Maria das Graças de Almeida Felipe
Anuj Kumar Chandel *Editors*

Current Advances in Biotechnological Production of Xylitol

Fermentative Production of Xylitol

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Preface

Biotransformation of lignocellulosic biomass into biofuels and biochemicals offers numerous strategic and environmental benefits. One of the valuable compounds which can be made from lignocellulosic biomass is Xylitol. It is a commercially viable sugar alcohol that has several applications in the food, confectionaries, odontological, and pharmaceutical sectors. Based on the growing demand for xylitol, the US Department of Energy (USDOE) has included xylitol in the top 12 value-added chemicals produced from lignocellulosic feedstock [1]. Xylitol production from the hemicellulose hydrolysate is considered more profitable than ethanol or co-production of ethanol and furfural [2].

Xylitol is a white crystalline powder, soluble in water, and similar in taste to table sugar with a lower calorific content, and it has anticariogenicity and antimicrobial properties, among other properties. Besides the common use of xylitol as a low calorific sweetener, it can be considered as a platform chemical for the chemical synthesis of xylaric acid, ethylene glycol, glycerol, etc. [3]. Therefore, in recent years, the commercial demand for xylitol has increased significantly. Currently, xylitol has a market of US$823.6 million which is expected to rise approximately to US$ 1.37 billion by 2025 [4]. Currently, this increased demand worldwide is being met through the chemical conversion of hemicellulosic hydrolysate. However, chemical synthesis has several environmental and sustainable challenges like high chemical requirements, high carbon footprints, high water usage, and reactor corrosions. In order to replace the chemical transformations, biotechnological methods of xylitol production have the advantages of being eco-friendly and sustainable [5]. Xylitol production by microbial fermentation offers several unique advantages because it has a very low amount of inhibitors compared to the chemical synthesis. The fermentation broth after separation of biomass can be directly used for xylitol recovery and concentration using minimum steps of purification. The concentrated xylitol solution can also be directly used in the confectionaries or in toothpaste formulations avoiding the requirement of crystallization.

Recent developments made in lignocellulosic feedstock pretreatment for efficient xylose recovery, fermentation methods and economic media formulations, and strain improvement for enhanced xylose conversion and xylitol recovery have shown the potential of microbial xylitol production to carter its growing demands in the market. For this, the requirement of high-quality concentrated xylose sugar syrup without the inhibitors is very important. A considerable research progress has been made for the best-quality xylose sugar recovery as this is the primary feedstock for xylitol production followed by strain improvement, fermentation of xylose solution, and downstream processing of microbial xylitol production. The major barrier to the microbial xylitol production at the commercial scale is the requirement of more time in the fermentation step in turn reducing the xylitol productivity. This book is aimed to bring the recent research output in addition to some important advancements made in the xylose recovery, fermentative production of xylitol and strain improvement, etc. Besides the improvements made in xylose production, the book will also disseminate cutting-edge information on the demand for xylitol from various commercial sectors and its applications.

This book has compiled **11** specialized chapters written by experts from various universities/institutes/industries. Chapter 1 of **de Almeida et al**. presents the recent progress and future perspectives on hemicellulose destruction aiming to xylose recovery with desired yields with a minimum amount of plant cell wall-derived inhibitors. These inhibitors include weak acids, phenolics, furans, and others. These inhibitors influence the microorganism's growth and xylitol productivities. This is necessary to remove these inhibitors from the hydrolysate prior to the fermentation of hydrolysates in order to obtain the optimum productivity of xylitol. Chapter 2 by **Velmurugan et al.** provides a critical assessment of various detoxification strategies applied to the lignocellulose hydrolysates conditioning and to remove the inhibitors. Various new wild xylitol-producing microbial strains have been found in nature. These strains offer the important characteristics of growth and xylitol production profile. These strains display the different growth profile and substrate utilization rates eventually showing different xylitol production profiles. So, Chap. 3 by **Queiroz and colleagues** summarizes the important features of xylitol-producing microorganisms and growth characteristics employing various substrates. In order to achieve the desired product yield and productivity, continuous efforts are being made to develop the superior microorganisms by classical and new molecular biology techniques. In this sense, Chap. 4 by **Gupta et al.** appraises the various strain improvement methods for enhanced xylitol production. Various fermentation strategies have been assessed for xylitol production from a variety of lignocellulose hydrolysates. These techniques include batch, fed-batch, semi-continuous, and continuous cultivation. Recycling of microbial cells in free or immobilized form has also been attempted to make the xylitol production process robust and economic. Besides the development made in fermentation methods, optimization of media and fermentation parameters have been studied in detail. Optimization of fermentation media using economic carbon and nitrogen sources is essential to obtain the economic xylitol production. These insights are important for the xylitol production at a large scale. Therefore, Chap. 5 by **Prado et al.** provides a comprehensive review on a variety of fermentation methods and optimization of fermentation parameters and media ingredients. For the commercial viability of microbial xylitol production, this is necessary to adopt integrated approaches in sugarcane, wood, or corn processing mills in order to reduce the capital and operational expenses. This will save the biomass handling cost, steam and electricity savings and reduce the biomass handling and processing cost. Taking this into consideration, **Martinez et al.,** in Chap. 6, elaborate on the critical elements for xylitol production in integrated sugarcane and corn processing mills. After the fermentation of hemicellulosic hydrolysate, downstream processing for xylitol recovery is done by precipitation, purification, and crystallization steps. Chapter 7 by **Durán-Aranguren and co-authors** is aimed to present a critical analysis on downstream processing methodologies for xylitol recovery. Techno-economics analysis of any bio-based process is utmost important to have the key insights into the fermentative production of xylitol. This will enable the economic viability of the xylitol production process considering various process variables in different case scenarios. These insights are valuable for xylitol production at a commercial scale. Therefore, Chap. 8 by **Piedrahita-Rodríguez and Alzate** provides a detailed information on techno-economics analysis of xylitol production covering important scenarios for xylitol production in standalone and integrated biorefineries. Even though a significant progress has been seen toward the fermentative production of xylitol in research institutes, there is still a big gap that exists for xylitol production at a commercial scale via fermentation routes. Chemical synthesis of xylitol is the a primary source of growing xylitol demand in society. There are several reasons which hinder the large-scale production of xylitol by microbial routes. These issues and their possible solution have been highlighted in Chap. 9 by **Rao and colleagues.** In fact, this chapter presents a closer perspective on the commercial production of xylitol by microbial routes.

