



Production Strategy of Functional Oligosaccharides from Lignocellulosic Biomass Using Enzymatic Process: A Review

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Abstract

Lignocellulosic biomass (LB) is a promising source that has the potential to revolutionize the world of bioenergy, bioproducts, and nondigestible dietary components. As a substrate, LB has proven to be particularly attractive for the production of high-value cello-oligosaccharides (COS) and xylo-oligosaccharides (XOS), which offer diverse applications in the food, biopharmaceutical, and other industries, as well as potential health benefits, including prebiotic and antidiabetic effects. However, despite these promising developments, the manufacturing of these oligosaccharides remains a challenge due to slow reactions and low yields. Therefore, this review presents various pretreatment techniques to improve enzymatic hydrolysis, as well as the possibilities of employing a multi-step process and utilizing thermostable enzymes to enhance the production of COS and XOS from LB. Additionally, this review addressed the potential for by-product recovery during the XOS and COS production and the separation of β -glucosidase enzymes using the “separation–adsorption” method in high-temperature and continuous systems for COS production.

Keywords Cellulase · COS · Lignocellulose · Multi-step · Thermostable · XOS

Overview

Lignocellulosic biomass (LB) is a promising source of renewable energy, bioproducts, and nondigestible dietary components (Itelima et al., 2013; Yuansah et al., 2019). Its versatility makes it an attractive material to explore, particularly in the production of high-value cello-oligosaccharides (COS) and xylo-oligosaccharides (XOS). As nondigestible dietary components, XOS and COS have diverse applications in industries. These oligosaccharides offer promised health benefits, including antidiabetic and prebiotic characteristics, making them valuable for the food and biopharmaceutical industries (Lin et al., 2016; Yang et al., 2015a; Zhong et al., 2020). However, despite these exciting developments, the production of these oligosaccharides remains a challenge due to slow reactions and low yields.

Lignocellulose is formed by three major biopolymer components: cellulose, hemicellulose, and lignin (Pointner et al., 2014). Cellulose and hemicellulose are carbohydrates that consist of glucose, xylose, mannose, arabinose, and galactose (Wang et al., 2016). When the homopolysaccharide, cellulose, is partially hydrolyzed by β -1,4-endoglucosidase, it will release COS and a little amount of glucose. While heteropolysaccharide, hemicellulose, will release some oligosaccharides (such as XOS, manno-oligosaccharides (MOS), arabino-xylo-oligosaccharides (AXOS), etc.) and monosaccharides (Saville & Saville, 2018; Yuansah, 2019).

A diverse range of oligosaccharides derived from LB has been found to exhibit various functions. For instance, when the *Bifidobacterium* group in the human gut fermented XOS, it resulted in a notable increase in acetate production (Takagi et al., 2016). On the other hand, COS showed a significant increase in acetic acid and propionic acid, while butyric acid and formic acid remained unchanged (Karnaouri et al., 2019). Additionally, the fermentation of AXOS by fecal bacteria produced acetate, propionate, and butyrate (Broekaert et al., 2011). The specific type of short-chain fatty acids (SCFAs) produced was dependent on the source of the fecal inoculum and the type of oligosaccharides. The discrepancies in SCFAs produced result in various health effects caused by these metabolites in the human body (Karnaouri et al., 2019; Jian Tan et al., 2014).

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The conversion of LB into high-value oligosaccharides involves various pretreatment processes and enzymatic hydrolysis. However, the enzymatic conversion of the cellulose fraction is a slow hydrolysis reaction. To optimize the oligosaccharide yield from the highly resistant lignocellulose complex structure, proper pretreatment techniques are necessary (Zoghلامي & Paës, 2019). Inhibitors such as lignin and other polyphenolics must be handled during pretreatment before entering the enzyme hydrolysis phase (Lei Qin et al., 2016; Zoghلامي & Paës, 2019). The pretreatment breaks down the crystalline structure of cellulose, reduces the degree of polymerization, removes the lignin, and breaks down hemicelluloses (Baruah et al., 2018). Therefore, proper pretreatment procedures are essential to achieve the highest oligosaccharide yield from various lignocellulosic substrates.

