshold; additional levels and continuous validation are still required.

For slaughterhouse waste, LCFA and ammonia can dominate performance. A disintegration step that releases more soluble lipid without improving stable conversion is mechanistically unsuccessful even if particle size falls substantially. Controlled feeding, co-digestion, adsorption support, biomass retention, or phase separation may therefore deserve priority. Figure 3 presents this branching logic and treats a change in mechanism—including a return to zero pretreatment—as a valid decision when the first candidate treatment does not address the dominant constraint.

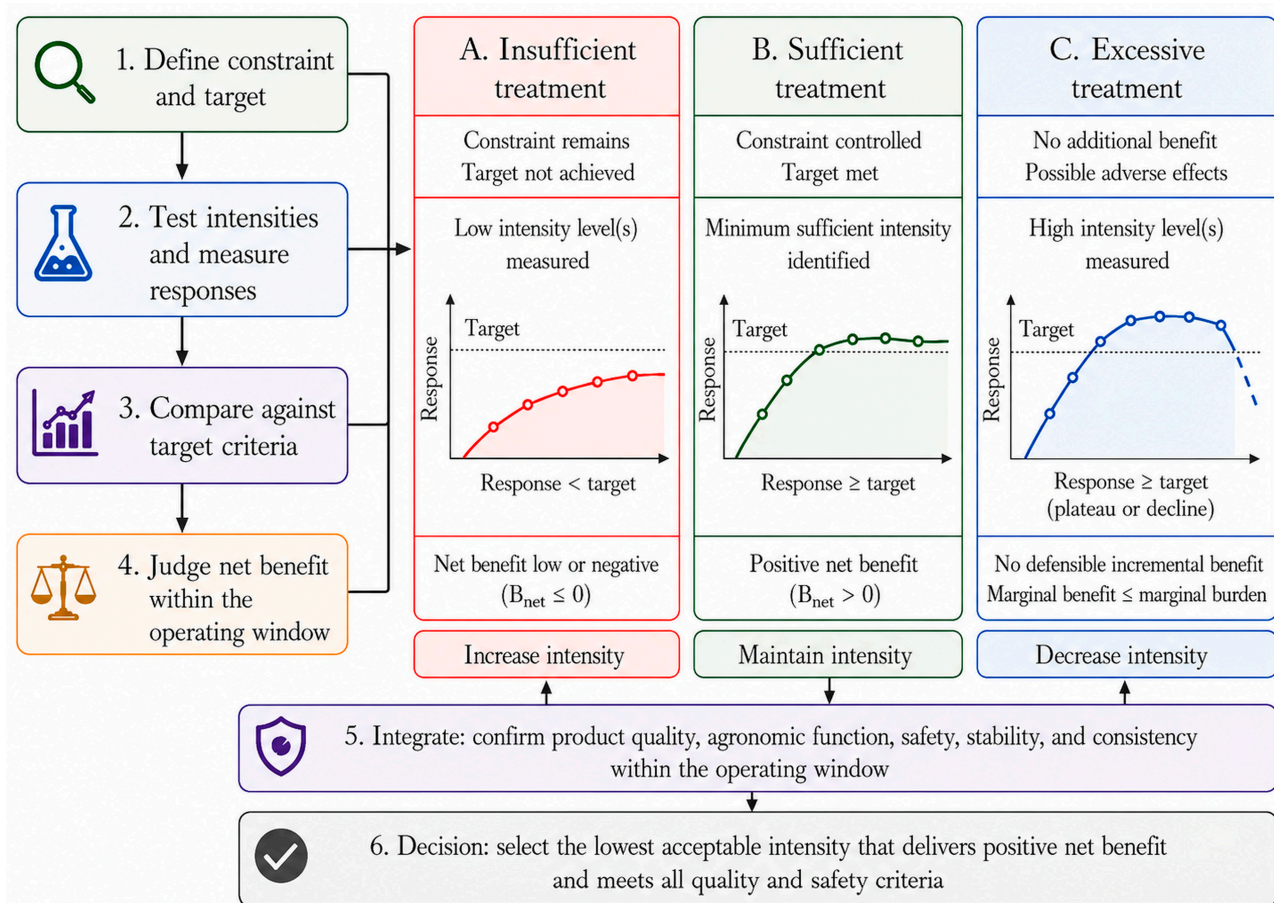


Figure 3. Decision logic for identifying insufficient, sufficient, and excessive pretreatment. The lowest acceptable intensity, including zero pretreatment where appropriate, is selected only when the primary response, net benefit, stability, product quality, and safety requirements are satisfied.

11.4. Experimental Validation of the Heuristic

Validation should use a dose–response design rather than a simple untreated-versus-treated comparison. For initial screening, a control and at least three non-zero intensities are recommended, with additional levels where needed to resolve curvature or a plateau. Biological replication is required, and the control should be processed identically except for the active treatment. Energy and auxiliary demand should be measured at the same scale as the treatment. The primary endpoint, acceptable stability range, minimum relevant effect, and product-quality limits should be specified before the results are interpreted.