In the last two or three decades, there is a continuous rise in demand for xylitol in health, food, and medical sectors because of the growing health and food habit concerns. Recently, several new potential applications of xylitol have been found in medical applications in the form of low calorific sweetener, odontological applications, otitis media, antimicrobial property, and osteoporosis. Chapter 10 by **Arruda and co-workers** essentially provides the key and valuable information on the applications of xylitol in health and medical applications, confectionaries, and toothpaste, among others.

Finally, Chap. 11 by **Hans et al.** provides an important information on the market, consumption trends, and commercial status of xylitol production. This information is pivotal for the commercial-scale production of xylitol in the biorefineries.

In a nutshell, this book provides a comprehensive review on the current state and future perspectives of the technological developments in microbial production of xylitol. The book features the authoritative contribution from leading researchers from both academia and industry on xylitol production and its applications. Editors would like to thank Padma Subbaiyan, Werner Hermens, and Anthony Doyle from Springer Nature for their continuous encouragement and support to edit this important book.

Among the editors, **M. G. A. Felipe** is grateful to the National Council for Scientific and Technological Development—CNPq, the Research Council for the State of São Paulo—FAPESP Brazil, and Coordenação de Aperfeiçoamento de Pessoal de Nível Superior—CAPES for providing necessary research infrastructure and support for conducting research on various aspects of microbial conversion of lignocellulose biomass into xylitol and other bioproducts. **Anuj K. Chandel** is also grateful to the Research Council for the State of São Paulo (FAPESP), Brazil, for providing financial assistance (Process No. 2020/12559-6) through *jovem pesqvisador* program and the National Council for Scientific and Technological Development (CNPq) for the fellowship of scientific productivity (PQ: 1-309214/2021-1) in the area of biomass valorization into low-carbon fuels and chemicals.

The editors are confident that the book will serve the purpose to provide the colossus information to the readers of different disciplines who are keen to see the advancement in lignocellulose biotechnology, xylitol production, and sustainability.

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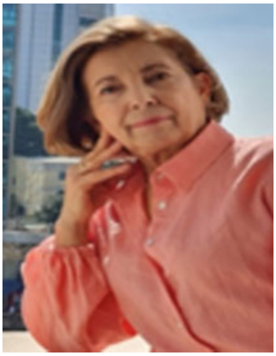
About the Editors



Prof. Dr. de Almeida Felipe Maria das Graças is a Full Professor in the Department of Biotechnology at the School of Engineering of Lorena—EEL, University of São Paulo—USP since 2020. She is working for almost 34 years at EEL-USP teaching in the area of applied microbiology and conducting research on the sustainable production of xylitol and other biomolecules such as ethanol from lignocellulosic biomass. Her line of research is advancing the scientific and technological knowledge to limit the bottlenecks in the biotechnological use of plant biomass concurrently imparting the training to undergraduate, and post-graduate students. Prof. Felipe in the Scopus database (March, 2022) figures as the second author with the largest number of publications in the world on the scientific work on "xylitol production" with the H-index of 33, while in Web of Science database she had H-index of 30, and 43 according to The Scholar Google database. Prof. Felipe has published 111 peer reviewed journals and 25 book chapters in the area of lignocellulose biotechnology, in a context of circular bioeconomy.

Prof. Chandel Anuj Kumar has 22 years research experience in industrial biotechnology working with Dalas Biotech Ltd, Bhiwadi India, University of Delhi, Celestial Biolabs Ltd, Hyderabad, India and Sugarcane Technology Centre-Piracicaba, Brazil. Dr. Chandel has also awarded MBA in agri-business having specialization in Biorefinery commercialization. Anuj has worked as a post doctorate fellow at University of Stellenbosch, South Africa, EEL-University of São Paulo, Brazil and University of Arkansas, Fayetteville, USA for 6 years. Anuj has worked at EEL-University of São Paulo-Lorena as a visiting professor for 3 years. Currently, he is working as a professor at USP-Lorena. He is an editor of 12 books on D-xylitol, lignocellulose degradation and Brazilian Biofuels Development, Biogas production, amongst others. Dr. Chandel has published 98 articles in peer-reviewed journals, 44 book chapters and recorded one Brazilian Patent. His contributions span industrial biotechnology, the circular carbon economy and policy domains. Dr. Chandel is an active consultant to some Biotech companies and startups in biomass conversion and biorefinery and industrial enzymes production.

Chapter 1
Methods for Hemicellulose Deconstruction Aiming to Xylose Recovery: Recent Progress and Future Perspectives

Sâmilla G. C. de Almeida, Veronica T. F. Silva, Jonas P. de Souza,
Cleiton D. Prado, Débora K. S. Oliveira, Débora D. V. Silva,
and Kelly J. Dussán

Abstract Lignocellulosic biomass currently represents the most significant potential to produce biofuels and biochemical compounds because of its abundance and cost savings. For using these materials, pretreatment is one of the essential steps in the biomass conversion process. However, the physical and chemical barriers from the main constituents (cellulose, hemicellulose, lignin) and their interactions form a hardheaded structure, creating a barrier to recover the fermentable sugars. Hemicellulose hydrolysis results in a xylose-rich hydrolysate that can be converted to high add-value chemicals, like xylitol. Each type of pretreatment will affect most downstream processes and represent a portion of the costs of the bioprocess. In this way, the selection of one pretreatment cost-effective and viable is a significant

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challenge. Moreover, each specific pretreatment will act differently in the lignocellulosic matrix; therefore, the choice must consider the configuration of the process used and the characteristics of the subsequent fermentation. This chapter aims to provide an overview and critically about hemicellulose deconstruction techniques aiming at xylose recovery, approach the perspectives, and show promising methods for disrupting the lignocellulosic matrix.

