



Environment friendly emerging techniques for the treatment of waste biomass: a focus on microwave and ultrasonication processes

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Abstract

In the recent past, an increasing interest is mostly observed in using microwave and ultrasonic irradiation to aid the biological conversion of waste materials into value-added products. This study is focused on various individual impacts of microwaves and ultrasonic waves for the treatment of biomass before the synthesis of value-added products. Following, a comprehensive review of the mechanisms governing microwaves and ultrasonication as the treatment methods, their effects on biomass disruption, solubilization of organic matter, modification of the crystalline structure, enzymatic hydrolysis and production of reducing sugars was performed. However, based on the lab-scale experiments evaluated, microwaves and ultrasonication were studied to be economically and energetically ineffective despite their beneficial effects on the waste biomass. This article reviews some of the difficulties associated with using microwaves and ultrasonic irradiation for the efficient processing of waste biomasses and identified some potential directions for future study.

Keywords Lignocellulosic biomass · Biomass pretreatment · Microwave irradiation · Ultrasonic irradiation · Crystallinity index · Cellulose depolymerization · Acoustic cavitation · Thermo-chemical conversion

Abbreviations

LCB	Lignocellulosic Biomass
MAB	Marine algal biomass
CO ₂	Carbon dioxide
kHz	Kilohertz
MHz	Megahertz
GHz	Gigahertz
MCC	Microcrystalline cellulose
NaOH	Sodium hydroxide
CI	Crystallinity index
SB	Sugarcane bagasse
DP	Degree of polymerization

Introduction

The world has become more dependent on fossil fuels as there is a massive increase in the human population. The ever-increasing human population may equal 8.5 billion in 2030 and 9.7 billion in 2050, according to a United Nations estimate (Wiebe et al. 2019). Food and energy needs are anticipated to increase correspondingly (Kumar Sarangi et al. 2022). In order to lessen our dependence on petroleum products, substantial research has been done over the past few decades to identify renewable energy sources (Singhanian et al. 2022b). The International Energy Agency (IEA) defines biorefinery as “the sustainable processing of different biomass to various biomass-based materials such as food, chemicals, feed, and bioenergy such as biofuels and heat.” Carbon sequestration, the production of bioenergy, and the creation of bioproducts are only a few advantages of biomass, a regenerative carbon source. Only when conversion and valorization processes are well thought out and methodical can biomass be used as a viable raw material in a biorefinery. Biorefineries may employ any kind of biomass produced by companies, homes, agriculture, and aquaculture. Although it has a large supply, biomass has a poor energy density. Low calorific value, seasonality, and location

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are obstacles to the use of biomass (Basu 2018). The different conversion wastes are converted into worthwhile bioproducts using the integrated biorefinery process. The fundamental building block of a biorefinery is the systematic integration of all conversion techniques, which results in the production of sustainable bioproducts like bioenergy, biofuels, biochemicals, and other value-added bioproducts from biomass raw feed like LCB, algae, and other wastes. Although biorefinery is a renewable process for converting biomass using various treatment techniques into value-added products utilizing various technologies, its implementation and utilization mostly depend on its economic value, availability, and other factors. The predominant categories of sustainable sources are lignocellulosic biomass (LCB), marine algal biomass (MAB) (Anto et al. 2020), and starchy food crops (Sidana and Yadav 2022). These resources are used to produce biofuels and other compounds with additional value (Duarah et al. 2022). The advantage of using food crops in this situation is that no substantial preparation is required. However, the rivalry for cultivable land prevents them from being employed for very long (Etim et al. 2020; Pocha et al. 2022). MAB is a promising raw material; however, farming operations need expensive infrastructure for optimal development, harvesting, and drying (Tan et al. 2018).

According to the annual index, there are around 1.3 billion tonnes of LCB produced worldwide each year, with just 3% coming from non-food bioproducts, biobased chemicals,

and biobased energy (Baruah et al. 2018). Nevertheless, although it is widely distributed worldwide, LCB generally needs to be pretreated. Organic materials with hydrogen, carbon and oxygen atoms make up biomass resources (Shahni et al. 2021). Different types of lignocellulosic biomass contain a different proportion of cellulose, hemicellulose, and lignin as shown in Table 1.

The current energy-climate change problem may be resolved using biomass-derived energy sources, such as biogases and biofuels, from various organic-based matter types (Santana et al. 2021; Singhanian et al. 2008; Yadav et al. 2022). Additionally, since the quantity of CO₂ emitted during combustion is equal to the amount of CO₂ absorbed by trees and plants during photosynthesis, the production and consumption of biofuels produce no net emissions of the gas (Gambhir and Tavoni 2019). More study of advanced production of bioenergy technology is required to maximize yield, reduce environmental pollution, and enhance energy efficiency (Foong et al. 2021; Joglekar et al. 2019). Interestingly, the pretreatment stage may determine whether the entire bio-refining process is successful or unsuccessful (Shabbirahmed et al. 2022). The high expenses of pretreatment procedures and overpriced hydrolytic enzymes may be the cause of the delay in the development of lignocellulose-based biorefineries in underdeveloped nations (Sreekala et al. 2022). Furthermore, even after significant investments, the established methods might not yield attractive returns.

Table 1 Constituents of various lignocellulosic biomasses

Biomass	Annual production (in tons)	% Cellulose	% Hemicellulose	% Lignin	References
Eucalyptus leaves	—	46.7	19.8	27.9	(Salem et al. 2021)
Bamboo	168 million	43.2	27.1	26.7	(Salem et al. 2021)
Yam peels	—	36.8	14.7	8.64	(Awoyale et al. 2021)
Acai seeds	1.1 million	32.8	32.5	22.6	(de Paula Protásio et al. 2022)
Corn cover	120 million	24.8	51.4	12.8	(Singh et al. 2020)
Corn cob	—	32.6	48.8	32.6	(Singh et al. 2020)
Cocoa pod husk	7800 million	14.1	10.5	27.6	(Kley Valladares-Diestra et al. 2022)
Coconut husk	—	33.3	14.6	32.4	(Din et al. 2021)
Oil palm empty fruit bunch	44.84	39.0	23.1	20.1	(Yimlamai et al. 2021)
Sorghum stalk	63.5 million	38.2	33.0	9.9	(Xu et al. 2017)
Corn cob	—	42.0	45.9	2.8	(Pérez-Rodríguez et al. 2017)
Buckwheat husk	0.76	40.0	32.0	27.0	(Nakamura et al. 2019)
Rice husk	100 million	32.5	13.2	25.0	(Hafid et al. 2021)
Cassava pulp	—	17.7	9.3	2.4	(Papathoti et al. 2021)
Sugarcane bagasse	54 million	38.9	23.7	34.1	(Philippini et al. 2019)
Cotton stalks	50 million	37.6	12.4	30.1	(Meehian and Jana 2017)
Corn stover	120 million	36.6	21.2	16.9	(Sheng et al. 2021)
Groundnut shell	11 million	37.0	18.7	28.0	(Subhedar et al. 2018)
Coffee grounds	0.5 million	33.10	30.03	24.52	(Huang et al. 2016)
Olive pomace	30	19.0	22.0	40.0	(Álvarez et al. 2018)

