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Review

Recent advances on physical technologies for the pretreatment of food waste and lignocellulosic residues

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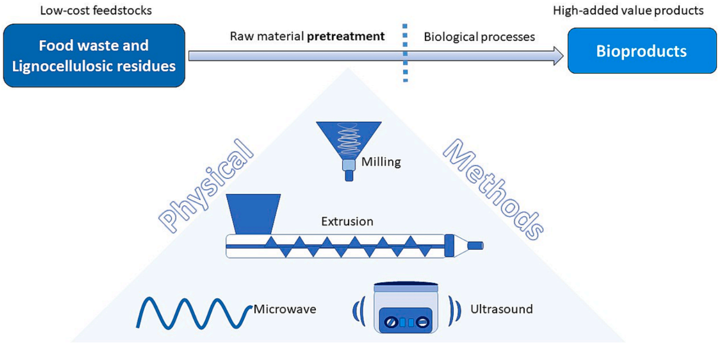
HIGHLIGHTS

- Food and lignocellulose residues are sustainable feedstocks for use in biorefineries.
- Physical pretreatments can provide key advantages in comparison to other methods.
- Advances in milling, extrusion, ultrasonication and microwave pretreatment are given.
- Further research is needed to make feasible the scaling up of these technologies.

GRAPHICAL ABSTRACT



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ABSTRACT

The complete deployment of a bio-based economy is essential to meet the United Nations' Sustainable Development Goals from the 2030 Agenda. In this context, food waste and lignocellulosic residues are considered low-cost feedstocks for obtaining industrially attractive products through biological processes. The effective conversion of these raw materials is, however, still challenging, since they are recalcitrant to bioprocessing and must be first treated to alter their physicochemical properties and ease the accessibility to their structural components. Among the full pallet of pretreatments, physical methods are recognised to have a high potential to transform food waste and lignocellulosic residues. This review provides a critical discussion about the recent advances on milling, extrusion, ultrasound, and microwave pretreatments. Their mechanisms and modes of application are analysed and the main drawbacks and limitations for their use at an industrial scale are discussed.

1. Introduction

Today, the world transitioning from a fossil carbon-based economy to a bio-based economy that relies on renewable natural resources. However, on a planet already pushed to the limit, it is essential to make better use of the available resources, following the principles of the

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circular economy. In this context, biorefineries will help comply with the UN's Sustainable Development Goals, as they align the objectives from both the bioeconomy and the circular economy by enabling the use of biomass at a large scale in a sustainable way. According to the definition of the organisation IEA Bioenergy, the concept of biorefinery includes integrated industries that, based on many types of biomass feedstocks (e.g. lignocellulosic or municipal solid waste) and a variety of different technologies, produce energy and a range of marketable products such as biofuels, chemicals, materials, food, and feed (Lindorfer et al., 2019).

Among the variety of biomasses, residues derived from food loss and waste along with lignocellulosic-based materials, are considered appropriate feedstocks for exploitation in biorefineries. Some of the characteristics that support the valorisation of these types of biomasses as raw materials are the generation of these wastes in huge quantities globally, their biochemical natures (source of C-rich material), and the environmental and economic impacts due to inappropriate storage and disposal (water and soil contamination, growth of unwanted organisms and pests). Furthermore, energy extraction from this kind of wastes is usually inefficient when discarded, so there is room for further upgrading the use of these materials from a biorefinery perspective (Moreno et al., 2020).

The Food and Agriculture Organization (FAO) has reported that the majority of food losses at global level in 2016 were oil-bearing crops, tubers and roots, followed by vegetables and fruits (Fig. 1). Approximately 30 % of the land employed for agriculture is used to produce food that is then lost or discarded (FAO, 2019). In economic terms, these food losses cost approximately US$ 680 billion in developed countries and about half of that in developing countries (Mehariya et al., 2018), which has an impact on the global economy of more than US$ 900 billion (Mak et al., 2020). Using these wastes as resources would benefit the agro-food industry not only in environmental terms by preventing the exploitation of new sources but also in economic terms by lowering the cost of both existing and new processes in which these raw materials are involved.

Regarding the lignocellulosic residues from agriculture, forestry or the urban context, they represent an ideal source of sugars derived from cellulose and hemicellulose, in addition to other compounds such as lignin (Dharma Patria et al., 2022). An estimation of the amount generated of the main crop and wood residues in 2020 is presented in Table 1. The relevance of using lignocellulosic feedstocks in the biorefinery industry is highlighted by the fact that it makes part of numerous EU-funded projects, which pretend the installation and the consolidation of lignocellulosic-based biorefineries in Europe (Hassan et al., 2019).

The feasibility of using lignocellulosic biomass (LB) to obtain bio-based products highly depends, however, on the modifications of the structural characteristics of these feedstocks after the pretreatment step. Pretreatment is required to alter biomass structure to ease the accessibility of the corresponding carbohydrates and/or to fractionate biomass into its main components (Mankar et al., 2021). Therefore, this process stage is considered a key element in the profitability of a biorefinery scheme and directly influences operational and capital process costs.

The most general classification divides many existing pretreatments into physical, chemical, physicochemical and biological methods. Physical pretreatments, i.e. milling, extrusion, ultrasounds and microwave (MW) radiation, use mechanical or wave energy to alter the biomass structure. These methods are sometimes relegated to particle size reduction purposes and criticised for their high-energy consumption (Mankar et al., 2021). However, the search for greener processes that