The enzymatic hydrolysis of lignocellulosic biomass for oligosaccharide production can be optimized by using thermostable enzymes and high temperatures. Utilizing thermophilic or thermotolerant microbes to produce such enzymes can overcome the limitations of lignocellulose hydrolysis. The use of thermostable enzymes in high-temperature reactions can significantly improve the efficiency and speed of the process while reducing the risk of contamination (Giovannoni et al., 2020). Due to the stability of thermostable enzymes, their use in enzymatic hydrolysis leads to the production of more specialized products and fewer by-products. This allows for a longer hydrolysis time and greater flexibility in the configuration process (Giovannoni et al., 2020; Vasconcellos et al., 2015).

Recent studies have demonstrated that the addition of surfactants, such as PEG2000, to alkali treatment, can enhance the efficiency of the delignification and saccharification process of lignocellulosic biomass. This approach can result in more effective removal of lignin, microstructural destruction, and higher levels of reducing sugars compared to regular alkaline treatment. Moreover, surfactants can improve sugar recovery, reduce enzyme loading, and inhibit enzyme–lignin interactions during enzymatic hydrolysis (Cheng et al., 2022). Sonication treatment, particularly ultrasonic-assisted hydrolysis, can also increase the yield of XOS by disrupting the molecular aggregation on substrate chains. However, the energy consumption and production expenses associated with surfactants and sonication-mediated treatments must be considered when applied to a wide range of substrates (Zhou et al., 2021).

To achieve optimal oligosaccharide production from lignocellulosic biomass, a multi-step process integrating various stages can be implemented to minimize inhibitors and improve product yield and process efficiency. Chu et al. (2014) successfully improved COS yield to 51.78% and enzymatic hydrolysis yield to 75.56% by using a three-stage enzymatic hydrolysis process and separating the product

after each hydrolysis step to prevent product accumulation and enzyme inhibition. According to the literature, proper pretreatment and a multi-step process using thermostable enzymes can further optimize the enzymatic activity and product yields of XOS and COS. This article provides a brief description of lignocellulose sources and their potential as enzyme substrates for functional oligosaccharide production to facilitate the selection of the optimal substrate for the manufacture of COS and XOS. With the idea of enhancing product yields through suitable pretreatment and a multi-step enzymatic process, this article aims to make it easier for researchers to find the strategies to produce COS and XOS.

Lignocellulose Source for Functional Oligosaccharides Production

Agricultural waste is the primary source of lignocellulose, which is made up of biopolymers such as cellulose, hemicellulose, and lignin. The agro-waste product lignocellulose consists of cellulose (40–50%), hemicellulose (20–30%), and lignin (10–25%) (Maheshwari, 2018; Sharma et al., 2019). Because of their availability and abundance of essential polysaccharides, cellulose (consisting of D-glucose) and hemicellulose (consisting of glucose, mannose, xylose, arabinose, galactose, etc.), agricultural waste such as rice straw (Hu et al., 2021; Li et al., 2018), rice husk (Khat-udomkiri et al., 2020), wheat straw (Collins et al., 2014; Shrivastava et al., 2014), sugarcane bagasse (de Souza et al., 2013; Guilherme et al., 2015), sugarcane straw (Barbarosa et al., 2020), cacao pod (Lu et al., 2018), sago pith residue (Husin et al., 2019), corncob (Li et al., 2014; Silva et al., 2015), apple pomace (Hijosa-Valsero et al., 2017), red algae (Rabemanolontsoa & Saka, 2013), silvergrass (Kendrick et al., 2022), jiuzaio (Liqin Qin et al., 2022), banana pseudostem pulp (Díaz et al., 2021), *Eucalyptus* by-product (Neto et al., 2020), and birch and spruce wood (Karnaouri et al., 2019) can be potential candidates as an enzymatic substrate to produce xylo- and cello-oligosaccharides.