Keywords Lignocellulosic biomass · Pretreatment · Xylitol · Fermentation

1.1 Introduction

Lignocellulosic biomass has been targeted as a non-fossil, renewable, and low-cost feedstock for many different areas of application, such as fuels, energy, and biobased chemicals (Mankar et al. 2021; Choi et al. 2019; Arruda et al. 2021). The main aspects that characterize this biomass as an attractive alternative as feedstock for industrial purposes are its composition in carbohydrates and phenolic fraction, being relatively abundant, and its renewable and sustainable nature. Furthermore, applying the concept of biorefinery consists of processing a base material in a sustainable, economical, and environmental way to generate energy and bioproducts. The utilization of lignocellulosic biomass enables the obtention of a wide range of bioproducts such as biofuels (bioethanol, biodiesel) and chemicals with high add-value, namely xylitol, mannitol, furfural, among others (Arevalo-Gallegos et al. 2017; Romaní et al. 2020).

As many advantages as it has, lignocellulosic biomass presents a significant issue, a natural resistance to physical, chemical, and biological modification, limiting its usage. As a result of this resistance, also defined as recalcitrant, the extraction of cellulose, hemicellulose, and lignin fractions are quite complicated. Therefore, the application of pretreatment is required, as an essential step, to overcome this negative aspect of lignocellulosic biomass.

Pretreatments are classified in physical, chemical, and biological, and a combination of these techniques can be made to increase the efficiency of lignocellulosic deconstruction (Kim 2018; Kumar and Sharma 2017). However, selecting the ideal pretreatment must be low energy intake, selective toward the desirable fraction to be extracted, generating limited amounts of inhibitors products, and generally, the pretreatment must be feasible in terms of overall cost and sustainability (Mankar et al. 2021; Liao et al. 2020; Haldar and Purkait 2021).

The deconstruction process of lignocellulosic biomass enables the fractionation of cellulose, hemicellulose, and lignin and its subsequent transformation into high added-value products. Cellulose is a well-known base material on an industrial scale, and lignin and hemicellulose have been gaining attention regarding their high potential as starting materials.

Here it will highlight some characteristics of hemicellulose structure and methodologies and processes to its deconstruction aiming xylose recovery. Hemicellulose fraction consists mainly of a pentose (C5), as xylose. The sugars that constitute this fraction can be converted into bioethanol, antioxidant products (xylooligosaccharides), biofilms, xylitol, furfural, and gelling agents (Geng et al. 2019; Zhang et al. 2018; Jofre et al. 2021). Among these bioproducts, xylitol production has been extensively investigated due to its beneficial features in various industrial sectors, such as food, dental, pharmaceutical, and cosmetics (Jofre et al. 2021; Saha and Kennedy 2020). Xylitol is a high-value product whose price has been rising as reported by the global market, with an estimated value of US$1.3 billion up to 2027, with a 5.5% Compound Annual Growth Rate (CAGR) from 2020 to 2027 (Research and Market Reports, Xylitol - Global Market Trajectory Analytics 2021). This market expansion justifies the search for methods that enable the implementation of its production through a biotechnological route in biorefineries.

1.2 Factors Responsible for the Recalcitrance of Lignocellulosic Biomass

The complex and entangled structure of lignocellulosic biomass confers to this material a natural resistance against physical, chemical, and biological deconstruction. This natural resistance, so-called recalcitrant, has many sources within the constitution of lignocellulosic biomass. First, its highly organized structure (Fig. 1.1), where the cellulose microfibrils are embedded in an amorphous matrix made up of hemicellulose and lignin. The lignin by itself works as a physical barrier, protecting the carbohydrate fraction (cellulose and hemicellulose) from physical and chemical damages and biological attacks. The presence of lignin also provides adhesion and cohesion among the biomass fractions (Kim 2018; Brienzo et al. 2016; Ek et al. 2009). Besides the physical resistance conferred by its organized structure, the recalcitrance of lignocellulosic biomass to some extent is also due to the physical and chemical composition of each fraction of the biomass (Yoo et al. 2020).

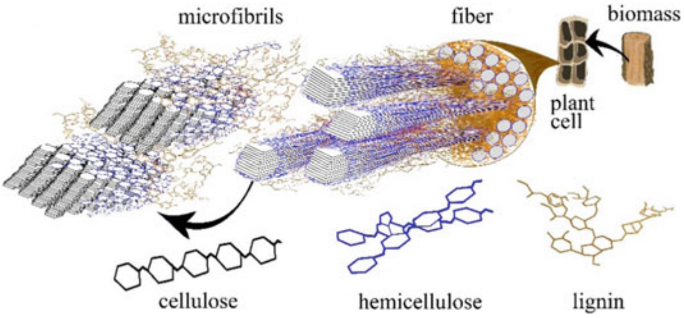

Fig. 1.1 Model proposed for the arrangement of the components in lignocellulosic biomass

Hemicellulose is a physical barrier that surrounds cellulose fibers, protecting them (Gírio et al. 2010). It is a heterogeneous and amorphous polysaccharide that has its chain built up from more than one type of monosaccharide such as pentoses (xylose and arabinose); hexoses (glucose, mannose, galactose), and other sugars (fucose, rhamnose). The hemicellulosic chain may also contain 4-O-methyl-glucuronic acid and be esterified with acetyl and feruloyl groups (Liao et al. 2020). The hemicellulose backbone is formed by the $\beta(1 \rightarrow 4)$ bond between the building blocks carbohydrates. In nature, hemicellulosic can differ between plant species and tissue types. Thus, it is possible to find different hemicelluloses, such as glucuronoxylan and glucomannan in hardwood, galacto-glucomannan, and arabino-glucuronoxylan and xylan, in hardwood grasses and softwoods (Fengel and Wegener 1984; Biely et al. 2016). However, the xylans are the most relevant and abundant hemicellulose type, and their hydrolysis releases monomers (mainly xylose) and oligomers (i.e., xylooligosaccharides) (Hilpmann et al. 2016).