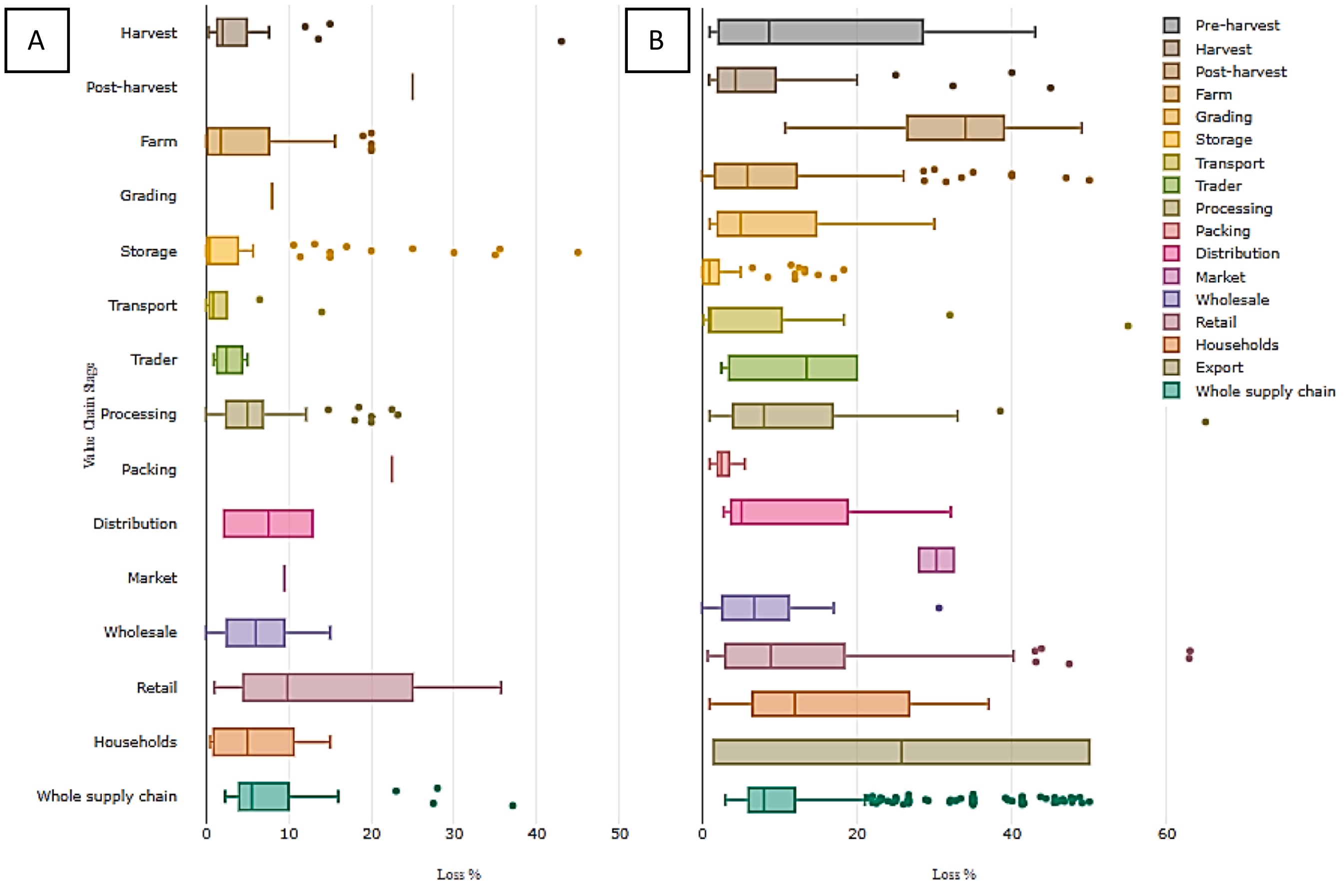

Fig. 1. Percentage losses in the food value chain in worldwide: A) Roots, tubers and oil-bearing crops. B) Fruit and vegetable (screening from 2012 to 2020) – FAO https://www.fao.org/platform-food-loss-waste/flw-data/en/.

Table 1
Theoretical residue production (does not discriminate by use) of the top ten crops cultivated in the world and wood residues production. Data from 2020.

Type of residue	Production[a] (Mtonnes)	Residue ratio[b]	Theoretical residue production (Mtonnes)
Sugarcane	1870	0.16	299
Maize	1162	1.27	1476
Wheat	761	1.18	898
Rice	757	1.58	1196
Oil palm fruit	418	0.26	109
Potatoes	359	0.11	39
Soybeans	353	3.50	1237
Cassava	303	0.52	157
Sugarbeet	253	0.27	68
Tomatoes	187	1.19	222
Total crop residues			**5702**
Wood			153[c]
Recovered post-consumer wood			29
Total wood residues			**182**

[a] Data obtained from FAOSTAT.
[b] Dry mass of residue/fresh mass of crop, obtained from Searle and Malins (2015).
[c] Wood residue production in FAOSTAT is given in m^3, to calculate the theoretical residue production a mean wood density of 655 kg m^{-3} has been used (Martínez et al., 2020).

use fewer chemicals, produce less waste and favour the simplicity of operation (Ab Rasid et al., 2021; Bychkov et al., 2014) has brought these kinds of pretreatments back into the spotlight.

The present review aims to offer a comprehensive view of the latest progress obtained for the most representative physical pretreatments, highlighting their strengths and weaknesses, with a critical approach to evaluating of their future role in biorefineries from food and lignocellulosic wastes.

2. Mechanical comminution

Among the many technologies already tested and proven to be effective as biomass pretreatments (Mankar et al., 2021; Periyasamy et al., 2022), mechanical milling significantly improves the digestibility of pretreated materials and therefore represents a valuable approach to alter the physical structure of biomass.

Milling is included in the mechanical actions, including impact, shearing, compressing, crushing, friction, and stretching. It is frequent to find the terms milling and grinding used equally in the literature. During milling, the material is subjected to intense mechanical stress, causing a series of effects on the physicochemical features of the raw biomass, which finally result in an increase in its reactivity.

As a widely accepted method to efficiently modify the structural features of solid biomass, milling is frequently combined with other pretreatment methods as a first step in processing raw lignocellulosic materials, both at a laboratory and industrial scale. A major and significant effect is exerted on cell membranes, particularly on the cellulose structure, by disrupting the cellulose fibres and decreasing the crystallinity and degree of polymerisation (Mankar et al., 2021). In addition, Zhou and Tian (2022) have reported an increase in the surface area accessible for the action of the enzymes. Supporting this statement, several research works have proven that smaller sizes of biomass particles are less resistant to enzymatic hydrolysis (EH) than larger ones. For example, Gu et al. (2018b) found an about 5-fold increase in the sugar production rate of Douglas-fir forest residuals milled for 30 min in a ball mill, compared to untreated biomass.

2.1. Milling technologies

A substantial number of milling processes have been developed and tested until now, as well as specific machines for each type of technology. Various reviews have been published in the last years describing the modes of action, devices and main effects in raw materials of milling methods as biomass processing techniques (Barakat et al., 2014; Lomovsky et al., 2016; Mayer-Laigle et al., 2018; Lomovskiy et al., 2020; Shen et al., 2020; Sitotaw et al., 2021). For example, Lomovsky et al. (2016) focus on the type of impact in the particles during milling, distinguishing between constrained or abrasive impact mills (a high probability of interaction and chemical reactions between particles), and free impact mills (interaction of a single particle with the milling body, much lower probability of contact among particles). The first group would include disk mills, bead mills, ball mills, planetary or tumbling mills, vibrational mills, vibrocentrifugal mills, and roller mills. The second would comprise pin mills or disintegrators, hammer and knife mills and jet mills. A scheme of the mode of action of a few milling technologies is presented in Fig. 2. Also, a brief overview of the main actions exerted in the most common types of milling that have been tested in LB pretreatment is provided in Table 2, with relevant operational parameters and some examples of specific applications in LB feedstock. In general, running time is a key parameter for any milling technique, so it is not included in Table 2 and is considered to affect all of them.

A search of up-to-date literature references about the application of almost all milling techniques in LB conversion allows concluding that the most common milling modes are hammer milling, (wet) disk milling, and, above all, ball milling (in the different existing types).

2.2. Hammer and disk milling

Hammer milling causes the impact (collision) of the biomass particles with grinding bodies (hammers) of large size that are placed in a gyratory rotor, plus the crushing and abrasion action of the particles pressed through a screen that is mounted under the rotor. Recently, Maitra and Singh (2021) have tested a novel approach to pretreat

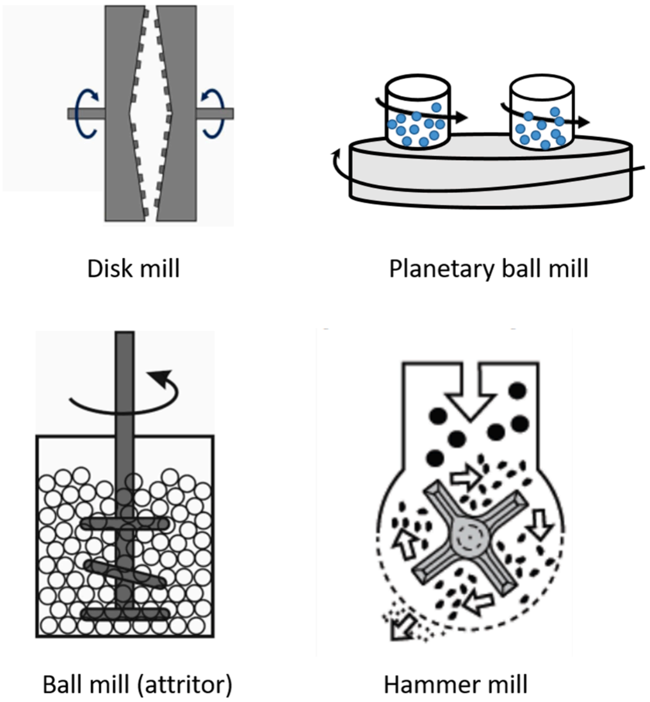

Fig. 2. Scheme of the mode of action of different types of milling. Adapted from Lomovsky et al., (2016) and Sitotaw et al. (2021).