The main component of lignocellulosic biomass is cellulose, which is the most widespread and easily accessible carbohydrate polymer on the planet and a primary polysaccharide element of plant cell walls (Maheshwari, 2018). Cellulose is a homopolysaccharide, a carbohydrate biopolymer composed of monomeric β-D-glucopyranose linked together by a 1,4-glycosidic bond. The structure of the cellulose chain consisted of 500–1400 β-D-glucopyranose (D-glucose) molecules that packed together to form microfibrils. A macrofibril (cellulose fibrils) is made up of microfibrils that have been joined together. Because of the presence of cellulose fibrils, lignocellulosic biomass is particularly resistant to enzymatic hydrolysis (Zoghلامي & Paës, 2019). Cello-oligosaccharides can be manufactured

by hydrolyzing cellulose using β -glucosidase-deficient cellulases (Chu et al., 2014). Hemicellulose is the second most common substance after cellulose (Maheshwari, 2018). Hemicellulose is a heteropolysaccharide made up of several different carbohydrate monomers, particularly pentoses sugar units such as xylose and arabinose, hexoses sugar units such as mannose, glucose, and galactose, along with acylated sugar, which is represented in varying ratios in diverse materials (Dionisi et al., 2014; Maheshwari, 2018). In hardwoods, xylan is the most common biopolymer, while in softwoods, glucomannan is the most abundant (Dionisi et al., 2014). Hemicellulose can be partially hydrolyzed and degraded into XOS utilizing endo-1,4- β -xylanase (Moser et al., 2014).

Enzyme Inhibitors in Lignocellulosic Biomass

The pretreatment of biomass plays a crucial role in reducing the presence of inhibitors, such as lignin, phenolic groups, and sugars, that can naturally accumulate or be generated during the production process (Hu et al., 2016; Lei Qin et al., 2016). Hydrothermal pretreatment, for example, can produce acetic acid and furan aldehydes as by-products, while mild alkaline pretreatment removes lignin and some hemicellulose, producing side products such as acetic acid, hydroxy acids, dicarboxylic acids, and phenolic compounds (Chu et al., 2018; Jönsson & Martín, 2016; Marcondes et al., 2020; Santo et al., 2018). Phenolic compounds, in particular, can strongly inhibit the activity of amylase and cellulase enzymes (Desseaux et al., 2018; González-Bautista et al., 2017). It is worth noting that some oligosaccharides, such as XOS and GOS, produced from hydrothermal pretreatment, can strongly inhibit the action of cellulase enzymes, particularly cellobiohydrolase (CBH). However, the inhibition of endoglucanase (EG) action by these oligosaccharides is weaker than that of CBH (Kont et al., 2013). High concentrations of monosaccharides can also significantly inhibit the activities of EG and CBH enzymes, although β -glucosidase is less affected, except by glucose (Hsieh et al., 2014) (Table 1). Thus, it is important to consider the inhibitor content of substances when selecting materials for the enzymatic process. Using materials with lower inhibitor content will require milder pretreatment, but using resistant materials is preferable for effective waste treatment.

Pretreatment Techniques to Improve Enzymatic Hydrolysis Process

There are several pretreatment strategies available to optimize the oligosaccharide yield from lignocellulose biomass conversion. Because of its complex structure, the crystallinity of cellulose, and porosity, lignocellulose is highly

resistant to enzymatic hydrolysis (Zoghلامي & Paës, 2019). Various inhibitors, such as lignin and other polyphenolic compounds, must be treated in the pretreatment step before entering the enzyme hydrolysis phase (Lei Qin et al., 2016; Zoghلامي & Paës, 2019). The objective of the pretreatment process is to break down the crystalline structure of cellulose and minimize the degree of polymerization, eliminating all lignin and breaking down hemicelluloses (Baruah et al., 2018). Proper pretreatment strategies are required to obtain the greatest yield of oligosaccharides from the bioconversion of different lignocellulosic substrates (Table 2).