Depending on the kind of biomass (sugarcane bagasse, sweet sorghum bagasse, barley straw, for example), the amount of hemicellulose can vary from around 18 to 30% (Dussán et al. 2016; Camargo et al. 2019; Moraes et al. 2020). Therefore, converting most hemicellulose into soluble sugars is essential to use this material in xylitol production efficiently.

1.3 Methods for the Deconstruction of Hemicellulose

Different methods can be employed to deconstruct the lignocellulosic biomass cell wall and the solubilization of sugars present in its fractions (hemicellulosic and cellulosic). There are physical, chemical, and physicochemical methods (Arora et al. 2020; Alokika et al. 2020). The choice of the technique must consider that maximum recovery of polymers from biomass is achieved, which will allow obtaining a hydrolysate rich in sugar and with a low content of toxic compounds, especially when it comes to hemicellulose hydrolysate (Arora et al. 2020; Alokika et al. 2020).

Considering the recalcitrance of lignocellulosic biomass, the efficiency in matrix degradation will depend on several factors, such as cellulose crystallinity, lignin content, and the existing interconnections between the hemicellulosic fraction and lignin (Bussemaker and Zhang 2013; Ravindran et al. 2017; Santos, et al. 2020). Therefore, sometimes a single pretreatment step is not enough for the hydrolysis of monomers; it will depend on the limitation of the pretreatment employed and other intrinsic disadvantages of the process (Flores et al. 2021). Thus, none treatment can be assumed to be fully efficient, and the use of combined treatments becomes pertinent; as it allows the incorporation of two or more lignocellulosic deconstruction techniques; maybe mechanisms of the same category or different (Mussatto et al. 2016), and allows to achieve high yields in sugar monomers with low severity conditions (Liu et al. 2017). Moreover, in some cases, providing the recovery of each component of lignocellulosic biomass in single streams, facilitating the subsequent conversion and valorization processes (Lomovsky et al. 2016; Moreno, et al. 2019).

Usually, the combination of pretreatment steps is closely associated with physical treatments (methods that are not modified chemically or biologically the lignocellulose structure). For example, the widest physicals pretreatment reported are milling, grinding, extrusion, and irradiation using ultrasound and microwave (Esfahani and Azin 2011; Nanda et al. 2016).

The use of unique chemical or mechanical pretreatment steps may require extreme conditions to deconstruct lignocellulosic biomass, which favors unsatisfactory monomer yields and a high generation of degradation products. Thus, efforts have been needed to combine treatments to perform well in mild processing conditions (Liu et al. 2017).

Each pretreatment method, despite its nature, has advantages and disadvantages, such as production cost, production of inhibitors, use of harsh chemicals. Thus, to select the best pretreatment for hemicellulosic extraction from lignocellulosic biomass aiming xylose production, it is crucial to follow some criteria, such as being able to reduce biomass recalcitrance by mitigating its causes, being efficient in pretreating different lignocellulosic materials, producing low content of inhibitors of microorganisms and be as sustainable and environmental-friendly as possible (Mankar et al. 2021).

This section will discuss the main methods of pretreatment of biomass for hemicellulose extraction aiming at xylose recovery. Future perspectives will be addressed as well in terms of new and advanced techniques.

1.3.1 Chemical Pre-treatments

Deconstruction of hemicellulosic fraction using acid, organic solvents, or the combination of them with other methods of pretreatment possibilities obtain hydrolysates rich in xylose that can be used for xylitol production. There are various possible pretreatments, but here it will be discussed those more common or preferred chemical pretreatment practices.

1.3.1.1 Acid Hydrolysis

Initially, acid hydrolysis was developed to separate or reduce the hemicellulose fraction of biomass, exposing the cellulose fibers to enzymatic action for bioethanol production. However, it was found that the large content of xylose in the C5 fraction should be considered as a raw material to produce both ethanol and other bioproducts (Moraes et al. 2020; Raj et al. 2021a; Yu et al. 2021). This fact has increased the interest in developing pretreatment methods that improve the efficiency of cellulosic hydrolysis and provide a hemicellulosic hydrolysate with a high content of xylose and the lowest possible content of inhibitor compounds, favoring its use as another raw material from lignocellulosic biomass.

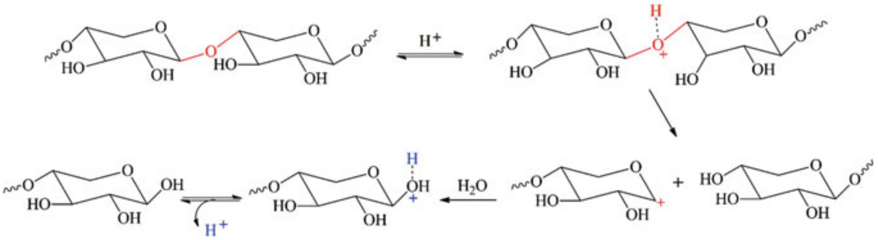

Fig. 1.2 General representation of acid hydrolyses in polysaccharides

In acidic pretreatments, depolymerization of carbohydrates also takes place, where the acid catalysis occurs in a few steps: a rapid electrophilic attack from proton (H^+) to the oxygen in the glycosidic bond (C–O–C), the generation of a carbocation ion that results from the mobility of the positive charge to the carbon one and the slow split of the glycosidic linkage (Fig. 1.2). As a result of these steps, a polysaccharide residue is released in the medium, such as oligosaccharides or monosaccharides. Finally, the remaining carbocation ion reacts with a molecule of water from the medium, and it is stabilized, and a proton (H^+) is released (Ek et al. 2009; Fengel and Wegener 1984).

For the reduction of hemicellulose polysaccharide in its monomers, acid hydrolysis has been conducted using organic (citric acid, acetic acid, formic acid) or inorganic acids (H_2SO_4; HCl, H_3PO_4), which are more effective (Camargo et al. 2019; Lavarack et al. 2002). Depending on the kind of biomass and the acid, it is possible to choose either concentrated (>10%) acid hydrolysis at a low temperature (<100 °C) or dilutes (<10%) acid hydrolysis at a high temperature (>100 °C) (Hilpmann et al. 2016; Lavarack et al. 2002). Other parameters such as solid-to-liquid ratio and the reaction time also are considered important for high hydrolysis efficiency (Moraes et al. 2020; Wijaya et al. 2014).