As a result, the pretreatment process plays a crucial role in using LCB as a feedstock. An efficient and hygienic way for the expansion of surface area and improved availability to enzyme-binding sites is provided by biomass pretreatment (Alkarimiah et al. 2022). By disorganizing the lignin, pretreatment has a tendency to break down lignocellulosic biomasses into subcomponents and curb their inherent resistance (Tong 2019). Thus, it makes hemicellulose and cellulose more susceptible to enzymatic hydrolysis for creating simple sugars, which on fermentation produce biofuels (Prasad et al. 2019). Thermal effects are regarded as a critical part of many reactions, and microwave-based technology is thought to make it possible to redefine a vast number of these reactions (Halder and Purkait 2021). Furthermore, the microwave-based heating technique is a prospective substitute technology for conventional heating due to its effectiveness and simplicity of use. The processing time is ten times faster than that of other heating systems, which results in a reduction in energy consumption (Arpia et al. 2021). It does not produce trash or smoke, nor does it call for the utilization of solvents, separation agents, or other auxiliary chemicals (Aguilar-Reynosa et al. 2017). The use of ultrasonic technology during the pretreatment of biomass has recently emerged as one of the leading technologies among other comparable ones due to its quick reaction time and efficiency in converting biomass (Babadi et al. 2022; Sodhi et al. 2022). When processing biomass, ultrasound in the 20 kHz to 1 MHz range is employed chiefly to create oxidizing radicals that disintegrate the intricate network of lignocellulosic structure (Sasmal and Mohanty 2018). Researchers are keen on microwave-assisted and ultrasonic pretreatment techniques among the approaches for converting lignocellulosic biomass into biofuels.

Additionally, a thorough comprehension of the mechanisms underlying how such environmentally friendly methods interact with biomass will help to increase the effectiveness of the pretreatment process (Uthirakrishnan et al. 2022). Numerous studies on various conventional techniques are examined (Singhanian et al. 2015), but only a few articles are available covering emerging pretreatment methods, including greener techniques. Furthermore, to our knowledge, mechanistic data and the parameters influencing these developing technologies are hardly ever examined in any of the prior literature. So, the present review article focuses extensively on the advancements in ultrasonic and microwave procedures to analyze the crucial elements of the process and get insight into the reaction process. The paper concludes with a discussion of the difficulties that regularly arise in lignocellulosic biorefineries and several solutions to these problems. Thus, all the significant elements of the microwave-assisted and ultrasonic pretreatment methods of lignocellulosic biomass for large-scale commercial applications will be covered in one piece for the readers of this article.

Mechanism of microwave and ultrasound processes

Mechanism of microwave process

Microwaves are electromagnetic energy waves with frequencies between 0.3 and 300 GHz and wavelengths between 1 and 1 mm (Chaturvedi 2018). Microwaves are converted into appropriate frequencies during transmission so that the heated item may absorb the energy. Dipoles in polar liquids undergo continual realignment and alignment due to changes in electrical fields (Meda et al. 2017). Internal friction is caused by the ongoing fluctuations in dipoles and the transport of ions. The substance is then heated internally due to friction in the form of heat distributed throughout the biomass (Kostas et al. 2017). Instead of producing surface-level heat like in traditional heating, the conversion of energy in microwave irradiation creates volumetric heat inside the target material as shown in Fig. 1 (Foong et al. 2020). Solid-state processes, chemical catalysis, and organic synthesis have all used microwave irradiation models. Models for microwave irradiation usage improve biomass metabolism (Govarthanan et al. 2022). Thus, several studies have explored suitable microwave pretreatment operational parameters to improve the conditions for the pretreatment of biomass with enhanced efficacy. Energy savings have been mentioned frequently by experts as a probable advantage of microwave irradiation (Wei et al. 2019). The walls of heating containers are directly penetrated by microwaves, which efficiently heats the material. At the atomic level, the electromagnetic energy is directly converted into heat thereby producing microwave-induced thermal energy, which evenly dissipates energy throughout the material (Ren et al. 2022). Materials exposed to microwave irradiation have significantly larger temperatures than ambient temperatures (Falciglia et al. 2018). The three types of substances that are employed for microwave heating are conductors, insulators, and absorbers. For instance, transparent sources utilized for microwave heating, such as ceramics and glass, are referred to as insulators; extremely conductive sources, such as

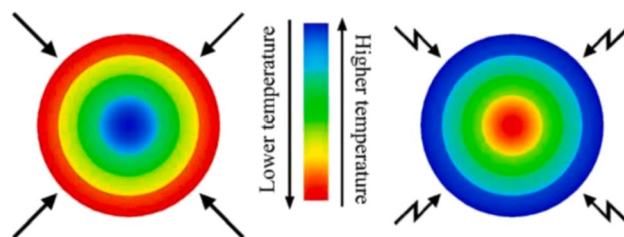


Fig. 1 The system of temperature distribution that is different between traditional and microwave heating (Reproduced permission from Hoang et al. (2021))

metals, are regarded as conductors; and components that can captivate and transform microwave energy into heat are regarded as absorbers (Hassan et al. 2018). The bulk of biomasses are often thought of as low-loss resources, and adding substances like activated carbons, graphite, pyrites, and charcoal can hasten the heating process. The dielectric properties of several biomasses used in the agro- and wood-processing industries were investigated, and low-loss dielectric materials were identified (Salema et al. 2017). Additionally, investigators claimed microwaves effectively break down cell walls and lignin, promoting enzymatic hydrolysis (Isaac et al. 2018). When microwaves align dipoles with the oscillating electric field, they produce thermal stresses that disrupt hydrogen bonds. Cell walls may be destroyed, and cellulose chains may be fractionated due to the breakdown of hydrogen bonds. As an outcome, the crystalline structure of the cellulose is disturbed, thereby facilitating hydrolysis (Halder and Purkait 2021). Direct biomass heating without utilizing ablative surfaces and hot gases is frequently made possible by selective heating with microwaves in a cool environment as shown in Fig. 2. Additionally, the volumetric heating provided by the microwave approach allows for the circumvention of conventional heat transfer constraints, resulting in significantly higher rates of heating, smaller sizes of processing devices, and greater processing capacity for big particle sizes (Kostas et al. 2017). As there are fewer needs for feedstock materials, apparatus sizes, and hot gas managing and restoration systems, the overall processing system can benefit from volumetric heating (Purkait et al. 2020). Microwave heating has a significant deal of possibility for lowering the running expenses of processing systems and boosting output capacity, despite having energy requirements equivalent to those of traditional processes

(Waudby and Zein 2021). Traditional techniques cannot provide these benefits. There are multiple advantages of microwave heating over conventional heating due to advancements in microwave irradiation heating methods.