The pretreatment process can result in the removal of lignin and the destruction of the microcrystalline structure of cellulose, as well as the formation of oligosaccharides and monosaccharides. For example, liquid hot water pretreatment generates sugar and oligosaccharides, but the concentration of the sugar is quite low (Huang et al., 2017). The hemicellulose concentration was drastically reduced, although only minimal alterations in cellulose and lignin were identified (Antczak et al., 2022). Alkali pretreatment can significantly reduce lignin while producing fewer unwanted inhibitors. It also consumes little energy. However, there remains a possibility that this pretreatment will result in unrecovered salt (Jinyu Tan et al., 2021). Acid pretreatment can also disrupt the lignocellulose and amorphous cellulose structures. Unfortunately, in contrast with alkali pretreatment, this pretreatment consumes a large amount of energy and generates a large number of byproducts (Solarte-toro et al., 2019; Jinyu Tan et al., 2021). Nowadays, research using greener technology using organic solvents such as ionic liquid (IL) (choline, pyridine, chloride, acetate, and imidazole) and deep eutectic solvent (DES) that are more favorable with enzymes and microbes. However, these approaches still face challenges because the solvent recovery of iLs will cost more than other solvents, while DESs have disadvantages such as high viscosity and toxicity (Jinyu Tan et al., 2021; Zhao et al., 2022). To improve the effectiveness of lignocellulosic waste pretreatment, a combination of pretreatment techniques is often used.

Potential Microbial Thermostable Enzymes Source for Lignocellulose Bioconversion

Enzymatic conversion of lignocellulosic biomass, particularly the cellulose fraction, is typically carried out at 40–50 °C, which is considered a slow hydrolysis reaction. The hydrolysis process has a low sugar yield, partial hydrolysis, and a high risk of microbial contamination (Patel et al., 2019). These limitations could be overcome by using thermophilic or thermotolerant microbes to produce thermostable enzymes for lignocellulosic bioconversion (Table 3). Thermostable enzymes have the advantage of being able

Table 1 Enzyme inhibitors in lignocellulosic biomass

Inhibitor	Source	Inhibited enzymes	Ref
Crude phenolic	Sugarcane bagasse, apple pomace, pecan	CMCase, xylanase, α -amylase, α -glucosidase	Feng and Kong (2022), González-Bautista et al. (2017), and Hijosa-Valsero et al. (2017)
<i>p</i> -Coumaric acid	Corn stover, cocoa pod husk	Cellulase, xylanase	Chen et al. (2020) and Lu et al. (2018)
Quercetin	Cocoa pod husk, cocoa powder, <i>Mucuna pruriens</i> seed	Cellulase	Lu et al. (2018), Qin et al. (2016), Rai et al. (2017), Sorrenti et al. (2020), and Stamogiannou et al. (2021)
Kaempferol	Water lily	α -Glucosidase, cellulase	Mugaranja and Kulal (2020), Qin et al. (2016), and Stamogiannou et al. (2021)
Tannins/tannic acid	Oak bark, acorn caps	β -Glucosidase, cellobiohydrolase, endoglucanase	Jönsson et al. (2013), Liu et al. (2021), Majewska et al. (2022), and Mhlongo et al. (2015)
Gallic acid	Douglas fir, Black wattle, cocoa pod husk, <i>Mucuna pruriens</i> seed	β -Glucosidase, endo- β -1,4-xylanase	Jönsson et al. (2013), Lu et al. (2018), and Mathibe et al. (2020)
Vanillic acid	Douglas fir, Black wattle, vanilla orchid	Endo- β -1,4-xylanase, α -amylase, α -glucosidase	Alexandre et al. (2022), Gallage and Møller (2015), and Mathibe et al. (2020)
Vanillin	Cocoa powder, vanilla orchid	Endocellulase, exocellulase (cellobiohydrolase), β -glucosidase, amyloglucosidase, xylanase	Gallage and Møller (2015), Hidayatullah et al. (2020), and Sorrenti et al. (2020)
Syringic acid	Grass hay, oak woodchip	α -Amylase, α -glucosidase	Alexandre et al. (2022), and Ziolkowska et al. (2020)
Syringaldehyde	Grass hay, oak woodchip	Endocellulase, exocellulase (cellobiohydrolase), β -glucosidase	Ziolkowska et al. (2020)
Trans-cinnamic acid	<i>Cinnamomum cassia</i> bark (Chinese cinnamon)	Endocellulase, exocellulase (cellobiohydrolase), β -glucosidase	Qin et al. (2016) and Stamogiannou et al. (2021)
Hydroxybenzoic acid	Cocoa powder	Endocellulase, exocellulase (cellobiohydrolase), β -glucosidase	Sorrenti et al. (2020)
Catechin	Tea, cocoa pod husk, cocoa powder, <i>Mucuna pruriens</i> seed	α -Amylase	Lu et al. (2018), Rai et al. (2017), Sorrenti et al. (2020), and Sun et al. (2016a, b)
Oligosaccharides mixture (XOS and GOS)	Bioconversion process	Cellobiohydrolase	Kont et al. (2013)
High concentration of monosaccharides (glucose, galactose, mannose, fructose, xylose)	Bioconversion process	Endoglucanase, cellobiohydrolase, β -glucosidase (only affected by glucose)	Hsieh et al. (2014)