Acid hydrolysis of lignocellulosic biomass (pretreatment) promotes the extraction of pentoses and hexoses present in hemicellulosic fraction with efficiency over 80%, which is desirable to use them as raw material for xylitol bioconversion (Lavarack et al. 2002; Canilha et al. 2011). Silva et al. (2020) evaluated xylitol production from bagasse and straw and obtained 83% hydrolysis efficiency using diluted H_2SO_4 (100 mg/g dry matter, 1:10 solid–liquid ratio, 121 °C, 20 min), with low content of toxic compounds. Moraes et al. (2020) reported a diluted-acid pretreatment efficiency of 99% with minimal inhibitors content and energy consumption of 8.41 KW/Kg xylose for barley straw hydrolysis with 1.0% H_2SO_4 (w/v) at 120 °C, 40 min. Moreover, according to Dalli et al. (2017), the hydrolysis of poplar wood with diluted H_2SO_4 (1.75% (w/w) and 120 °C) resulted in a hydrolysate with three times higher xylose content and less by-product formation, improving xylitol production. Table 1.1 summarizes the xylose solubilization rates after diluting acid pretreatment with different feedstocks, acids, and other hydrolysis conditions.

As noted in Table 1.1, phosphoric acid has been less used than sulfuric acid, which can be 15 times more expensive than sulfuric acid (Siripong et al. 2016), resulting in

Table 1.1 Different acid hydrolysis of hemicellulose for xylose recovery

Feedstock	Hydrolysis parameters	Xylose recovery	References
Pinewood	4.49% phosphoric acid (H_3PO_4), 106.7 °C, 4.57 h, solution-to-feed ratio of (mL/g) 12.51	90.95%*	Cao et al. (2018)
Rice husk	2.60% sulfuric acid (H_2SO_4), 127 °C, 60 min, solid/liquid ratio (w/v) of 10	87.7%*	Temiz and Akpinar (2017)
Quercus mongolica (hardwood)	4.0% sulfuric acid (H_2SO_4), 121 °C, 105 min solid/liquid ratio (w/v) of 7	83.0%*	Jang et al. (2018)
Corn stover	0.7% hydrochloric acid (HCl) followed by wet milling could, 120 °C, 40 min, solid/liquid ratio (w/v) of 10	81.0%*	Liu et al. (2016)
Wheat straw	0.1 mol/L maleic acid ($C_4H_4O_4$), 150 °C, 40 min, 70 g/L solid/liquid ratio (w/v)	77.12%*	Liu et al. (2021)
Bioenergy sorghum	2% sulfuric acid (H_2SO_4), 120 °C, 5 min, 10% solids loading, after hydrothermal pretreatment and disk milling	64.9%*	Cheng et al. (2020)
Pine tree wood	5% sulfuric acid (H_2SO_4), 121 °C, 60 min, 1:10 solid/liquid ratio	92.0%*	Gonzales et al. (2016)
Empty palm fruit bunch	5% sulfuric acid (H_2SO_4), 121 °C, 30 min, 1:10 solid/liquid ratio	93.4%*	Gonzales et al. (2016)
Sugarcane straw	1% sulfuric acid (H_2SO_4), 121 °C, 20 min, 1:10 solid/liquid ratio	18.6 g/L	Hernández-Pérez et al. (2016)
Sugarcane bagasse and straw (1:1)	1% sulfuric acid (H_2SO_4), 121 °C, 20 min, 1:10 solid/liquid ratio	71.6 g/L**	Jofre et al. (2021)
Sugarcane bagasse and straw (1:1)	100 mg of H2SO4 per gram of dry matter, 1:10 solid–liquid ratio, at 121 °C for 20 min	18.76 g/L	Silva et al. (2020)
Rapeseed straw	2% sulfuric acid (H_2SO_4), 130 °C, 1 h, (10% w/v feedstock load)	10.30 g/L	López-Linares et al. (2018)

(continued)

Table 1.1 (continued)

Feedstock	Hydrolysis parameters	Xylose recovery	References
Corn cobs	0.5% nitric acid (HNO_3) 121 °C, 30 min, 1:10 of solid/liquid ratio	15 g/L	Kumar et al. (2018)

*Based on xylan
**After vacuum evaporation

a higher cost of the process. Nonetheless, its use is investigated for being associated with a lower generation of degradation compounds and minor corrosion in the reactors. On the other hand, Sulfuric acid is the most used in acid hydrolysis. It is the main method of effective depolymerization and enhancement of C5 fraction from lignocellulosic biomass due to its easy operation and low cost. However, its main disadvantage is the consumption of alkalis to neutralize the hydrolysate obtained (Loow et al. 2761; Girisuta et al. 2013; Norrrahim et al. 2021; Akhlisah et al. 2021). Wan et al. (Wan et al. 2021) evaluated to overcome this problem by employing hydrolysis with solid acid sulfated zirconia. However, detoxification of hydrolysate continued to be recommended for its use as a fermentation medium for xylitol production. Acidic solids catalyzed hydrolysis has shown promise, emphasizing the use of heteropoly-acids, zeolites, mesoporous materials, sulfonated or modified with acid oxide derived from tungsten or zirconium (Amarasekara and Wiredu 2012; Guo et al. 2012; Lu et al. 2021). Their utilization has been gaining notability, as it provides easy separation from the final product, with the perspective of recovery and reuse of the catalyst (Suganuma et al. 2008; Lanzafame et al. 2012; Ye et al. 2021).

Detoxification of hydrolysate is necessary because during acid hydrolysis, even in low temperature and short time, the reactions release sugars, and other compounds, which are inhibitory to fermentative processes. Such compounds can be classified into four main groups: by-products derived from sugars (furfural originated from the degradation of pentoses and hydroxymethylfurfural, formed in the degradation of glucose), acetic acid (from acetyl groups present in hemicellulose fraction), lignin degradation products that include a wide variety of aromatic and polyaromatic compounds. Furthermore, the pretreatment using strong acids, high temperature, and pressure promotes the wear of the equipment and the presence of metal and minerals that can be inhibitors for microorganism metabolism (Palmqvist and Hahn-Hägerdal 2000; Silva et al. 2004, 2014; Alvira et al. 2010).