Table 2
Main milling technologies and selected examples of relevant applications from literature.

Type of milling	Main mode of action	Relevant variables to consider	Biomass feedstock	Application	Main outcomes	Reference
Disk	Friction Beating Shearing Pressing	• Distance between disks • Disk design	Corn stover	• Fermentable sugar production (thermochemical + disk refining)	• Differences in sugar yield in relation to lignin extracted and final lignin content • Significant effect of disk refiner's gap size on net specific energy consumption	Chen et al. (2020)
(Wet) disk	Shearing Crushing	• Solid/liquid ratio	Sugarcane bagasse	• Ethanol production (+LHW)	• Substantial increase of ethanol production by combining LHW under low severity conditions and wet-disk milling	Wang et al. (2018)
Hammer	Crushing Abrasion	• Number of grinding bodies • Mesh size of screen	Wheat straw	• Biogas production	• Significant increase of biochemical methane production and EH yield in pretreated biomass, compared to untreated biomass (13.5 and 20.6 % respectively)	Victorin et al. (2020)
Knife	Cutting Crushing Abrasion	• Number and position of knives • Mesh size of screen	Wheat straw	• Ethanol production	• The use of large particle size (mm) may be advantageous over smaller sizes (≤0.5 mm) when total sugar yield (pretreatment + EH) is compared	Yang et al. (2022)
Ball-thumbler	Impact Compression Shearing Collision	• Ball/biomass ratio • Material of ball • Rotation frequency • Bead type and diameter	Corn stover	• Biomass deconstruction	• Demonstration of modification of the supramolecular structure of cellulose hemicelluloses-lignin matrix and depolymerization of polymeric structure of cell wall	Liu et al. (2019)
Stirred ball-attritor	Grinding Dispersion Shearing	• Impeller rotation speed • Bead filling ratio	Cedar wood	• Glucose and ethanol production through simultaneous enzymatic saccharification and comminution (SESC)	• Saccharification efficiencies of 80 % (laboratory scale) and 60 % (large scale). High fermentation yield of SESC-slurry	Navarro et al. (2018)
Planetary ball	Impact Friction Shearing	• Rotation speed	Pine wood waste	• Biobutanol production	• Good pretreatment performance in terms of high saccharification and improved biobutanol yields and low inhibitory compounds production	Kwon et al. (2016)
Vibrational ball	Crushing Impact	• Vibrational frequency • Amplitude of vibration	Corn stover	• Sugar production by EH	• Structural changes at cellular level: size reduction, increased surface area, decrease in crystallinity index. Significant improvement in cellulose conversion	Chen et al. 2022
Roller	Abrasion Crushing	• Space between rollers • Number of rollers	Corn straw	• Sugar production by EH	• Decrease of crystallinity index, increase of specific surface area (SSA) and specific pore volume. Two fold increase in carbohydrate hydrolysis	Bychkov et al. (2014)
Rod	Impact Friction	• Operation speed • Rod diameter	Wheat straw	• Biomass pyrolysis	• Reduction of particle size and cellulose crystallinity, and increased SSA and pore volume. Improvement of pyrolysis efficiency	Bai et al. (2018)
Jet	Collision Impact	• Acceleration speed • Air flow	Sugarcane bagasse	• Sugar production by EH	• Improvement of cellulose hydrolysis yield, but negligible effect on crystallinity index. Comparison with other milling techniques	Licari et al. (2016)

sugarcane bagasse by hydrothermal pretreatment with cryogenic grinding to minimise inhibitor production and increase sugar recovery. The cryogenic grinding was carried out after the hydrothermal process (140–200 °C for 10 min) in a hammer mill microfine grinder device prepared with a 0.5 mm sieve and liquid nitrogen, being continuously fed into the mill in a ratio of 10:1 (v/w) of liquid nitrogen: biomass in each milling cycle (five). The results support a good perspective in the utilisation of this novel strategy with hydrothermal pretreatment at low temperatures (150–170 °C), thus contributing to a low-severity approach.

Disk mills consist of two disks with grooves and bars that rotate (counter-rotating or one rotating and other stationary). At the same time, the material passes between them, causing shearing, friction, beating and pressing (Table 2). This milling process has been successfully applied to pretreat several biomass feedstocks aimed at improving sugar release by EH. For example, Hu et al. (2017) investigated and modelled at the laboratory scale the response during EH tests of maize straw pretreated with a disk micro-grinder until reaching different particle sizes (from 53 to 180 μm) for sugar production. Interestingly, the authors found that the effect of milling mainly influences the available surface area of pretreated material and increases the affinity of the enzyme towards this surface.

On the other hand, in the wet-disk mill, particles are scattered in a liquid to form a slurry, which is pumped through a grinding chamber. The particles ride along in the liquid and are crushed among the grinding media, using the recirculation process to achieve the targeted particle size. Wet milling is claimed as a low-energy consuming process, and several studies report the application of this pretreatment alone or in combination with other pretreatments to obtain sugars (Zakaria et al., 2015; Huang et al., 2019) or cellulose nanofibers from oil palm fibers (Ariffin et al., 2021).

2.3. Ball milling

Focusing on ball milling technology, an overview of the state of the art of the main ball milling modes (tumbling, attrition, vibratory and planetary) is provided in a recent review by Sitotaw et al. (2021). The authors describe the fundamentals of each type of milling and discuss in depth the main factors influencing the results of such a technique: the characteristics of milling balls, the milling time and temperature, the filling volume, the rotational speed of the milling pot, the grinding ball to biomass ratio and if it is performed using dry or fresh biomass. Moreover, the effects of ball milling in the LB features are discussed, focusing on the crystallinity index, the morphology, and specific surface area of pretreated materials. In the same line of contribution, the thermo-physical and optical properties of lignocellulosic biomass of corncob subjected to ball milling have been analysed in depth by Zhang et al. (2019) using updated methodologies such as scanning electron microscope, X-rays diffractometer, thermogravimetric analysis, and Fourier transform infrared spectroscopy. It is proven that changes in ball milling parameters significantly affect the crystallinity of cellulose, the content of both cellulose and hemicellulose and the thermal decomposition pattern of the biomass, which can be used to select the best operation conditions for a specific target product. In this case, the authors focus on producing bio-hydrogen, representing a novel application of mechanical pretreatment as ball milling. Liu et al. (2019) provide evidence of the effect of ball milling in lignocellulosic fibers by increasing material digestibility in studies carried out in corn stover. The results of this study reveal that ball milling causes depolymerisation of the polysaccharide chains, disruption of the orderly fibrillar matrices and the degradation of lignin-carbohydrate complex bonds.

Another recent and novel advance using ball milling considers its application intermittently during EH of aspen branches leftovers at low enzyme doses (Wu et al., 2021). The authors claim a positive effect of milling in the distribution of enzymes into the cellulose, improving the efficiency of the enzymatic attack during the static period and resulting in a shortening of the incubation time to reach a similar final sugar concentration than in a conventional EH incubation. The combination of milling and EH has also been demonstrated to be effective in a simultaneous enzymatic saccharification and comminution (SESC) process in cedar softwood at a large scale (ball milling equipment with a processing capacity of 10 L of aqueous slurry) for ethanol (Navarro et al., 2018) and methane (Navarro et al., 2020) production. In this line of applications of ball milling in biological biomass processing, Balch et al. (2020) report on an innovative application of this technology in the fermentation of various biomasses (corn stover, poplar and switchgrass) by *Clostridium thermocellum* and four other fermenting microorganisms in a custom-made ball milling bioreactor (named cotreatment). This interesting research proves that ball milling can boost carbohydrate solubilisation for all three feedstocks without previous pretreatment or added enzymes, with significant differences in the tolerance of the studied microorganisms to milling under the conditions tested.