Mechanism of ultrasonication process

As mechanical waves with a range of frequencies, ultrasound is primarily understood to convert energy into a reaction medium through acoustic cavitation (Sarker et al. 2021). The acoustic phenomenon is the occurrence of microbubbles developing and deflating in a liquid media due to an ultrasonic field (Mondal et al. 2021). The solvent vapor, which likewise changes over time as the acoustic energy moves over the liquid medium, produces several microbubbles. The microbubbles are shown to enlarge to a maximum diameter of 400 μm (Mankar et al. 2021). The dynamics of acoustic oscillations affect the microbubbles' fundamental characteristics. The microbubbles are shown to periodically expand and contract under the effect of low acoustic energy (Wu et al. 2021). On the other hand, when high-intensity acoustic oscillation is present, microbubbles become unstable. Additionally, the collapsing of microbubbles results in the formation of microjets, which propagate quickly toward solid surfaces at high pressure and temperature (Hasaounia et al. 2022). Furthermore, the abrupt collapse of bubbles results in shockwaves that create pressure of about 103 MPa (Reuter and Mettin 2016). Compared to conventional methods, the circulation of fluid around cavitation bubbles (micro-convection) is superior for transporting liquids and solids because it produces stronger shear forces as shown in Fig. 3 (Hoo et al. 2022). Notably, the localized hotspots are created by the bubbles collapsing suddenly, releasing a pressure of about 50 MPa and a temperature of about 5000 $^{\circ}\text{C}$

Fig. 2 The mechanism of microwave heating involved in the pretreatment of lignocellulosic biomass (Reproduced permission from Aguilar-Reynosa et al. (2017))

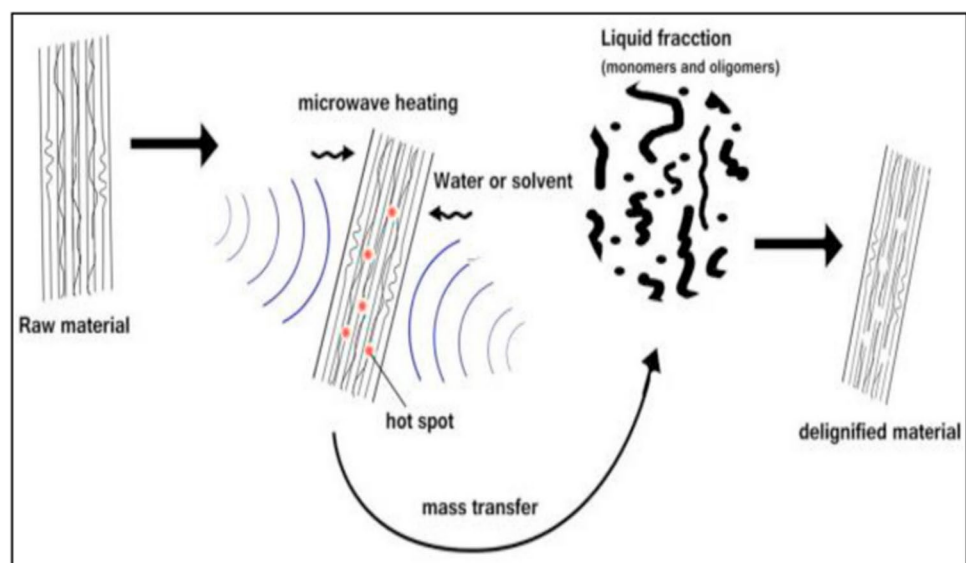
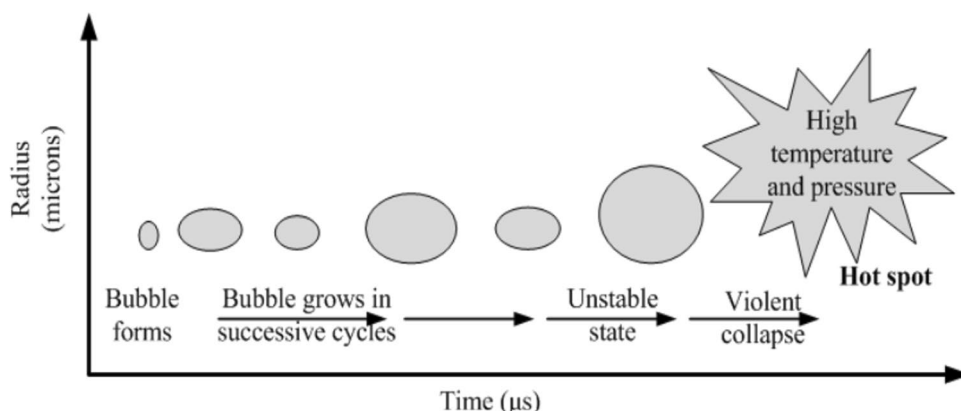


Fig. 3 The mechanism of bubble growth and collapse during ultrasonication (Reproduced permission from Bundhoo and Mohee (2018))



(Kerboua et al. 2022). As a result, the crystalline structure of solid particles was destroyed by extremely high pressure and temperature. Additionally, the generation of highly reactive H^{\cdot} and OH^{\cdot} derived from the disintegration of H_2O molecules is one of the critical elements of the ultrasound-based cavitation effect. By severing the glycosidic linkages, these radicals accelerate the breakdown of lignocellulosic materials (Wang et al. 2018). When subjected to the impact of ultrasound technology under optimal operating conditions, the whole phenomenon promotes or enhances the biochemical processes of lignocellulosic biomass (Balasundaram et al. 2022). The recalcitrant cellulose of the biomass now displays considerable hydrophobicity in the reaction media due to the abundance of intramolecular and intermolecular hydrogen bonds (Calcio Gaudino et al. 2021; Haldar and Purkait 2021). The resistant character of cellulose necessitates deconstruction with significant activation energy to reduce crystallinity. Additionally, adequate mass transfer is required for pretreatment when dissolving solid biomass in a liquid medium. To comply with such demands, pretreatment must therefore be performed in a system with a relatively large energy input while maintaining mild reaction conditions, including a short residence period. As a result, ultrasonic pretreatment meets all of these requirements while having a minimal impact on the reaction system.

Effects of microwave radiation on the pretreatment of biomass

Pretreatment by microwaves breaks down lignocellulosic materials and keeps track of hemicellulose, cellulose, and lignin amounts. Monitoring is done to break down polymers, dissolve the crystalline orientation of cellulose molecules, and solubilize other organic substances such as lipids, carbohydrates, and proteins (Mohan et al. 2018). The impact of microwave irradiation on hydrolysis before biofuel conversion was examined in several follow-up experiments.