1.3.1.2 Organosolv

Organosolv is a pretreatment method that uses a solvent system for the deconstruction of lignocellulosic biomass using organic aqueous solvent (i.e., methanol, ethanol, and acetone), in a temperature range of 100–250 °C and must occur under controlled conditions due to the associated risk of volatile solvents (Raj et al. 2021a; Espinoza-Acosta et al. 2014; Zhao et al. 2009). As a result, monomers from hemicellulose and lignin are obtained dissolving in the liquid fraction, and the solid fraction obtained is rich in cellulose (Zhao et al. 2009; Teramura et al. 2018; Batista Meneses et al. 2020). In addition, high purity xylose and lignin are obtained during posterior treatment of liquid fraction (Zhang et al. 2016).

When alcohol is used as an organic solvent in lignocellulosic biomass at high temperatures and pressure, the hemicellulose and lignin will be fragmented and solubilized in the liquor (Wildschut et al. 2013). As a result, the availability of sugar monomers and degradation compounds may occur. Therefore, filtration is used to separate the cellulosic fraction from the black liquor. For solvent recovery, the liquid is evaporated, the concentrated fraction is mixed with acidic water so that the lignin is precipitated. A further filtration and distillation step are employed to separate components such as furfural, acetic acid, and xylose; from the hydrolysis of hemicellulose (Zhang et al. 2016). Thus, obtaining at the end of the process a pulp consisting mainly of cellulose, high-purity lignin, and an aqueous fraction containing the constituent monomers of hemicellulose and their degradation products (Wildschut et al. 2013; Rio et al. 2012).

The ethanol process was applied to fractionate wheat bran, evaluating the temperature (160–200 °C) and ethanol concentration (30–60%w/w) for 30 min, and conversion of hemicelluloses to xylose 60% was obtained (Reisinger et al. 2014). Previous research on wheat straw used a three-step aqueous pretreatment biorefining approach to pre-hydrolysis hemicellulose to sugars, (2) organosolv delignification, and (3) enzymatic hydrolysis of cellulose to glucose. Fractionating wheat bran using 60% ethanol, 10/1 (v/w) liquor/solid ratio, 220 °C and 60 min, 67% xylose yield xylose was obtained (Huijgen et al. 2012).

Organosolv pretreatment is commonly performed at harsh conditions (high concentrations of organic solvents (>50%) and high temperatures) and may be necessary to use a catalyst to obtain a higher xylose yield (Zhang et al. 2016; Huijgen et al. 2012; Meng et al. 2020). In addition, mineral acids such as hydrochloric acid, sulfuric acid, and phosphoric acid can accelerate the process of delignification and degradation of xylan. Still, organic acids (formic acid, oxalic, acetylsalicylic, salicylic acid) can also be used as catalysts (Zhao et al. 2009; Huijgen et al. 2012).

Teramura et al. (2016), aiming to reduce processing cost, evaluated fractioning sorghum bagasse with low solvent concentration. The authors reported effective biomass fractionation using 12.5% 1-butanol or 1-pentanol as the solvent, 1% H_2SO_4, 180 °C, 45 min. Teramura et al. (2018) also evaluated the solvent recovery using a nanofiltration membrane. They reported that this step could be beneficial for using the hemicellulosic hydrolysate in fermentative processes.

According to Zhang et al. (2016) and Salapa et al. (2017), especially in a process with not very high temperatures (<195 °C), ethanol and methanol could be chosen as a solvent, since they are more easily recovered, preferably ethanol, due to lack of toxicity. After treatment, the liquor (black liquor) is evaporated and condensed, recovering the alcohol solvent, and additional filtration is applied to recovering xylose.

Other possibilities also have been explored. For example, Huijgen et al. (2010) reported 82% hydrolysis of hemicellulose from wheat straw employing 50:50%

(w/w) acetone–water for 60 min at 205 °C. Furthermore, Yu et al. (2015) showed that 94.1% of xylan was converted in xylose from sugarcane bagasse pretreated with green liquor-ethanol combined with H_2O_2 without using xylanase.

1.3.2 Hydrothermal Technologies

Hydrothermal pretreatments are the most promising fractionation technologies for lignocellulosic biomass-based biorefineries (Díaz et al. 2021). They consist of a combination of physical and chemical principles operating in an aqueous medium under different temperature and pressure ranges. In addition, hydrothermal pretreatment is an effective technique for extracting hemicellulose fraction highlights: steam explosion and autohydrolysis (Beig et al. 2020; Sarker et al. 2021).

1.3.2.1 Steam Explosion

Steam explosion (SE) is a widely used method for fractionating lignocellulosic biomass and disrupts xylan structure. This method combines thermal, chemical, and mechanical effects on biomass. As a result, it is possible to obtain hemicellulosic hydrolysate, change cellulose crystallinity, and induce lignin transformations (Duque et al. 2016; Cebreiros et al. 2021).

The steam explosion system comprises a steam generator that feeds a reactor, which uses high pressure saturated steam at temperatures 160 and 260 °C and is maintained from seconds to several minutes (Biswas et al. 2015; Bhutto et al. 2017). Subsequently, a sudden depressurization occurs, the material is ejected from the reactor and recovered in the explosion tank (Jacquet et al. 2015).

The steam explosion occurs without a catalyst, promoting acetyl groups' liberation present in the biomass (Carvalheiro et al. 2008). As a result, cleavage of glycosidic bonds and hemicellulose solubilization occurs at lower temperatures (190 °C, 10 min) or short residence time (270 °C, 1 min). As a result, a liquid fraction and insoluble solids from the pretreated material. The liquid, or hydrolyzed, contains mainly hemicellulose sugars solubilized and almost also degradation products generated during pretreatment (Carvalheiro et al. 2008; Mosier et al. 2005).