The decision rule should be marginal as well as absolute. A selected intensity must satisfy every mandatory constraint and provide positive total net benefit, but the next higher intensity should also be examined. If its incremental gain is smaller than the incremental burden, or cannot be distinguished from measurement and biological uncertainty, the lower intensity is sufficient. This plateau rule discourages selection of a nominal maximum that is not meaningfully better than a less intensive setting.

Validation must then move from batch tests to continuous operation. The selected treatment window should be challenged with realistic feedstock variability and organic loading for long enough to observe microbial adaptation, operational disturbances, and product consistency. Failure of a legal, safety, or product-quality requirement overrides an energetic gain. Conversely, a modest methane response can still be worthwhile when a verified reduction in mixing, retention time, transport, or nutrient-management burden improves the complete configuration. Table 6 should therefore be read as a reporting and decision template; site-specific targets require predefinition and experimental calibration.

12. Scale-Up, Monitoring, and Industrial Readiness

Scale-up changes the meaning of pretreatment performance. Laboratory studies usually work with homogenised samples, controlled temperature, and short operating periods. Industrial facilities receive variable batches and must handle sand, fibres, plastics, grit, foaming, abrasion, pump blockage, cleaning, and unplanned interruption. A positive BMP response is therefore evidence of potential, not proof of industrial readiness.

Continuous and pilot-scale studies show why dynamic interpretation matters. Speciated VFA monitoring can reveal instability that total VFA or pH alone may miss, while co-digestion and pilot pretreatment studies demonstrate the importance of repeated feeding, adaptation, and long-term operating conditions [201–203]. Reference [202] concerns waste-activated sludge and source-sorted biowaste and is used only as an operational comparator for pilot- and full-scale reactor behaviour; its numerical results are not transferred to agricultural feedstocks. Full-scale operation also exposes energy interactions absent from batch bottles, including viscosity effects, mixing demand, recirculation, and maintenance.

Evidence from continuously fed reactors, pilot-scale co-digestion, and full-scale digestate-management assessments further shows that microbial adaptation, stable feeding, and downstream logistics must be evaluated together [204–206]. A condition that performs well during one BMP cycle may fail when soluble organics are released every day or when the resulting digestate becomes expensive to manage. Pilot validation should therefore include feedstock variability, start-up, steady-state operation, upset recovery, product handling, and mass balance.

A practical scale-up programme can be organised around four evidence gates. The first confirms the mechanism and treatment window under controlled conditions. The second verifies continuous biological stability and measured energy demand. The third qualifies recovered products and contaminant pathways. The fourth demonstrates reliable throughput, maintainability, availability, and economics at pilot or demonstration scale. Equipment geometry, active-zone configuration, throughput, and recirculation should be documented within these gates so that pretreatment performance can be reproduced. The gates are proposed as an engineering sequence derived from the reviewed evidence, not as a regulatory TRL classification.

Full-scale monitoring and dry-digestion studies show that product logistics can determine feasibility even after the biological reactor is technically successful [207–209]. Storage, dewatering, transport distance, land availability, nutrient demand, and the timing of application should therefore be considered from the beginning of facility design. Table 7 summarises the minimum reporting dataset needed to make future pretreatment-conversion-product studies comparable.

Monitoring is useful when it is connected to action limits. Feedstock TS, viscosity, or pump load can trigger a change in treatment intensity; VFA species, alkalinity, ammonia, and methane productivity can trigger loading adjustments; separation performance and product conductivity can trigger diversion or reformulation. A sensor becomes operationally meaningful only when its signal is tied to a defined response and is periodically checked against laboratory analysis. This avoids both delayed intervention and unnecessary control of normal biological variation.

Industrial readiness also requires maintainability. Wear parts, ferromagnetic elements, cavitation devices, heat exchangers, membranes, and pumps should be assessed for access, cleaning, replacement interval, and effect on uptime. Parasitic electricity during start-up, idling, and recirculation belongs in the measured energy balance. Demonstration studies should report availability, unscheduled stops, operator interventions, replacement frequency, and off-specification product batches because annual performance is often controlled more strongly by these variables than by a short period of peak methane yield.

Table 7. Minimum reporting dataset for comparable pretreatment-conversion-product studies.