Effects of microwave radiation on surface area and particle size

Through raising temperature and pressure, microwave-based pretreatment of biomass triggers the breakdown of cell walls. The structure of the lignocellulosic biomass is fragmented, resulting in smaller particles and more outer surface area (Mohan et al. 2018). After 20 min of 800 W microwave pretreatment, the surface area of MCC (microcrystalline cellulose) increased by 56% (Peng et al. 2014). By using NaOH-assisted microwave irradiation, the surface area was enhanced by an additional 75%. The size of the particle was reduced, and the surface area rose together with higher temperatures and longer microwave pretreatment times in sugarcane bagasse. The structure of the bagasse deteriorated following microwave treatments at different solution temperatures, as seen by the emergence of many fissures on its surface. At 160 °C, the lignocellulosic structure was more significantly disrupted (Hoang et al. 2021). The increased accessibility of cellulose molecules enhances biofuel production after hydrolysis, which tends to rise with surface area.

Effects of microwave radiation on lignin, hemicellulose, and cellulose content

Numerous researchers have discussed how the thermal properties of microwave radiation in numerous resources have caused the lignocellulosic structure to break, leading to delignification (Roy et al. 2020). Other investigations showed post-pretreatment hemicellulose removal from wheat straw and cornstalk. The lignin removal in the biomass was studied concerning the temperature and time of the microwave radiation during the pretreatment of biomass (Louis and Venkatachalam 2020). It was discovered that the delignifying capacity was enhanced with longer pretreatment times and higher temperatures. Microwave-assisted acid pretreatment techniques are more effective than alkaline pretreatment techniques at removing hemicellulose, whereas microwave-assisted alkaline pretreatment techniques extract lignin (Karungi et al. 2020).

Effects of microwave radiation on the crystalline structure and depolymerization of cellulose

Pretreatment of biomass attempts to decrease the crystallinity and polymerization of cellulose (Preethi et al. 2022). By severing hydrogen bonds, the heating properties of microwaves can aid in the depolymerization of cellulose and can further cause the crystalline structure of the cellulose molecules to disintegrate (Zhu et al. 2016). Subsequently, the hemicellulose molecule interacts with the microwave electromagnetic fields. It ensures a high reactivity with the OH groups proximal to the glycosidic linkages of the cellulose, following which the main hydroxyl groups of hemicellulose are most affected during microwave treatment (Bundhoo 2018). The development of “hot spots” and microplasma during microwave treatment, which affects the lignocellulosic biomass, has also been linked to these changes. Furthermore, it is well known that microwave radiation encourages the degradation of the amorphous regions (lignin and hemicellulose) of the biomass, thereby enhancing molecular mobility and improving the ability of amorphous zones to interact with microwave radiation (Romero-Zúñiga et al. 2022). As a result, these interactions influence the structural reorganization of the amorphous molecules, thereby altering the crystalline structure of the biomass. When the microwave power was amplified to 50 W during the processing of cellulose in ionic solutions, it was noticed that polymerization decreased. Researchers also discovered that microwave pretreatment reduced the crystallinity index (CI) by 9.2% (Bundhoo 2018). An intricate parameter for analyzing pretreatment productivity is the CI. Increases in the crystallinity index signify the breakdown of lignin and elevated availability of cellulose, further representing the effectiveness of the pretreatment (Haldar et al. 2022). Decreases in the crystallinity index indicate the disruption of the crystalline arrangement of cellulose and show the effectiveness of the pretreatment (Hoang et al. 2021). Following the microwave-supported alkali-assisted pretreatment of the cassava rhizome maize, the CI rose from 30.92 to 42.72% (Sombatpraiwan et al. 2019). The impact of microwave radiation on the biomass, which is more evident in the amorphous regions than in the crystalline region of cellulose, was found to promote enhanced crystallinity and reduced hemicellulose and lignin content (Sorn et al. 2019). There was a claim that hydrolysis could not only be restricted by crystallinity and was discovered to have a less substantial impact on hydrolysis than the polymerization level (Karungi et al. 2020).

Effects of microwave radiation on solubilization of organic matter

The thermal effects of microwave irradiation disrupt cell membranes and transfer organic particles to the dissoluble phase (Kang et al. 2020). Sludge was the main focus of most investigations on microwave irradiation pretreatment for

improved solubilization of organic material (Bozkurt and Apul 2020). Kitchen garbage irradiated using a microwave led to a 40% increase in solubilization (Arpia et al. 2021). Similarly, after microwave pretreatment, the solubilization of waste and the organic component of municipal solid waste improved (Ahmed et al. 2021).

Effects of microwave radiation on hydrolysis and the production of reducing sugar

As previously stated, MW pre-treatment helps to break down the lignocellulosic matrix and increases the amount of cellulose that is available for hydrolysis. Following MW-assisted acid pre-treatment of water hyacinth in 1% H₂SO₄ solution at 140 °C, researchers obtained a reducing sugar yield of 26.7 g/100 g volatile solids (VS), and this yield grew to 96% of the theoretical value after 72 h of enzymatic hydrolysis (Cheng et al. 2013). Cheng et al. found reducing sugar production of 29.6 g/100 g cassava residues (60% of theoretical yield) with MW-assisted acid pre-treatment, and this rose to 79% of the theoretical reducing sugar yield after enzymatic hydrolysis. Similarly to this, Liu and Cheng discovered that pre-treated corn stover at 700 W for 90 min in 0.2 N H₂SO₄ increased the content of soluble sugar from 5.2 (control) to 12.3 g/L (Bundhoo 2018). Lin et al. also discovered that raising the MW temperature from 150 to 190 °C caused an increase in the production of reducing sugars from 0.022 to 0.028 g/g VS while raising the MW temperature further to 230 °C caused a reduction in the production of reducing sugars. Similarly, previous investigations have found that increasing MW pre-treatment temperatures increased reducing sugar yields, which then decreased over a particular threshold. The main cause of the decrease in the reducing sugar yields was sugar degradation at high pretreatment temperatures (Purkait and Haldar 2021).

Effects of microwave power

Microwave-aided pretreatment is claimed to yield massive power densities compared to the traditional heating pretreatment approach, thus increasing the production pace and lowering costs (Devi et al. 2021). It was contended that microwave energy enhanced the glucose output from the alkali-assisted pretreatment of wheat straw by 69.7% (Alawad and Ibrahim 2022). However, sugar yield was slightly improved when sugarcane bagasse was pretreated using microwave and acid techniques (Ajala et al. 2021). It was established that during microwave-assisted acid pretreatment, the reduction in sugar yield and the increase in microwave power are inversely associated (Tsegaye et al. 2019). At varied microwave powers, the amounts of decreasing sugar yields fluctuate differently. Additionally, preparation time gets shorter as microwave power rises. The breakdown of lengthy chains of cellulose into short molecular groups

lowers the degree of cellulose polymerization (Ling et al. 2019). According to reports, 180 W is the ideal microwave power since it provides enough time for the pretreatment of the biomass while minimizing volumetric losses in the soluble phase (Chen et al. 2021). Comparative investigations in a glass reactor and an in-house manufactured jacketed glass reactor built to avert heating of the sample examined the nonthermal and thermal properties of microwaves. Under test situations, only thermal microwave effects were seen. To boost the bio-solubilization of the biomass components, microwave pretreatment with improved pressure and power density was tested. The solubilization of 33% hemicellulose content significantly impacted the cell walls. The outcomes open the door for commercial uses (Bichot et al. 2018).