Some factors can influence the efficiency of the steam explosion, the main ones being particle size, temperature, moisture, and residence time (Duque et al. 2016). The high residence time favors complete hydrolysis of the hemicellulosic fraction and contributes to fermentation (Jacquet et al. 2015). However, the oligosaccharides and monosaccharides generated may undergo dehydration, fragmentation, and condensation reactions. The products obtained from these reactions are furfural, hydroxymethylfurfural, levulinic and formic acid, and aromatic compounds and are generated due to increased retention time (Jacquet et al. 2015). All these degradation products are fermentation inhibitors, so they must be minimized. Thus, high temperatures (220 °C) decrease overall carbohydrate yield due to the parallel reactions

(Ruiz et al. 2008). In addition, particle size and raw material moisture influence heat transfer into of reactor. For example, small biomass particles have better and faster heat transfer, avoiding surface overheating, production of degradation products, and incomplete hydrolysis (Duque et al. 2016; Brownell et al. 1986).

Bonfiglio et al. (2021) reported that to obtain hydrolysates of switchgrass and *Eucalyptus globulus* biomasses rich in sugars, avoiding a subsequent concentration step before fermentation, the better conditions were 200 °C and 10 min residence time, and the hydrolysates obtained were efficient for xylitol production.

An alternative to improve the solubilization of hemicellulose is to use a combination of acid catalysts such as SO_2, H_2SO_4, H_3PO_4, and steam explosion process (Duque et al. 2016; Devi et al. 2021). Furthermore, when the steam explosion is made with softwood, it is essential to combine this pretreatment with the impregnation of biomass with acids. This combination helps to reduce the residence time and temperature of the process and produce low degradation products (Devi et al. 2021; Behera et al. 2014).

Wang et al. (Wang et al. 2015) proposed a pretreatment of corn straw combining impregnation of biomass with 1% H_2SO_4 followed by a steam explosion at 180 °C for 9 min and obtained a hydrolysate xylose-rich and with low content of furfural and 5-HMF, that was fermented, without detoxification step, with effective xylitol production by *Candida tropicalis.*

Walker et al. (2018) also evaluated the use of steam explosion pretreatment (12 bar(g), 3 min, 1.2% H_3PO_4, and 500 g substrate) for different feedstock (wheat straw, corn stover, Miscanthus, and willow). They yielded up to 94% xylose release with minimal fermentation inhibitor production, allowing xylitol production without detoxification, by *Schefferomyces shehatae.* Still, the hydrolysates were concentrated to increase xylose concentration.

The use of H_3PO_4, although less common, has been pointed out more advantage over the H_2SO_4 as a catalyst for SE, to be less toxic and corrosive, contributing to reducing the capital cost of the plant (Duque et al. 2016; Geddes et al. 2011). Furthermore, it is possible to recover phosphoric acid and use it as a source of nutrients (López-Linares et al. 2013).

1.3.2.2 Auto-Hydrolysis

Auto-hydrolysis or hot water pretreatment (LHW) is a pretreatment process without using chemicals, in which compressed liquid water is used to hydrolyze the hemicellulose fraction. This process has a mechanism similar to acid hydrolysis, and the reaction conditions and decomposition mechanism have been widely studied (Yu et al. 2012; Zhuang et al. 2012). In this method, the self-ionization of water takes place, releasing hydrogen ions, promoting the depolymerization of hemicellulose fraction. Besides, the acetyl groups, present in lignocellulosic biomass, also act as catalysts, improving the reaction mechanism (Yu et al. 2012; Zhuang et al. 2012; Gullon et al. 2012; Serna-Loaiza et al. 2021; Carvalheiro et al. 2009).

Autohydrolysis allows the selective hemicellulose extraction at high temperatures, without catalysts, and without lignin solubilization, obtaining a liquid fraction rich in soluble saccharides and a solid fraction rich in cellulose and lignin.

However, studies show that hemicellulosic hydrolysates obtained by autohydrolysis of biomass are complex. Such hydrolysates contain a mixture of oligosaccharides (60–80%), monosaccharides (especially pentoses, 10–15%). They can also present inhibitory compounds of microbial metabolisms, such as acetic acid and some furans (10–15%), such as furfural and 5-hydroxymethylfurfural, which can further develop decompose, producing formic and levulinic acid (Yu et al. 2012; Zhuang et al. 2012; Carvalheiro et al. 2009; Kuhad et al. 2010; Amendola et al. 2012), requiring a further detoxification step.

The factors that affect autohydrolysis are temperature associated with heating and cooling profile, reaction time, and liquid–solid ratio. The most commonly used temperatures are in the ranges of 150–250 °C, under high pressures to keep water in a liquid state, with S/L ratios of 2–100 (w/w) and reaction times of seconds to hours. Under these conditions, the extraction processes of the hemicellulosic fraction present 55–84% recovery (Gírio et al. 2010; Gurgel et al. 2014; Garrote et al. 1999; Walsum 2001).

This procedure is economical and environmentally correct when comparing autohydrolysis with other biomass pretreatment methods (Carvalheiro et al. 2009). In addition, LHW also has other advantages such as low energy requirement and high yields, few corrosive problems due to its moderate pH, and can be used in a variety of lignocellulosic biomasses, such as sugarcane bagasse, grape stalks, corn cobs, sweet sorghum bagasse. Therefore, it could be used in biorefineries, contributing to less waste generation, and promoting the efficient use of raw materials, minimizing environmental impacts (Carvalheiro et al. 2008; Amendola et al. 2012; Gurgel et al. 2014; Garrote et al. 1999; Amiri and Karimi 2015; Khalili and Amiri 2020; Rivas et al. 2002).

Some authors have reported the combination of LHW with other pretreatments, mainly using acid, trying to increase xylose extraction. Brandenburg et al. (2016) pretreated birch wood chips with hot water extraction (165 °C, 90 min) followed by acid hydrolysis (4% H_2SO_4, 120 °C, 60 min) and obtained a hydrolysate with 45.06 g/L xylose, 0.46 g/L glucose, 13.07 g/L acetic acid, and 4.70 g/L furfural. Kim et al. (2015) reported the use of acetic acid in the hydrothermal fractionation for method sugar recovery from empty fruit bunches, achieving 50.7% sugars (xylose, mannose, and galactose) recovery yield (and 1.0 g/L furfural) with 6.9 wt.% acetic acid, at 170 °C, 18 min.