The review by Sitotaw et al. (2021) also examines the ball milling-assisted chemical pretreatment, which combines ball milling and a chemical agent, such as acids, alkalis, organosolvents and metal salts, providing a summary of updated studies performed in feedstocks such as corn stover, barley straw or sugarcane bagasse. Shen et al. (2020) explore the same concept. They introduced the term “mechanochemical-assisted pretreatment” in reference to the utilisation of auxiliary reagents during ball milling, aimed at improving the effectiveness of mechanical action and decreasing energy consumption. According to Zhou and Tian (2022), this combined pretreatment has the advantage of reducing the amount of solvent used and constitutes a promising new method in biorefining.

Summing up, the research carried out in the last years has demonstrated that mechanical comminution or milling is an efficient technology in biomass processing, both as a sole pretreatment process and in combination with other techniques. Moreover, it facilitates the storage, densification and, ultimately, the supply chain of raw materials in a biomass processing plant, contributing to ease the logistic operations (Barakat et al., 2014). Another advantage of milling is the low concentration of inhibitory compounds released during this pretreatment process, which eases the downstream processing and constitutes a clear advantage compared to traditional techniques using catalysts and/or high temperatures and pressures (Kwon et al., 2016; Mankar et al., 2021). In addition, Shen et al. (2020) highlighted the lower biomass losses occurring during milling technology, such as ball milling, compared to other pretreatment methods.

Nevertheless, it is crucial to consider that milling is a high-energy consumption technology that may hinder its scalability due to high economic and energy costs. However, it always depends on the equipment used, the operational parameters, the physicochemical features of the biomass and the desired particle size (Barakat et al., 2014; Lomovsky et al., 2016). Thus, hardwood requires notably more energy for particle reduction than herbaceous materials, as proven by Cadoche and López (1989). For the same final particle size (1.6 mm) and mill type (hammer mill), these authors found that the energy required was 14.8 kWh/kg for corn stover, 42 kWh/kg for wheat straw and as much as 130 kWh/kg for hardwood. The same authors also studied the effect of the particle size on the energy spent to grind 22.4 mm hardwood chips in a knife mill, which notably increased from 8 kWh/kg for a slight particle reduction of 12.7 mm to 130 kWh/kg when the final particle size was 1.6 mm. Thus, selecting the most suitable grinding machine and the most favourable process parameters is crucial to promote the efficiency of this processing step (Moiceanu et al., 2019). Ball milling is known for its high-energy consumption compared to other milling devices. To overcome this handicap, multi-step grinding strategies or the combination of ball milling with chemical or physicochemical processes have been proposed, achieving substantial reductions in energy consumed (Sitotaw et al., 2021). On the other hand, the scalability of any milling methodology has been recognised as determining feature of the potential application in a large-scale LB processing strategy (Kim et al., 2016). The scalability is linked to the productivity or milling capacity of each type of method and clearly limits the potential of a particular technology to be used at an industrial scale.

Further research is therefore needed to overcome the drawbacks and limitations mentioned earlier and to develop more energy-efficient machines and novel applications of the different types of milling technologies. This will undoubtedly contribute to the more effective implementation of the most applicable milling techniques in biomass processing and conversion, paving the way to innovative advances in this research line.

3. Extrusion

Extrusion is a continuous physical pretreatment that unites mechanical and thermal effects in a single machine. It is carried out in extruders, which consist of one or two axis with different screw elements that can be adjusted to the desirable screw profile (see supplementary material). The screws spin inside a tight barrel whose temperature can be regulated. The biomass is continuously fed to the machine, where it is transported and forced to pass through the narrow space between the screw and the barrel wall, generating high shearing forces that alter fibres structures mechanically (Duque et al., 2017).

The main effects of extrusion pretreatment are particle size reduction and fibrillation (Chen et al., 2013; Tian et al., 2019). The impact of this pretreatment on the crystalline structure of lignocellulose is unclear. Some authors report an increase in the crystallinity index of extruded biomasses (Banvillet et al., 2021; Gu et al., 2018b). Others observe a decrease (Ai et al., 2020; Lu et al., 2018) or no changes at all (Marone et al., 2019).

When used alone, extrusion does not seem to affect the chemical composition of lignocellulosic biomass (Duque et al., 2018; Hjorth et al., 2011; Marone et al., 2019; Tian et al., 2022). However, the use of different reagents can promote the solubilisation of lignin (Liu et al., 2018), hemicellulose (Han et al., 2020; Lu et al., 2022) and other non-structural compounds (Duque et al., 2014b; Lu et al., 2018). Reactive extrusion is a particular mode of operation in which a chemical catalyst is used in the process, adding a chemical effect to the physical alteration of the biomass and improving the global performance of the pretreatment. The mechanical compaction of the material caused by extrusion removes air and water, enhancing the penetration of solvents into the biomass fibres (Liu et al., 2018; Lu et al., 2018). For instance, Liu et al. (2018) have proved the synergy of physical and chemical effects during extrusion processing. They extruded corn stover using NaOH as a catalyst and then compared the results obtained with those of an alkaline pretreatment followed by Paperindustriens Forskningsinstitute (PFI) refining. They found that the sugar yield of the NaOH-extruded corn stover was notably higher (78.75 %) than the yield obtained using only the alkaline pretreatment (50.08 %) or applying the sequential alkali plus mechanical refining treatment (54.83 %). Alkaline extrusion using NaOH has been widely studied, and promising results have been obtained from this pretreatment in the last years (Doménech et al., 2020; Duque et al., 2018; Liu et al., 2018; Tian et al., 2022). However, recent studies have explored the addition of other chemicals that are more environmentally friendly, such as ionic liquids (da Silva et al., 2013; Guiao et al., 2022b; Han et al., 2020), deep eutectic solvents (Ai et al., 2020), or glycerol (Lu et al., 2018). For example, Ai et al. (2020) highlighted the advantages of using a neutral-pH and biocompatible deep eutectic solvent (choline chloride: glycerol) to enable the continuous pretreatment of sorghum bagasse at high solids loading, improving the enzymatic digestibility of the extruded biomass up to 85 %. In some instances, the catalysts added during reactive extrusion also play a crucial role as flow modifiers, enabling a continuous and stable material flow inside the machine. In fact, extrusion of dry lignocellulosic biomass can lead to wearing of the machine and excessive torque values (Duque et al., 2017; Han et al., 2020) and water, in the form of moisture or added externally, may sometimes not be enough either (Duque et al., 2018; Kuster Moro et al., 2017).

Herbaceous materials have traditionally been considered more suitable for extrusion processing than woods and other highly lignified residual biomasses due to their greater softness (Duque et al., 2017; Ismail et al., 2022). However, the most recent publications about extrusion pretreatment show a renewed interest in using woody species and less common feedstocks, such as eucalyptus (Duque et al., 2018), Douglas fir (Gu et al., 2018a), aspen (Tian et al., 2019), pussy willow (Han et al., 2020), bamboo (Lu et al., 2018), olive stones (Doménech et al., 2020), or vegetal tomato plant waste (Moreno et al., 2021).

Extrusion technology has also been employed to pretreat food wastes, such as pecan nut shell (Villasante et al, 2021), soybean residue (Lee et al., 2019), fish by-products (Makoure et al., 2020), or grapefruit peel (Trujillo-Juárez et al., 2021). The aim of these investigations was to promote the release of sugars, or the extraction of interesting bioproducts (lipids, essential oils).