Effect of microwave radiation time

The primary variable that impacts the pretreatment process is microwave heating or response time (Shangdiar et al. 2022). Through the improvement of hemicellulose removal and cellulose digestibility, the synergetic effect between reaction time and microwave power significantly impacts biomass digestibility (Abraham et al. 2020). However, speeding up the microwave's reaction time and power can cause the materials to heat up too much, which will cause the released sugar to decompose. Extrusion-microwave and extrusion pretreatment procedures were used in experimental evaluation on switchgrass and big bluestem pretreatment (Gallego-García et al. 2023). The extrusion-microwave technique enhanced sugar retrieval from switchgrass and big bluestem by 15.2% and 14.2%, respectively, compared to the extrusion pretreatment method. However, the sugar recovery was only slightly impacted by extending the exposure to microwaves by 10 min (Guiao et al. 2022). It was also discovered that a temperature rise could lead to a drop in monomeric sugar yields. Remarkably, the temperature of the reaction was found to be the utmost perilous factor for the liberation of monomeric sugars (Martins et al. 2022). The process of sugar dehydration was also impacted by higher pressure and longer exposure times, which led to lower quantities of galactose/xylose and arabinose as well as higher amounts of levulinic acid, HMF, and furfural. The process of sugar dehydration was also impacted by higher pressure and longer exposure times to the microwaves, which led to lower quantities of galactose/xylose and arabinose as well as higher amounts of levulinic acid, HMF, and furfural. Ma et al. further emphasized how crucial it is to optimise the settings for microwave pretreatment of rice straw in order to achieve a high level of cellulose hydrolysis efficiency. Without optimization, such as when the temperature is too high, simple sugars are converted into byproducts that prevent alcoholic fermentation, such as HMF and furfural, which reduce their quantity.

Effect of microwaves in combination with various reagents

The use of microwave pretreatment has a lot of potential in the field of lignocellulosic biorefinery because the heat produced by the procedure raises the temperature throughout the biomass rather than just on its surface, demonstrating greater energy efficiency than traditional heating methods. The oscillating motion of the dipoles helps to break up the hydrogen bonds particularly placed within the carbohydrate polymers found in the biomass in addition to producing the heating effect. When microwave pretreatment is used in conjunction with the other integrated approaches, the breakdown of lignocellulosic biomass and its conversion into useful products becomes more significant. Three distinct kinds of biomasses, namely maize stover, miscanthus, and switch grass, were subjected to ultrafast microwave-aided pretreatment by Chen et al. utilizing choline chloride: lactic acid as DES (Chen and Wan 2018). For all of the samples that were processed for 45 s at 800 W of microwave radiation, cellulose recovery of 65–67% was seen. When DES is present in the reaction system, significant lignin removal occurs, increasing the recovery of cellulose during the microwave process. In addition to DES, adding acid or alkali to the reaction mixture considerably enhances biomass hydrolysis under the impact of microwave pretreatment. However, the degree of the effect greatly varies from one other type of pretreatment due to the compositional diversity of LCB. Table 2 illustrates the various process requirements for an efficient microwave heating application on various lignocellulosic biomass. The table also demonstrates the impact of microwave pretreatment on a variety of lignocellulosic biomass when combined with other techniques.

Effects of ultrasonic irradiation on the pretreatment process of biomass

It has been used as a stand-alone pretreatment method and in combination with other techniques. For pre-treatment of feedstocks, ultrasonic irradiation is commonly integrated with a chemical such as an alkali, ozone, acid, and ionic liquid, or with a physical approach such as heat, microwave irradiation, and supercritical carbon dioxide. The chemical and physical properties of the biomass, such as surface area and particle size, hemicellulose, cellulose, and lignin content, the extent of polymerization and crystallinity of cellulose, and solubilization of organic matter, among several others, are affected by ultrasonication. The various effects of ultrasonic irradiation both alone and in combination with other pre-treatment technologies on the lignocellulosic biomass are tabulated in Table 3.

Table 2 Microwave irradiation in combination with various other reagents

Biomass	Microwave heating parameters	Major outcome	Reference
Oil palm empty fruit bunch	MHP was conducted at 190 °C for 3 min in the presence of 1.1% (v/v) oxalic acid	34.6% of reducing sugars was obtained after enzymatic hydrolysis	(Anita et al. 2020)
Rye and wheat stillage	Microwave irradiation was done at 300 W, 54 psi for 15 min in the presence of 0.2 M H ₂ SO ₄	A cellulose hydrolysis yield of 75% was obtained	(Mikulski and Kłosowski 2020)
Rice straw	Microwave-assisted acidic salt pretreatment using FeCl ₃ (0.15–0.35 M) and H ₃ PO ₄ (2–5%) was investigated at 700 W, 155 °C for 10 min	~58% of sugar yield was obtained	(Kumar et al. 2019)
Corn cob	11.6% Na ₃ PO ₄ ·12H ₂ O was used in a microwave-assisted alkaline salt pretreatment process for 6 min at 700 W	Sugar yield increased sevenfold, with the highest reducing sugar yield being 0.8 g/g	(Sewsynker-Sukai and Gueguim Kana 2018)
Sugarcane bagasse	In the presence of the ionic liquid ChOAc, microwave pretreatment was carried out at 300 W for 10 min	30% of glucose yield was observed	(Sakdaronnarong et al. 2017)
Sugarcane leaf	Microwave-assisted salt alkali pretreatment with 1.7 M ZnCl ₂ and 1.5 M NaOH was investigated at 400 W for 10 min	73% lignin was removed and 1.2 g/g of reducing sugars were produced	(Moodley and Gueguim Kana 2017)

Effects of ultrasonic irradiation on surface area and particle size

Through oxidative stress, heat, and shear during cavitation, ultrasonication has reportedly been shown to support cell disruption and disintegration of cellulose (Poddar et al. 2022). As a result, particle size is reduced and the surface area is raised. Smaller sizes of particles and greater surface areas were produced in maize straw, and wheat straw as a result of the cell structures in these materials being broken down by longer sonication periods (Zhou et al. 2021). Similarly, the breakdown of the lignocellulosic matrix caused the particle size of cassava chip slurry to fall by 40 times after ultrasonic irradiation at 8 W/mL, whereas it was found that the particle size of sonicated corn slurry declined by 20 times compared to the control (Wu et al. 2022).