Lyu et al. (2018) also evaluated LHW but used mixed acid (lactic acid + acetic acid) to treat wheat straw and corn straw. The authors reported a mixed acid synergistic catalysis (3 wt%) on strengthening LHW pretreatment at 180 °C for 60 min, obtaining a C5 sugars yield of 93.83%.

Such results encourage new studies to adjust this pretreatment to become a viable alternative in biorefineries.

1.3.3 Physical Pretreatments

Physical pretreatments include mechanical operations, irradiations, and ultrasonic processes (Bhutto et al. 2017; Subhedar and Gogate 2016; Sun et al. 2016). However, these methods alone cannot obtain oligomers and monomers to be hydrolyzed and used as substrates in biotechnological processes, so they are usually combined with another type of pretreatment.

1.3.3.1 Ultrasound

The association of conventional pretreatments with the application of ultrasonic irradiation is an excellent approach to intensify the deconstruction of lignocellulosic biomass. Furthermore, studies have addressed that the application of ultrasound associated with another pretreatment significantly improves the yield of polysaccharides from biomass (Flores et al. 2021; Minjares-Fuentes et al. 2016).

The treatment is based on the formation of cavities/bubbles micro size, which results from the propagation of ultrasound waves in a liquid medium. This phenomenon is called cavitation, which results in physical and chemical effects in liquid solutions (Ravindran et al. 2017; Zheng et al. 2014; Leong et al. 2011). The physical impact caused is a collapse of the cavitation bubbles formed and intensified the fluids transport and furthered the dissolution of solid particles. In addition, this result will produce chemical changes through the formation of free radicals (Flores et al. 2021; Gallo et al. 2018). The combination of these effects generates impact and destruction of the plant cell wall.

The result of these cavities results in high temperatures and pressure gradients in specific regions. This entire process lasts for a short period, creating a hot-spot effect in the liquid (Kentish and Ashokkumar 2011). In addition, the high energy generated in the cavitation regions results in a significant collapse in the lignocellulose matrix, which leads to substantial changes in morphology, with a high contact surface, being advantageous for the intensification of other pretreatments due to the greater surface area that results in better mass transfer (Yan et al. 2021).

Based on that, the energy generated by the ultrasound process has been reported as an efficient method for enhancing several types of pretreatment, which is associated with the turbulence of the process, making the oligomers that compose the biomass more available for subsequent operations, and a decrease in lignin content (Bussemaker and Zhang 2013). Besides, ultrasound has the advantage of removing inorganic materials associated with biomass, especially when using low operating frequencies (Yunus et al. 2010).

Thus, the associated use of ultrasound with other technologies, such as chemical treatment to fractionate the biomass and obtain the hemicellulosic fraction, can increase yields and generate less waste and toxic by-products.

The application of ultrasound beyond the cavitation zones results in intense turbulence, shock waves, and shear forces with high pressures and temperatures for a short period due to the bubble collapse (Subhedar and Gogate 2016; Kentish and Ashokkumar 2011). Furthermore, cavitation results in a mechanical effect called microjets, one of the main mechanical effects. Bubbles will collapse asymmetrically in the solid surface, and a formed liquid jet implodes on the particle's surface, decreasing the size of these particles (Leong et al. 2011; Singh et al. 2014). On the other hand, micro-streaming is a consequence of cavitation in the presence of suspended powder. Thus, it can be assumed that these mechanical effects act in synergy, whereas microjets reduce particle size and disperse particles facilitating the dissolution.

Hence, the technique employed results in a mechanochemical effect (Rooze et al. 2013) that will accelerate the extraction of organic compounds from the lignocellulosic biomass by causing a kind of cell wall rupture, increasing the contact surface and mass transfer cell content (Yuan et al. 2010; Luo et al. 2014). Firstly, the radicals formed during cavitation will cleavage the process of lignin and xylan networks, together with the shear forces of the ultrasonic mixture (Ong and Wu 2020).

This combination of factors leads to significant physical and morphological changes, like an increase in surface area, a reduction in the degree of polymerization, which will result in a greater degree of intensification of the associated treatment, bringing more expressive yields as well as greater use of lignocellulosic biomass (Yunus et al. 2010; Sun et al. 2021). Table 1.2 shows various ultrasound-assisted pretreatment for the many lignocellulosic biomass.

A study by Yunus et al. (2010) studied the effect of dilute sulfuric acid with ultrasonic pretreatment of oil palm empty fruit bunch. The ultrasound treatment showed to be more effective for recovery xylose (58%) of the hemicellulosic fraction when compared with the condition in the absence of ultrasonic (22%). Yang and Wang (2019) also used the combination of acid treatment with ultrasound to improve biomass pretreatment efficiency for fermentable sugars recovery. This ultrasound-assisted dilute acid pretreatment also reduces the required amount of acid for the pretreatment, which is more environmentally attractive.

Minjares-Fuentes et al. (2016) realized an ultrasound-assisted alkaline treatment to extract pentoses from grape pomace; the maximum yield extract was 8.6%. Thus, the author concluded that it is possible to obtain the hemicellulose fraction using low concentrations of KOH and short extraction times when associated with ultrasound; however, it is still a low performance when considered a large-scale application.

The frequency used in ultrasound is one of the main parameters influencing the physicochemical changes in biomass fiber. It is generally used in the range of 16–100 kHz; the increased frequency is necessary to create the cavitation zones, in addition to causing the flow of liquid required and turbulence (Luo et al. 2014). Another parameter is the time that the ultrasonic is applied. Thus, it can be established that the processing time will depend on the biomass recalcitrance used, mass transfer resistances, and the desired fractionation (He et al. 2017). Studies used for the recovery of hemicellulose state that, in addition to a time established as optimal, excessive severity in the use of ultrasound can result in an unchanged fractionation or even be harmful to the pretreatment process, and consequently results in an aggregate of particles reducing the surface area of the biomass (Loow et al. 2016; Ong and