Domain	Report at Minimum	Preferred Basis/Unit	Why It Matters	Evidence Gate
Feedstock	Origin, sampling period, moisture, TS, VS, ash, C/N; fibre, protein, lipid, salts, and contaminants as relevant	Mean, range, and number of batches	Defines mechanism and variability	Characterisation
Pretreatment	Equipment, geometry, dose/intensity, time, temperature, recirculation, and throughput	Per batch plus continuous rate where applicable	Makes treatment reproducible	Mechanism
Energy/material inputs	Core and auxiliary electricity, heat, chemicals, water, and wear/replacement	kWh Mg ^{−1} fresh, TS, and VS where calculable	Prevents denominator and boundary errors	Net benefit
Biological operation	Inoculum, loading, HRT, SRT where relevant, temperature, mixing, adaptation, and duration	Standard reactor units	Separates treatment from operating effects	Continuous validation
Conversion response	Gas volume, CH ₄ , productivity, VS destruction, kinetics, and soluble COD	Absolute value plus change relative to control	Distinguishes rate, yield, and solubilisation	Performance
Stability	VFA species, alkalinity, pH, ammonia, sulphide, foaming, and process upsets	Time series plus operating envelope	Detects transferred biological burden	Stability
Mass balance	C, N, P, water, and relevant contaminants across every output	Recovery %, closure %, and mass/time	Prevents hidden loss or redistribution	Integration
Recovered product	Composition, purity, stability, dose, shelf life, intended claim, and outlet	Product-specific specification	Connects recovery to real use	Qualification
Safety/agronomy	Risk-based contaminants, hygiene, phytotoxicity, nutrient-matched controls, and greenhouse/field response	Legal limits plus effect size	Verifies safe functionality	Product gate
Scale/economics	Scale, availability, maintenance, CAPEX/OPEX, prices, uncertainty, and sensitivity	Annual throughput and common currency year	Tests industrial feasibility	Demonstration

Note: NR values and non-convertible reporting bases should remain explicit rather than being harmonised through unsupported assumptions.

A minimum dataset cannot remove differences among plants, but it can make those differences interpretable. Feedstock statistics, functional unit, treatment dose, scale, energy boundary, loading and retention time, gas and mass balance, stability indicators, product allocation, safety tests, and uncertainty should be reported together. Table 7 is therefore not an exercise in administrative completeness. Its purpose is to prevent an apparent treatment advantage from being created by an omitted denominator, an unreported auxiliary load, or an unqualified downstream product.

13. Research Gaps and Priorities

The first research priority is harmonised reporting. Whenever the underlying measurements allow it, pretreatment energy should be given on fresh-mass, TS, and VS bases together with throughput and auxiliary energy. Methane response should include absolute yield, production rate, and the operating variable the treatment is intended to improve. A

percentage increase alone is difficult to interpret when control yield and solids concentration differ among studies.

The second priority is the downstream product. Gas composition is often characterised in detail while digestate is reduced to TS, VS, or total nutrient content. Future studies should report nutrient forms, salinity, stability, dewaterability, source-relevant contaminants, and the intended product specification. Where a biostimulant claim is proposed, nutrient-matched dose–response experiments and greenhouse or field validation are needed to separate biostimulation from fertilisation.

A third priority is continuous feedstock-adaptive control. Sensors for solids, viscosity, conductivity, pH, VFA, ammonia, and gas composition could support adjustment of treatment intensity, recirculation, and loading. Data-driven control is useful only when it remains anchored to mass balance and biological mechanism. A predictive model trained on one stable feedstock should not be transferred to a different waste stream without external validation and an explicit check that the same limiting mechanism still applies.

The final priority is integrated TEA, LCA, and agronomic evaluation. A technically effective pretreatment can be economically unattractive; an energy-positive process can shift emissions to land application; and a nutrient-rich recovered fraction can still be agronomically unsuitable. Studies that follow carbon, nutrients, energy, contaminants, cost, and crop response through the complete chain will provide the strongest evidence for selecting a genuinely minimum sufficient treatment.

14. Conclusions

Agro-industrial organic wastes require feedstock-specific rather than technology-centred process design. Manure, lignocellulosic residues, food-processing waste, spent mushroom substrate, and lipid-rich animal-processing residues differ not only in the mechanism limiting conversion but also in the quality and safety constraints that govern their recovered products. No pretreatment can therefore be ranked as universally superior.

Mechanical and mechanochemical methods are rational when accessibility, particle structure, or homogenisation limits performance. Cavitation, thermal, chemical, and biological methods can provide stronger disintegration or greater selectivity, but their energy, reagent, inhibitor, maintenance, and product-quality burdens have to be included in the same assessment. The VLR evidence illustrates the central point particularly well: the same low-energy operating principle increased biogas production from swine manure and spent mushroom substrate but did not improve slaughterhouse waste, where inhibition remained the dominant constraint.

The proposed minimum-sufficient-pretreatment heuristic defines sufficiency through a predeclared response, positive net benefit, process stability, safety, and product quality. Zero pretreatment remains a legitimate decision when another intervention addresses the constraint more effectively. The framework now needs validation through multi-intensity experiments followed by continuous operation under realistic feedstock variability. Until such validation is available, it should be treated as a structured engineering heuristic rather than a universal optimisation method.

Cascaded recovery of methane, nutrients, fertilisers, humic-like fractions, microbial or non-microbial agricultural products, and carbon-rich or demonstrably stabilised carbon fractions is feasible only when the outputs are compatible with the selected feedstock and process. The preferred route is therefore determined by feedstock properties, target products, biological response, energy and material balances, contaminant risk, market outlet, and industrial readiness. The practical implication is simple: pretreatment should be justified by the limitation it removes and by the value it creates across the complete process, not by the intensity of the physical effect alone.

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