Continuous operation and the capability to work at high solids loadings are two of the most distinctive characteristics of extrusion pretreatment (Ai et al., 2020; Duque et al., 2014a; Zheng et al., 2014). The superior mixing capacity of extrusion, especially at high solids loadings, as well as the adaptability of extruders to the different process needs, are other of the advantages of this pretreatment (Duque et al., 2017; Fu et al., 2018; Gatt et al., 2019; Santala et al., 2013). In this respect, the modular configuration of some extruders allows them to carry out multiple operations. For instance, the bioextrusion process presented by Duque et al. (2014a) integrates the pretreatment, conditioning of the pretreated biomass and beginning of the EH, in a continuous process conceived to be performed in just one extrusion machine. Several variables influence the performance of extrusion, such as screw speed, temperature, liquid-to-solid ratio, torque or screw profile, all of which have been extensively studied in the literature (Duque et al., 2018; Gu et al., 2019; Han et al., 2020; Negro et al., 2015). Residence time inside the extruder is relatively short, varying between 2 and 15 min, according to the values reported in the literature (Choi et al., 2017; Gatt et al., 2019). Extrusion has been reported to shorten the time required for the pretreatment compared to batch experiments: i.e. da Silva et al. (2013) pretreated sugarcane bagasse with the ionic liquid 1-ethyl-3-methylimidazolium acetate and compared the results carrying out the process in an extruder with a batch reactor. A glucose yield of 91 % could be obtained in approximately 8 min of extrusion, while the time consumed in the batch reactor was 120 min for a similar 94 % glucose yield. However, for slower processes, the residence time may not be enough to complete the reactions, and extended incubation time is needed, as it is the case of hydrolytic enzymes (Vandenbossche et al., 2014). Alkaline extrusion with NaOH could also benefit from an external reaction time extension, as suggested by Liu et al. (2013). Other researchers have sought to extend the reaction time by recirculating the extruded material for successive extrusion passes (Ai et al., 2020; Banvillet et al., 2021; da Silva et al., 2013; Kuster Moro et al., 2017; Tian et al., 2022).

Sequential or hybrid pretreatments can be the answer for specific fractionation necessities in biorefineries. Extrusion has been proposed as a secondary pretreatment after milling (Fu et al., 2018), alkaline thermal pretreatment (Doménech et al., 2021), or steam explosion (Oliva et al., 2017). It has also been used as a first step before the diluted acid steam explosion (Ismail et al., 2022), ultrasonication (Byun et al., 2020; Zhang et al., 2020), ozone pretreatment (Karunanithy et al., 2014b) and MW (Karunanithy et al., 2014a). These sequential configurations usually successfully improved the production of sugars with respect to the first applied pretreatment. Another clear objective of these configurations is to use milder conditions in one or both pretreatments. For instance, Byun et al. (2020) tested alkaline extrusion followed by ultrasonication. They obtained 7 % more sugar production, higher lignin removal (17 % more), and a reduction of 26 % in the amount of alkali used compared to the case without ultrasonication. According to Karunanithy et al. (2014b), the previous extrusion of switchgrass and big bluestem reduced the time and ozone consumption compared to applying only ozonation. Furthermore, some authors highlight the advantage of sequential pretreatments to achieve chemical-free processes without losing effectivity (Fu et al., 2018; Karunanithy et al., 2014a; Zhang et al., 2020). Nevertheless, the actual application of these sequential pretreatments remains uncertain since all the tests so far have only been carried out at the laboratory or bench-scale, and the integration of the different technologies could be challenging at a greater scale, also in terms of increased costs.

There is few information in the scientific literature about the energy aspects of extrusion pretreatment (Guiao et al., 2022a). The mechanical energy consumption in extrusion is usually expressed as the amount of energy required to produce a kg of pretreated material (specific mechanical energy, SME), and it heavily depends on several variable operations, namely the torque, the motor power, the screw speed and the mass flow inside the extruder. SME values vary for different feedstocks and processes, with typical values between 80 and 370 Wh/kg (Doménech et al., 2020; Duque et al., 2018; Gu et al., 2020). The energy input can be further reduced when a previous chemical method is applied to soften the rigid lignocellulosic structure (Tian et al., 2019). Extrusion pretreatment has been considered less energy-intensive than conventional pretreatments for certain applications. For instance, using extrusion resulted in up to 63 % energy saving compared to ultra-fine grinding (Rol et al., 2017). A comparison study among different milling strategies concluded that extrusion had a much lower specific energy consumption than grinding mills, i.e. 0.14 kWh/kg (extrusion) compared to 0.26 kWh/kg (hammer milling) or 0.74 kWh/kg (impact milling) (Fu et al., 2018). Furthermore, the energy balance for biogas production using extrusion as pretreatment was positive, with energetic gains between 10 and 68 % (Hjorth et al., 2011; Souza et al., 2022).

To sum up, the scientific community continues nowadays exploring the potential of extrusion as a pretreatment method to produce a diversity of bio-based products. The latest studies have tested the performance of new biomasses, new catalysts and process configurations. Some have offered new insights into how extrusion affects lignocellulosic biomass. Table 3 summarises some examples of the most recent advances. In spite of the uncertainties about the energetic and economic costs and the fact that the current research has not yet reached the level of development necessary for industrial applications, the technology is expected to continue attracting research interest for its use in biorefineries in the following years, given the flexibility and adaptability of extrusion pretreatment.

4. Ultrasonication

Ultrasonication is another common physical technology applied to

Table 3
Selected examples of recent publications involving extrusion pretreatment.

Feedstock	Type of extruder	Operating conditions	Application	Main findings	Reference
Barley straw / Vine shoots	Twin-screw extruder	Addition of water 0.2 L/S; 500 rpm; 85 °C	• Anaerobic digestion	• Increase of the surface area exposed using extrusion. • No positive effect on the biodegradability of the biomasses due to accumulation of volatile fatty acids.	Hidalgo et al. (2022)
Corn stover	Twin-screw extruder	Addition of NaOH 0.06 g NaOH/g dry matter 2 L/S Heat preservation, 1 h, 99 °C	• Bioethanol production	• 50 % more delignification using alkaline extrusion than only alkali. • Higher total sugar yield obtained using alkaline extrusion (79 %) than alkaline pretreatment (51 %), or sequential alkali plus refining (55 %). • Synergy between chemical and physical effects in reactive extrusion.	Liu et al. (2018)
Vegetal tomato plant waste	Twin-screw extruder	Addition of NaOH 0.06 or 0.12 g NaOH/g dry matter 150 rpm; 100 °C 1 or 3 passes	• Sugars production	• Recovery of the major part of extractable compounds in vegetal tomato plant waste. • Enhancement of the enzymatic digestibility. Increase with the amount of catalyst and number of passes in the extruder.	Moreno et al. (2021)
Olive stones	Twin-screw extruder	Addition of NaOH 0.05 or 0.15 g NaOH/g dry matter 2 L/S; 150 rpm; 100–125-150 °C Neutralization with H_2SO_4	• Sugars production	• Most of the carbohydrates in olive stones recovered in the solid fraction. • Increase of the enzymatic digestibility at high NaOH loading and temperature up to 125 °C. • 60 % conversion of carbohydrates to glucose and xylose.	Doménech et al. (2020)
Sorghum bagasse	Twin-screw extruder	Addition of choline chloride:glycerol (CholCl:Gly) 1–2 L/S; 35 rpm; 150 or 180 °C 4 to 10 passes	• Fractionation for the production of sugars and lignin	• Reduction of pretreatment time to 16 min and increase of the solids loading up to 50 % compared to batch experiments (2 h, 10 % solids). • Higher destructuration and increased enzymatic digestibility. • Positive impact of the number of passes and temperature. • Lignin kept the basic structure characteristics after the pretreatment.	Ai et al. (2020)
Pussy willow powder	Twin-screw extruder	Addition of 1-ethyl-3-methylimidazoliun acetate (EmimAC) (plus dimethyl sulfoxide, DMSO) 1–6 L/S; 2–15 rpm; 140 or 160 °C	• Sugars production	• Disruption and fibrillation of the cell wall, solubilization of components, destruction of the crystalline structure and increase of the enzymatic digestibility. • Addition of DMSO to EmimAc did not improve the results • Better results at low screw speed and high temperature.	Han et al. (2020)
Bamboo	Single-screw extruder	Biomass impregnated in glycerol, 1:8 ratio, 120 °C, 2 h 55 rpm; 50 °C 2 passes	• Lignocellulose nanofibrils	• Removal of lignocellulose and ash, retention of hemicellulose and cellulose. • Small decrease in crystallinity to 48 % compared to 58 % without extrusion. • Increase of the solvent absorption ration from 30 to 78 % after two extrusion passes. • 60 kWh/ton energy consumption, less than conventional refining.	Lu et al. (2018)

lignocellulosic biomass and wastes materials in biorefining processes. Ultrasonication technology is based on the cavitation effect caused during radiation with ultrasonic energy, an acoustic wave that oscillates at frequencies above 16 kHz.