Effects of ultrasonic irradiation on hemicellulose, cellulose, and lignin content

During ultrasound pre-treatment, the delignification of lignocellulosic biomass is believed to have occurred due to the intense temperature and pressure during cavitation in addition to the oxidative stress caused by the radicals that were produced during ultrasonic irradiation (Ong and Wu 2020). As hemicellulose is amorphous rather than having the crystalline phase of cellulose, which makes them more difficult to degrade due to hydrogen bonding in the

microfibrils of cellulose, it can be removed or dissolved during pre-treatment (Bundhoo and Mohee 2018). As a consequence, after pre-treatment, there is a strong feasibility for a drop in hemicellulose and lignin content, which causes a subsequent surge in the cellulose content (Ravindran and Jaiswal 2016). During ultrasound-aided alkali or ozone pre-treatment of SB (sugarcane bagasse), it was found that the contents of lignin and hemicellulose had decreased, while the contents of cellulose had increased (Utekar et al. 2021). Likewise, after ultrasonic-aided alkaline pre-treatment of the feedstock, it was observed that 68% of lignin from bonborigori, 65% of lignin from areca nut husk, and 64% of lignin from moj had been removed (Irani et al. 2021). Similarly, due to ultrasound-aided alkaline pre-treatment, the delignification efficacy of 70.4% from groundnut shells, 80.0% from coconut coir, and 78.9% from pistachio shells were reported (Subhedar et al. 2018). Following ultrasonic-assisted acid pre-treatment, Mohapatra et al. observed delignifying effectiveness of 82.1% and 80.4% for Hybrid Napier grass and Denanath grass, respectively. Additionally, the authors noticed that increasing sonication power increased delignification efficiency (Mohapatra et al. 2017).

Effects of ultrasonic irradiation on the crystalline structure of cellulose

Biomass contains both amorphous and crystalline forms of cellulose. The majority of cellulose is attributed to its

Table 3 Pretreatment techniques based on ultrasonication on various lignocellulosic biomass

Biomass	Ultrasonication parameters	Major outcome	References
Soybean oil	200 W, frequency of 20 and 28 kHz, starting temperature of 25 °C, catalyst concentration of 1.8%	production of 96% (wt%) biodiesel	(Yin et al. 2017)
Date seed oil	24 kHz, 300 W, for 5 min at 60 °C, in the presence of 1% (wt%) of NaOH	Production of 96.4% (wt%) of biodiesel	(Allami et al. 2020)
Wheat straw	20 kHz, 650 W, for 30 min at 50 °C in the presence of NH ₄ OH	~92% of saccharification was obtained	(Zhong et al. 2017)
Oil palm empty fruit bunch	210 W, for 15 min at 50 °C in the presence of eutectic solvent ChCl: LA (choline chloride: lactic acid)	A maximum yield of 36.7% reducing sugars was obtained	(Lee et al. 2021b)
Mulberry powder	NaOH + 2% (v/v) alkaline peroxide solution treated biomass was sonicated for 10 min at 300 W	Xylan conversion of 64.2%, and glucan conversion of 87.3%, were obtained	(Baksi et al. 2019)
Rice Straw	1% w/v NaOH + 300 W, 22 kHz for 1 h at 50 °C	Degradation of more than 57% of lignin and 46% of hemicellulose had been achieved	(Wu et al. 2017)
Watermelon rinds	180 W, 40 kHz, for 40 min, at 120 °C in the presence of eutectic solvent ChCl: LA (choline chloride: lactic acid)	83.03% of total reducing sugars and the highest concentration of 60.17% glucose were obtained	(Fakayode et al. 2021)
Oil palm fronds	80% amplitude for 3 min in the presence of aqueous ChCl-oxalic acid (16.4 wt% H ₂ O)	A furfural yield of 56.5% was obtained	(Lee et al. 2021a)
Oil palm fronds	60% amplitude for 30 min in the presence of 2.5% NaOH-aqueous DES	47% of lignin was removed and a glucose yield of 90.12% was obtained	(Ong et al. 2021)

crystalline structure, whereas a small amount is comprised of amorphous cellulose, which is made up of disorganized cellulosic strands (Seddiqi et al. 2021). The hydrolysis efficiencies of moderately refined cellulose substrates have been believed to be significantly influenced by the crystalline percentage of cellulose. The crystallinity index relies on its evaluation of crystallinity on the proportion of crystalline regions in a structure (De et al. 2021; Haldar and Purkait 2020). In cellulose-based biomasses, high-power ultrasonication reduces the crystallinity and increases their pore size. According to an investigation by Wang et al. when there was an increase in the sonication power from 0 to 200 W, the degree of crystallinity of microcrystalline cellulose (MCC) marginally dropped by 1%. The degree of crystallinity of MCC, however, significantly decreased as the power of ultrasonication was raised from 200 to 700 W. It is observed that despite the degree of crystallinity dropping to 54.6% after 15 min of ultrasonic irradiation at 700 W, the size of the crystal did not alter significantly (Karimi et al. 2014). In a similar investigation, it was discovered that after receiving ultrasonic irradiation of 300 W for 5 min, neither the degree nor the size of crystalline cellulose changed significantly (Hemmati et al. 2019). The amount of time cellulosic materials are exposed to low-power ultrasonic irradiation appears to not affect the crystalline nature of the cellulose (Szymańska-Chargot et al. 2018). In general, there is no linear relationship between treatment time and crystallinity while multiple powers of ultrasonication were used. Though ultrasonic power is an important element in the ultrasonication process, the

crystalline nature of cellulose does not linearly decline with an increase in the ultrasonication power (Gielen et al. 2017). This assertion is confirmed by the pertinent sonochemical theory, which states that the intensity of cavitation reaches the highest value at the right power of ultrasonic waves (Wen et al. 2018). The intermolecular hydrogen bonds of lignocellulosic biomass are successfully destroyed by ultrasound-assisted alkali pretreatment, which lowers the crystallinity of cellulose even though the reduction of cellulose crystallinity is possible only with high-power ultrasonication (Sasmal and Mohanty 2018). Under the ultrasound-assisted alkali pretreatment process, crystallinity reduction is achievable even with 80 W of power (Ji et al. 2021). According to an investigation conducted the total crystallinity indices of the cellulose content in sweet sorghum bagasse, as determined by the absorbance ratio, was found to be 0.83 for control samples and 0.70 for samples that had undergone ultrasound-assisted NaOH treatment (Karimi et al. 2014). Ultrasound-assisted alkali pretreatment is the cause of the reduced crystallinity and increased amorphous percentage of cellulose. Due to the weak inter- and intramolecular hydrogen bonds established by the alkali pretreatment, ultrasonic radiations efficiently disrupt the lignocellulosic biomasses. The strength of hydrogen bonds affects the crystallinity of cellulose (Sharma et al. 2022). As a result, the fact that ultrasonicated cellulose has a reduced degree of crystallinity than untreated cellulose indicates that ultrasonic waves have disrupted the hydrogen bonds in the cellulose. This assertion is supported by the finding that ultrasound-induced cavitation

can separate molecules in amorphous and crystalline zones, causing the breakdown of cellulose molecules' orderly packing and a reduction in crystallinity (Lee et al. 2021b). It was observed that the degree of crystallinity decreased significantly when the ultrasonic power was raised to 700 W from 200 W, as the hydrogen bonds were completely disrupted (Li et al. 2019). Likewise, it was observed that the switchgrass samples ultrasonicated at 40 kHz, disrupt the clumps of substance that were initially visible into thin and long strips of cellulose. Thus, it was concluded that the transformation of switchgrass from a highly crystalline structure into a lengthy band of material is substantial and that ultrasonication could modify the structural makeup of switchgrass (Cudjoe et al. 2017).

Effects of ultrasonic irradiation on the degree of polymerization of cellulose

The size and weight of cellulose vary according to their degree of polymerization (DP), hence larger weights and sizes of cellulose molecules are related to a higher degree of polymerization (De Silva and Byrne 2017). It is hypothesized that the length of the glucan chain contributes significantly to the hydrolysis of cellulose (Reiniati et al. 2017). The process of depolymerizing cellulose is influenced by the composition of the lignocellulosic biomass (Zhang et al. 2016). Irrespective of the substrate, it implies that the cellulose DP has “leveled off,” which is connected with the remaining crystalline cellulose's enhanced recalcitrance (Shi et al. 2017). DP is often assessed using size exclusion chromatography and viscosity. Lower DP can be produced by cavitation collapse, which damages the polymeric structure of cellulose (Madison et al. 2017). During heterogeneous cellulose hydrolysis reactions, a decrease in the polymerization degree is expected. In contrast to low-intensity ultrasonication, an increased level of ultrasonication pretreatment can remarkably accelerate the deterioration of cellulose (Wang et al. 2018). As the intensity of the ultrasonic waves rises over the same time, the DP of pretreated cellulose has a tendency to decrease and its dispersion becomes more restricted (Dilamian and Noroozi 2019). However, it was found that the DP of cellulose could only be moderately altered by ultrasonic irradiation individually (Csizsar et al. 2021). Despite the utilization of a high-power ultrasonication of 200–500 W for 5–20 min, the researchers felt that the power of the ultrasonication procedure was insufficient to disrupt the chemical interactions between cellulose molecules (Koshani and Jafari 2019). It is believed that the energy needed to disrupt the hydrogen and chemical bonds in cellulose molecules are 5–30 kJ/mol and 400–1000 kJ/mol respectively (Kumar et al. 2020). In the investigation by Wang et al. the maximum output energy of the ultrasound device was tuned to 600 kJ, which was able to break only the

hydrogen bonds in the cellulose molecules (Hoo et al. 2022). Although cellulose was exposed to ultrasound irradiation after being treated with an alkali or an acid, it appears that the effects of ultrasound on lowering DP are much less than those of alkali or acid pretreatment (SriBala et al. 2016). In a study by Mason and Lorimer, the shear stresses and the hydrodynamic forces produced by the collapsing of bubbles, which is the result of ultrasonic irradiation of polymeric solutions, were investigated (Qiu et al. 2019). A maximum of 30 min of ultrasonic radiation was applied to the test samples, and the extended sonication duration was associated with a further decrease in the molecular weight of the cellulose (Sarwono et al. 2017).

Effects of ultrasonic irradiation on the hydrolysis and reducing sugar production

Ultrasonication aids in cell disruption and the destruction of cellulose molecules, as was already mentioned. Enzymatic hydrolysis and the production of sugars are consequently improved by the increased surface area that is available for enzymatic and microbial attack (Singhanian et al. 2022a; Venturin et al. 2019). Numerous studies have documented improved hydrolysis after sonicating invasive weeds (Borah et al. 2016), poplar wood biomass, sugar beet shreds (Ivetić et al. 2017), and cotton cellulose (Szabo and Csizsar 2017). Increased yields of reducing sugars also have been noted in the literature as a result of improved hydrolysis. In comparison to the untreated sample, it was found that the sonicated cassava chip slurry produced 180% more reducing sugars than the control (Bundhoo and Mohee 2018). Similarly, it was also documented that sonicating the de-oiled *Jatropha* waste, demonstrated a rise in reducing sugar yield by 14% (Mudhoo et al. 2018). After acid hydrolysis of sugarcane bagasse with ultrasonic assistance, Xi et al. observed a 29.5% increase in the yield of sugar (Halder and Purkait 2021). According to Eblaghi et al. sonicated bagasse at 35 kHz, and 65 C, for 5 min increased sugar output to 5.78 g/L from 3.62 g/L for unprocessed sugarcane bagasse (Eblaghi et al. 2016). Similarly, it was documented that the xylose production from sonicated sample increased to 58% from 22% for the unprocessed palm oil empty fruit bunch (Saeed et al. 2021). Likewise, additional research has found that ultrasonicated biomasses, such as sugarcane bagasse and tops, cassava waste, rice straw, bamboo, ultradispersed wood particles, and cotton cellulose had higher yields of reducing sugars (Bundhoo and Mohee 2018). However, on the contrary, it was reported that ultrasonication did not substantially improve the saccharification of soybean fiber in comparison to the control (Subhedar and Gogate 2016). Similar results were obtained by Moscon et al. on comparing ultrasonic and conventional pretreatments of rice hulls for enzymatic hydrolysis (Moscon et al. 2014).

Combined effects of microwave radiation and ultrasonication on the biomass

Both ultrasonic waves and microwaves are forms of energy that can be utilized to process biomass to increase the availability of cellulose, oligosaccharides, and hemicellulose, and reduce its size, thereby making it easier to process the biomass further for the production of the desired chemicals (Hassan et al. 2018). The biodegradability and hydrolysis of agricultural residues such as olive pomace and grape pomace utilized for the production of biogas were found to be accelerated by microwave and ultrasonication pretreatments. Additionally, it can be concluded that using ultrasonication rather than just microwaves for pretreatment was more successful (Aylin Alagöz et al. 2018). The efficacy of combining microwaves and ultrasonic waves for the processing of biomasses was taken into account in several patents (Vinatoru et al. 2017). The selective degradation of waxes and lignin by such a hybrid method was discovered, and microwave radiations were documented to eliminate the waxlike covering from the surfaces of the biomasses to rise the surface area accessible to enzymatic action (Anu et al. 2020). In the process of hemicellulose degeneration, it was discovered that the use of both microwave and ultrasonication energy offered an additional way to heat up the biomass internally, hastening the hemicellulose hydrolysis (Modupalli et al. 2022). Corncoobs pretreated hydrothermally utilizing ultrasonic waves of 20 and 60 kHz frequency for 10 and 20 min, respectively, and microwave irradiation of 400 and 600 w for 100 and 130 min, respectively produced the maximum yield of xylose from the maize hydrolyzate core (Hoang et al. 2021).