Acoustic cavitation is induced when the ultrasonic wave is propagated in a specific liquid medium, promoting compression (when the molecules of the solvent are pressed together) and rarefaction (when the molecules of the solvent are separated) cycles that form microbubbles containing gas. The collapse of such microbubbles triggers different physical and chemical effects, including heating, acoustic cavitation, acoustic streaming, nebulisation, and radical formation, thus altering biomass structure (Flores et al., 2021; Gallo et al., 2018). The chemical effect (*i.e.*, radical formation) is more intense at sonication frequencies of ca. 300 kHz. In contrast, the microbubbles generated at frequencies around 20–50 kHz, produced in lower numbers but exhibiting higher diameters, release higher mechanical energy (Bussemaker & Zhang, 2013). Most ultrasonication pretreatment processes operate at frequencies below 50 kHz, making the physical effects the main mechanisms influencing biomass structure, although chemical hydroxide attacks can also occur. In addition, the energy released from the bubbles collapse during ultrasonication of biomass has low penetration potential and is therefore retained on the surface of the corresponding material. This makes delignification and extraction of high-added-value chemicals the main applications of ultrasonication on biomass (Ong et al., 2021; Sharma et al., 2021; Sharma et al., 2022; Subhedar et al., 2018). Ultrasonication induces lignin removal, as it is the external component of lignocellulosic biomass and is in direct contact with the solvent (Ong et al., 2021; Subhedar et al., 2018). On the other hand, ultrasonication can promote erosion and fragmentation of vegetal structures, increasing the accessibility of the corresponding solvent to biomass and leading to the solubilisation of the product(s) of interest (Chemat et al., 2017).

The most recent advances in using ultrasonication technology for both delignification of lignocellulosic residues and the extraction of added-value compounds from food waste are summarised in Table 4. Compared to conventional technologies (e.g., alkali extraction, organosolv), ultrasonication-assisted methods require lower process times and temperatures, and the reactor can operate at atmospheric pressure. These ultrasonic-assisted treatments also result in higher extraction/delignification efficiencies. For instance, ultrasonication of grass clipping slightly modified the chemical composition of this raw material. At the same time, ultrasound-assisted alkaline pretreatment with 0.5 % NaOH or 0.5 % $Ca(OH)_2$ reduced the lignin content by 40.5 % and 36.6

Table 4
Examples of ultrasound and ultrasound-assisted methods for biomass pretreatment and extraction of industrially relevant products.

Feedstock	Treatment	Operating conditions	Application	Main findings	Reference
Agave leaves	Ultrasound bath	42 kHz and 132 W, 30–60 min	Enzyme production	• 20–25 % lower extractives and ash • Increase phenol recovery • Delignification increases at longer residence times	Contreras-Hernández et al. (2018)
Grass clipping	Ultrasonic cell crusher/ ultrasound-assisted alkaline pretreatment	25 kHz and 5 W/mL, in presence and absence of 0.5 % NaOH or 0.5 % $Ca(OH)_2$	Sugar recovery	• Similar chemical composition with ultrasonication alone • Assisted-method decreased the lignin content by 37–41 % compared to 31–36 % with alkaline treatment alone • 2 times higher saccharification yields	Wang et al. (2017b)
Oil palm frond	Ultrasound-assisted alkaline treatment combined with aqueous deep eutectic solvent (DES)	Horn-type ultrasonicator, 20 kHz, amplitude of 70 % for 30 min; 1:4 water to DES ratio and 2.5 % (w/v) NaOH-aqueous DES	Sugar recovery	• 50 % delignification • Ultrasonication increased lignin removal by 1.5 times	Ong et al. (2021)
Rice straw	Ultrasound-assisted Fenton process	Horn-type ultrasonicator, 22 kHz, 200–600 W for 3 h; 1:1 solution of 1.76 M H_2O_2 and 0.04 M $FeSO_4·7H_2O$, both with a pH value of 2.5	Sugar recovery	• 1.5 times higher saccharification yields than treatment without ultrasonication • Higher specific surface area available • Lower DP (degree of polymerization) values	Xiong et al. (2017)
Sugarcane bagasse	Ultrasonication combined with surfactant-assisted ionic liquid treatment	horn-type ultrasonicator, 20 kHz, 100 W for 60 min; 5 g of 1-butyl-3-methylimidazolium chloride [Bmim]Cl and 300 mg of surfactant PEG-8000	Sugar recovery	• 1.3–1.5 higher sugar yields compared to ultrasound and surfactant-aasisted ionic liquid treatments, respectively • Promising green solvent strategy • Increase in surface area • Reduce crystallinity due to swelling and solubilization of cellulose	Sharma et al. (2021)
Birch sawdust	Ultrasound-assisted biological delignfication (*Myrothecium verrucaria*)	Ultrasonic Generator, 200 W for 20 min at 1:20 solid to liquid ratio	Delignification	• Delignification increased up to 70 % with combined pretreatment (1.5 and 5 times higher than with *M. verrucaria* and ultrasonication alone) • Significant modification of the surface morphology and chemical structure	Wang et al. (2017a)
Out hulls	Ultrasound-assisted alkaline pretreatment	35 kHz for 10 min in water, followed by an incubation in 5 M NaOH at 80 °C for 9 h	Hemicellulose extraction	• Recovery of 72 % of the total hemicellulose content • Higher yields than autoclaving, microwave, deep eutectic solvents and alkaline treatments	Schmitz et al. (2021)
Orange peel waste	Ultrasound-assisted dilute acid hydrolysis	40 kHz and 60 W for 34.2 min; working at 5.8 % solid loading with 1.2 % sulfuric acid concentration	Extraction of essential oils, pectin and bacterial cellulose	• Maximum production yields: 0.12 % w/w essential oils; 45 % w/w pectin; 5.82 g of bacterial cellulose per 100 g of waste. • Similar yields at laboratory and pilot scales	Karanicola et al. (2021)
Microalgae (*Nannochloropsis oceanica*)	Ultrasound-assisted N, N, N', N'-tetraethyl-1,3-propanediamine (TEPDA)	20 kHz and 0.5 W/mL power intensity at a volume ratio of 1:4 microalgae:TEPDA	Extraction of lipids	• Effective disruption of microalgae cell wall • Rearrangement of the cell membrane, resulting in cell membrane leakage • Almost complete lipid extraction within 2 h	Guo et al. (2022)
Corn stover	Combined ultrasound treatment with ball milling and hydrothermal methods as well as with pulsed electric field (PEF)	Ball milling at 220 rpm for 1, 3, and 5 h, ultrasonic generator, 40 kHz and 360 W for 30 min, followed by hydrothermal treatment (185–230 ℃) for 1–1.5 h	Extraction of xylooligosaccharides (XOS)	• Recovery of 80.4 % XOS, containing 27 % of the functional XOS (X2-X4) • The solid residue reduced the lignin content by 35 % • Very high yields (93 %) during the saccharification step	Zhang et al. (2022)
Grape stems	Combination of PEF with ultrasound	35 KHz and 320 W; low-electric field strength; 1 kV/cm for 30 min	Extraction of polyphenol and volatile compounds	• Combined treatment increase polyphenol recovery by 17 % in 1:1 (v/v) methanol:water • Ultrasonication extraction increased polyphenol recovery	Ntourtoglou et al. (2022)