Future perspectives

The current condition of the global consumption of energy is undoubtedly of great interest, as the dire circumstances show a persistent reliance on nonrenewable natural resources and point to the unquestionable necessity for a shift towards a more sustainable strategy. The sustainability and depletion of fossil resources, particularly crude oil, are in peril. To support both intergenerational and intragenerational equity for energy security, a policy shift in favour of biomass use as a renewable energy source would be highly welcomed. Lignocellulosic biomass, which has an annual output of 170–200 billion tonnes, may be the fundamental cause of this problem. Despite the amazing advantages of microwave irradiation and ultrasonication, the advancement of commercial microwave heating strategies and the incorporation of microwaves and ultrasonication into the physico-chemical approach for feedstock preparation is relatively inadequate.

The research in biorefineries is hopping on the bandwagon to identify the more acceptable and appropriate chemical solvents in combination with microwave and ultrasonication pretreatment processes to minimize potential environmental consequences from processing, in line with the worldwide green environment movement. Numerous solvents that claim to be “green” present an intriguing opportunity for study and education, where often increased levels of biodegradability, lower levels of toxicity, and reduced vapour emissions might be seen (New et al. 2022). In order to fully contribute to the information of the scientific community, the scrutiny of the nature of the electromagnetic interactiveness with lignocellulosic biomass has also rarely ever been stated. To acquire the highest amount of cellulose, operating parameters need also to be optimized. To increase environmental sustainability and overall effectiveness, operating parameter optimization should focus on lowering energy needs and chemical usage. Despite requiring a significant initial investment, microwave pretreatment has a high capital turnover ratio. When producing corrugated paperboard, the cost–benefit of using microwave-assisted drying vs the traditional steam platen process was evaluated. The initial cost of microwave pretreatment was \$7000, however, due to the power savings of this technology over the traditional steam platen process, this cost might be offset by \$128–380/h. The initial expenditure might be repaid in less than a year if the operation was based on the \$6000/h/year estimate. Importantly, the advantages of using a microwave over traditional cooking techniques were also assessed. These advantages included better quality, less waste, and less starch intake. Low maintenance costs in industrial-scale processes are thought to improve the process (Hoang et al. 2021). Although it has been shown that using microwave heating and ultrasonication to support physicochemical approaches has numerous advantages for incorporation into biomass pretreatment, several difficulties still need to be resolved and understood in detail. As a result, difficulties with the pretreatment of lignocellulosic biomass for the industrial and commercial mass manufacturing of biofuels persist and are centered on concerns with capital cost, energy efficiency, and sustainability (Singhania et al. 2021). These difficulties could involve problems with energy usage, a better understanding of the foundations of temperature gradient-induced biomass heating, and the dielectric characteristics of diverse biomasses. Significant research is required to determine the most effective ultrasonication and microwave-based technique for use in biorefineries to accomplish important approaches for the sustainable exploitation of accessible renewable resources, high efficiency, and clean energy. It is crucial to research balancing energy and the manufacture of products that are suitable for the commercial sector. It is necessary to thoroughly explore the transfer of biomass pretreatment

techniques from laboratories to industrial and commercial settings. Furthermore, when designing and creating cutting-edge procedures and strategies, energy integration needs to be carefully taken into account. For the synthesis of platform chemicals produced from biomass, such as levulinic acid (LA) and 5-hydroxymethyl furfural (5-HMF), the utilisation of functionalized ILs as a catalyst and solvent augmented with microwaves and ultrasonication in the right reaction pathways and ideal circumstances is important. (Grewal et al. 2022). It is undoubtedly essential for biorefineries to flourish on engaging and comprehensive techno-economic evaluations. As a result, if other commodities from co-products, such as lignin, oils, and proteins are achieved, the economic balance of biorefineries can be enhanced and improved (Singhania et al. 2022c).

Conclusion

The cell wall of cellulosic feedstocks is damaged by both microwaves and ultrasonic irradiation, which enhances the exposure of cellulase enzymes to cellulose fibers. By removing lignin or modifying the recalcitrant biomass components, combining microwave with physical or chemical pretreatment methods increased the yield of fermentable sugars. Optimization of other operating pretreatment parameters, such as biomass loading, microwave intensity, and irradiation time, have an impact on the production of inhibitors, and reducing sugar yields is also crucial. An additional benefit of microwave pretreatment discovered was that the inhibitory chemicals produced during the pretreatment process were insufficient to have any inhibitory effects on the fermentation process. One drawback of the microwave pretreatment process is that the majority of biomass is often regarded as being low-loss materials, therefore it has to be supplemented by materials that provide quick heating, such as graphite, charcoal, activated carbons, and pyrites. It is interesting to note that all of the biomass from the agricultural and wood-based industries had low-loss dielectric characteristics. These materials do not absorb microwaves well during microwave-assisted pyrolysis, at which point the microwave absorption will be noticeably increased. Cellulosic biomass may have its crystallinity reduced and its pore capacity increased by high-power ultrasound. The surface shape of lignocellulose materials can be altered by ultrasound pretreatment, which will increase the amount of sugar produced upon hydrolysis. The intermolecular hydrogen bonds of lignocellulose are successfully destroyed by ultrasonic-assisted alkali pretreatment, which reduces the crystallinity of cellulose. One of the more efficient pretreatments for the enzymatic hydrolysis of bagasse is the combination of an alkali such as NaOH and ultrasonication, which yields more than 90% of the theoretical glucose yield in just 70 h. An essential advantage of exposing the surface of solid substrates to the action of

enzymes is provided by mechanical shock produced by the collapse of cavitation bubbles, such as shock waves and micro-jets. The efficiency of pretreatment is normally increased by ultrasound, however, when an organosolv pretreatment is used, the effects of ultrasonic irradiation are reduced. This shows that not all pretreatment or biomass valorization techniques require ultrasound as additional energy. Additionally, not every reactor design will result in the full optimization of the energy utilized during ultrasonication. To ensure a good distribution of cavitation energy, it is essential to optimize the process parameters and reactor design. Economic competitiveness, in addition to energy waste during ultrasonication, is a significant obstacle to the use of ultrasound in industrial production. To achieve the maximum release of glucose, it is necessary to optimize the ultrasonication sonication duration and power. However, even a brief period of ultrasonic irradiation has been acknowledged as a suitable pretreatment for disintegrating the crystalline structure of cellulose for a wide range of substrates and substantially raising the glucose yield.

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Declarations

Competing interests The authors declare no competing interests.

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