(continued on next page)

Table 4 (*continued*)

Feedstock	Treatment	Operating conditions	Application	Main findings	Reference
				by 35 % when using water alone as solvent • Higher concentrations of volatile compounds can be obtained (from 0.73 to 2.44 mg/Kg)	

%, respectively, compared to 36.2 % and 31.4 % delignification rate of the alkaline treatment alone (Wang et al., 2017b). This ultrasound-assisted strategy also doubled the subsequent saccharification yields, underlying the high potential of this method for lignocellulose pretreatment. Ultrasound-assisted alkaline treatment has also been combined with an aqueous deep eutectic solvent (DES) for the delignification of oil palm fronds (Ong et al., 2021). This treatment reached delignification values as high as 47 %, increasing lignin removal by 1.5 times compared to treatment without ultrasonication (30.9 % delignification rate). An interesting approach has been published by Sharma et al. (2021), who sequentially combined ultrasonication with surfactant-assisted ionic liquid treatment, which is considered a promising 'green solvent' for biorefineries. This strategy increased sugar yields by 1.3–1.5, compared to ultrasound and surfactant-assisted ionic liquid treatments, from 176 mg/g and 199 mg/g to 254 mg/g, respectively. Biological delignification coupled with ultrasound assistance has also resulted in higher lignin removal when compared to ultrasound or biological processes alone. Wang et al. (2017a) treated birch sawdust with the fungus *Myrothecium verrucaria* and ultrasonication for lignin degradation. Compared to the separated processes, the use of fungal and ultrasound treatments resulted in a lignin degradation increase up to 68.0 % in comparison to 45.5 % and 13.8 %, respectively. Together with lignin removal, polysaccharide solubilisation can also be observed during biomass ultrasonication due to the formed radicals' oxidising capacity. Ultrasound-assisted alkaline pretreatment was the most effective method for hemicellulose recovery from out hulls (72 % of the total hemicellulose content) compared to autoclaving, ultrasonication, MW, deep eutectic solvents, and alkaline treatments (Schmitz et al., 2021).

Ultrasound and ultrasound-assisted treatments have also been applied to processing food-derived waste to produce industrial-relevant bio-based products within a circular bioeconomy perspective. These approaches have been previously reviewed in the literature elsewhere (Esteban-Lustres et al., 2022; Sharma et al., 2021; Sharma et al., 2022; Sirohi et al., 2020). Production of valuable commodities such as essential oils, pectin and bacterial cellulose can be obtained by ultrasound-assisted dilute acid hydrolysis from orange peel waste (Karanicola et al., 2021). Similarly, ultrasound treatment has also been combined with ball milling, hydrothermal methods, and pulsed electric field (PEF) treatment. Combination of ultrasound, ball milling and hydrothermal treatment allowed to recover of 80.4 % of xylooligosaccharides (XOS) from corn stover, containing about 27 % of the functional XOS (X2-X4) (Zhang et al., 2022). In addition, the remaining solid residue also reduced the lignin content by 35 % and reached 92.6 % glucose yield during the EH step. On the other hand, combining PEF with ultrasound technology promoted the extraction of volatile compounds and polyphenols from grape stems (Ntourtoglou et al., 2022).

Ultrasonication is an attractive physical pretreatment with a high potential for processing lignocellulosic residues and waste materials, either alone or in combination with other novel and conventional methods, to increase pretreatment efficiencies and promote process intensification during biomass conversion. Still, this technology is highly energy demanding. Depending on biomass type and process targets, sonication-specific energies vary greatly. In this regard, Bundhoo and Mohee (2018) reviewed the use of ultrasound irradiation for organic matter solubilisation and biofuel (biogas and biohydrogen) production, listing specific energies inputs between 0.5 kJ/g of total solids and 234 kJ/g of volatile solids. Overall, there is a need to investigate ultrasonication further to provide more information and optimise essential parameters, including energy requirements, to overcome current challenges and advance towards scaling up these processes.

5. Microwave radiation

A pretreatment that currently focuses much interest is pretreatment through MW irradiation. This technology enhances hydrothermal biomass fractionation by promoting the interaction between substrate and reaction medium, although applying low pretreatment severities (Tsubaki et al., 2016).

Microwaves are non-ionising radiations using wavelengths of 0.01–1 m and 300–300,000 MHz (Hassan et al., 2018; Kostas et al., 2017). MW heating promotes two main mechanisms that induce rapid heating in the presence of ions or polar molecules: ionic conduction and dipole rotation. The dipole rotations mechanism forces to realign the polar molecules with the electric field generated by MW. During this phenomenon, molecules interact with themselves, realising energy in the form of heat through dielectric loss and molecular friction. In the presence of an MW-absorbing material, this energy is absorbed irreversibly, causing rapid volumetric heating (Kostas et al., 2017). This unique heating mechanism benefits the MW system by reducing heating time, offering a uniform and selective volumetric heating performance, and increasing energy transfer efficiency. To promote selective heating, substrates must be heterogeneous, containing different materials or phases with varying dissipation factors. The latter are defined as the relation between the dielectric loss factor (the ability of the material to dissipate in the form of heat the absorbed electromagnetic energy) and the dielectric constant (the ability of the material to store electromagnetic energy) (Kostas et al., 2017; Tsubaki et al., 2018). Selective heating provides better control of the heating process, reducing the risk of generating biomass degradation products that can inhibit hydrolytic enzymes and fermentative microorganisms in subsequent conversion process steps. In addition, selective heating has no temperature requirements (i.e., MW-based pretreatments can be performed in cold environments), thus avoiding the need of ablative surfaces or hot gases, and results in higher heat transfer rates when compared to conventional heating systems. These advantages offer the possibility of processing large particle sizes and using smaller MW processing facilities, which positively impacts capital costs and may contribute to obtaining a wider range of bioproducts.

MW technologies have also emerged as advanced techniques in extracting various added-value compounds due to multiple advantages, such as improving extraction yields, reducing process time, and low solvent consumption. However, some of the drawbacks of this technique are the need for special equipment, low selectivity and the possible degradation of some bioactive compounds. Using suitable solvents and adjusting operating conditions during MW-assisted extraction make it possible to extract compounds of interest, such as anthocyanins, flavonoids or essential oils. Castro-Muñoz et al. (2022) review the latest insights on emerging techniques, including MW-assisted extraction, high voltage electrical discharge or ultrasound-assisted extraction, to extract compounds of industrial relevance from agro-food waste. The application of technologies that use MW on biomass has been widely studied in recent decades. Its use in a previous phase of drying, especially applicable as a previous phase to the processes of transformation of biomass through thermochemical processes, or the prevention of the degradation of possible bioactive compounds of biomass, has been reported (Chojnacka et al., 2021). Particularly the use of MW-assisted pyrolysis of biomass without oxygen under high temperatures (>400 °C) to convert biomass to liquid (bio-oil), solid (biochar) and gaseous (syngas) fraction (Siddique et al., 2022; Suriapparao and Tejasvi, 2022) or gasification (Arpia et al., 2022) has been considered.

During the pretreatment of MW lignocellulosic feedstocks, specific polar regions of this heterogeneous material support selective heating and create internal 'hotspots' that promote biomass disruption and swelling (Kostas et al., 2017). The internal fragmentation contributes to reducing particle size and cellulose crystallinity index and increasing the available surface area, improving process performance during the subsequent hydrolysis step (Hassan et al., 2018).

Most published MW pretreatment works are combined with other pretreatments (Table 5 shows some examples).

Regarding the limitations of MW technology, there is a need of experimental data about the performance of MW reactors for biomass pretreatment (Aguilar-Reynosa et al., 2017). Moreover, further research efforts must be done to develop novel MW systems and reactors that allow operating at high solid loads, while promoting a homogeneous heat transfer. In addition, Kostas et al., 2017 have pointed out at the necessity to make progress in the understanding of the changes occurred in LB pretreated by MW that would eventually support the design of more realistic and scalable biorefinering strategies using this technology.

6. Research Needs and Future Directions

Using alternative and novel feedstocks to obtain biofuels and bioproducts in a biorefinery-type strategy requires applying efficient and selective fractionation techniques that maximise the use of all biomass components while at the same time contributing to the sustainability of

Table 5
Examples of microwave assisted methods for biomass pretreatment.

Type	Feedstock	Operating conditions	Main findings	Application	Reference
Combined mechanical/microwave	Wheat Straw, *Miscanthus,* Short-rotation willow	200 °C −10 min (Microwave reactor) followed by wet-milling pretreatment (10 min milling time)	• Supplementary milling led to a 1.3×, 1.6 × and 3 × enhancement in glucose saccharification yield after 24 h for straw, *Miscanthus* and willow, respectively • Milling energy savings of 98, 97 and 91 %l compared to the unmilled case	Sugars production	Ibbett et al. (2018)
One-stage microwave-alkali-assisted pretreatment	Wheat straw	Temperature 100 °C 3 %NaOH concentration, 20 min.	• Evidence of the effect of temperature. Existence of the non-thermal effect of MW in MW-assisted biomass treatment. • Microwaves could reduce approximately 50 %–75 % of the reaction time or 67 % of the chemical consumption in comparison with traditional heating hydrolysis	Cellulose fibre isolation	Liu et al. (2021)
Combined Deep eutectic solvents; /microwave	*Miscanthus,* corn stover, switchgrasss	800 W power, temperature 152 °C, and 45 s ChCl: LA, 10 wt % solid loading.	• Lignin and xylan were substantially removed while most of cellulose was retained. • The pretreatment increased the cellulose digestibility by 2–5 folds. • Lignin was recovered with relatively high purity (85–87 %). • Microwave-assisted DES pretreatment appeared to require much less energy than DES pretreatment (3.6×10^4 J vs 2.49×10^6 J)	Biomass fractionation	Chen & Wan (2018)
One-stage microwave–chemical–assisted pretreatment.	Maize stillage	300 W (power of microwave generator), 54 PSI, 15 min	• Highest glucose concentrations (104 mg/g of stillage DW) and the highest total yield of cellulose hydrolysis (75.8 %), a relatively low concentration of by-products was observed (6.8 mg HMF/g of DW; 6.0 mg furfural/g of DM).	Bioethanol	Mikulski et al. (2019)
Microwave-assisted extraction	Brewer‘s spent grain (BSG), Kale Stem spent coffee ground	110 °C, 10 min, 0.5 M NaOH (BSG) 102 °C, 15.3 min, uncatalyzed (Kale)	• Best results microwave-alkali assisted (protein extraction yield 94 % • 72.59 % protein extraction yield for kale	Recovery protein from agro-food waste	Barrios et al. (2022)
Microwave plus deep eutectic solvent	Sugarcane bagasse	Choline chloride: Ethylene glycol: $NiCl_2.6H_20$ (CC:EG:NI) at a molar ratio 1:2:0.016 with 20w%; 100 °C microwave heating for 30 min	• 84 % delignification and 99 % digestibility of sugarcane bagasse	Production fermentable sugars	Chourasia et al. (2022)
Microwave-assisted acid pretreatment	Sugarcane straw	50-Hz reactor 162 °C and 0.6 % sulphuric acid (w/v) for 2 min.	• Total sugars recovery 72.2 % • Hydrolysates with low inhibitors concentrations which allows the hydrolysates to be fermented without a detoxification	Production bio-butyric acid	Fonseca et al. (2021)
Diluted acid/microwave	Agave	H_2SO_4 1.5 % (v/v); Biomass/ H_2SO_4 ratio 1:36 (w/v); at 140 °C 10 min	• Hemicellulose removal was superior in pretreatment-assisted with microwave (MW) (77.5 %) • Enzymatic hydrolysis yield was 2-fold higher in agave pretreated with MW. • Hydrolysates from MW resulted in a high glucose concentration without inhibitors. • MW pretreatment significantly reduce time compared to conventional heating.	Sugars production	Ríos-González et al. (2021)

the whole biomass conversion process. The considerable interest that the application of physical technologies as described herein to pretreat biomass feedstocks has raised is shown in the substantial number of investigations carried out in the last decade using a great variety of different biomass materials, including food wastes and lignocellulosic residues. Nevertheless, despite the amount of research already carried out, there are still gaps in the knowledge that hinder their industrial application potential. For instance, energy consumption is the primary concern associated physical pretreatment method, which is often overlooked in research studies. Some works do give figures for the energy demand of the equipment (Cadoche and López, 1989; Vidal et al., 2011), but these data need to be put in a critical context that goes beyond the machine and reaches the whole production process. In this regard, techno-economic analysis is an essential tool to determine the potential of the chosen pretreatments and highlight their advantages over conventional systems.

Furthermore, the machines where the physical pretreatments take place usually have a high degree of complexity, which raises some doubts about their scaling up for their use in industries with high production volumes. Alternatively, applications in the field of low-volume, high-added value bioproducts seem to be more convenient for this kind of pretreatments, especially for ultrasonication and MW (Castro-Muñoz et al., 2022; Ong et al., 2021). Thus, the adequate selection of the targeted bioproduct is crucial to promote the implementation of physical pretreatments at an industrial scale.

The analysis of the recent scientific literature reveals that combined pretreatments are the best way to exploit the full potential of physical and chemical methods to alter the biomass structure. The combination of a physical pretreatment with chemicals or catalysts usually results in a synergic effect that can lead to a reduction of the chemical consumption and/or the use of milder operation conditions (Liu et al. (2018), Zhou and Tian (2022). In this respect, it is essential to study the environmental and economic impact of combined process in comparison to separate pretreatments, advancing in the reduction and possible recycling of the catalysts used. In addition, the mechanistic basis that induce the changes in biomass structure at a molecular level still need to be fully understood, and the advance of fundamental research in this area must be considered.

A particularly interesting field of study that is recently emerging is the coupling of physical pretreatments with the biological conversion of biomass. To date, only a few studies have addressed this type of cotreatment process, using hydrolytic enzymes and microorganisms in combination with milling, extrusion or ultrasound methods (Balch et al., 2020;Duque et al., 2014a; Navarro et al., 2018; Wang et al. 2017a; Wu et al., 2021). This combined process presents clear advantages, such as mild temperature conditions and no chemicals addition; therefore, they constitute a promising line of research to explore. Research should advance the knowledge of the underlying mechanisms of cotreatment. Furthermore, techno-economic analyses that include the energy requirements should be performed to evaluate the cotreatment under industrial conditions.

Finally, the bibliographic review concluded that there is still further room for optimising ultrasonication and microwave conditions for biomass pretreatment. Although different studies can be found related to the use of MW technology in drying and extraction systems on a pilot scale, there are few studies on using MW reactors for biomass pretreatment. As Aguilar-Reynosa et al. (2017) pointed out, that this aspect should devote further research efforts. It is also advisable to advance in developing novel MW systems and reactors to operate at high solid loads and to favour homogeneous heat transfer. It is also crucial to understand the different behaviours of major lignocellulosic biomass components and their dielectric properties to design better processes for this technology (Kostas et al., 2017).

7. Conclusions

The high versatility of physical pretreatments is well demonstrated in the substantial number of applications tested up to now, leading to the obtaining of different compounds or products from biomass biorefinering. These technologies, alone or in combination with other hydrothermal, chemical or even biological pretreatments have been proven to be effective to alter the structure of a great variety of biomass feedstocks, representing a step forward in the search of a specific fractionation technique for biomass processing. However, there is still a need for further research to boost their application at a scale close to industrial conditions~~.~~

Declaration of Competing Interest

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

Data availability

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

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Appendix A. Supplementary data

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