Table 2 Pretreatment techniques for different lignocellulosic biomass

Biomaterials substrate	Pretreatment strategy	Efficiency/yield	Ref
Rice straw	<i>Acid</i> : ammonia, 20.93%; reaction time, 48 h; temperature, 42.74 °C <i>Combination</i> : 2 g/100 g DM xylanase and polyethylene glycol (PEG) 4000	± 13.91 g/L of fermentable glucose (± 87.24%) Glucose yield, 86.9%; xylose yield, 70.2%; and acetone-butanol-ethanol (ABE) yield, 135 g/kg pretreated straw	Kim et al. (2013) Yang et al. (2015a, b)
Sugarcane bagasse	<i>Alkali</i> : NaOH (0.4 M; t, 7 min) <i>Combination</i> : dilute NaOH 1–3% + Commercial Enzyme Primafast 200	Lignin was reduced from 31.71 to 12.07%. The difference between raw bagasse and processed bagasse is 19.64% 22.75–38.84% of saccharification/conversion	Zhu et al. (2016) Thite and Nerurkar (2019)
Sugarcane straw	<i>Combination</i> : alkali + <i>B. safensis</i> M35 <i>Steam explosion</i> : T, 200 °C; P, 15 bar; t, 10 min	15.6% of saccharification/conversion Soluble XOS yields > 35% (w/w) and fermentable glucose yields > ~78	Thite and Nerurkar (2019) Brenellia et al. (2022)
Sago pith	<i>Enzymatic</i> : 0.25 mL cellulose enzyme complex (618 CMC U/g and 139 PNPG U/g); substrate concentration 10% (w/w); t, 24 h	71.36% (w/w sago pith, db)	Pinyo et al. (2016)
Sago pith waste (Hampas)	<i>Enzymatic</i> : 71.4 U/g dextrzyme amylase and 20 FPU/g acromonium cellulase; substrate concentration, 0.09 g/mL <i>Combination</i> : microwave hydrothermal hydrolysis accelerated by carbon dioxide	67.0 g/L fermentable sugar 43.8% glucose and 40.5% ethanol yield	(Husin et al., 2019) Thangavelu et al. (2014)
Eucalyptus chip	<i>Combination</i> : instant controlled pressure drop (DIC) (controlled steam pressure (up to 7 bar) with heat (up to 170 °C) during a short time)	87% (g reducing sugar/100 g pretreated biomass)	Messaoudi et al. (2015)
Aleppo pine cone	<i>Combination</i> : instant controlled pressure drop (DIC) (controlled steam pressure (> 7 bar) with heat (up to 170 °C) during a short time)	74% (g reducing sugar/100 g pretreated biomass)	Messaoudi et al. (2015)
Opium poppy waste stalks	<i>Combination</i> : a combination of DIC (P, 5 bar; t, 540 s) and alkaline extraction (KOH concentration, 22.17%; t, 7 h; V, 53.28 mL)	26.23% hemicellulose extraction yield	Kocabas et al. (2020)
Bamboo	<i>Combination</i> : steam explosion and green-liquor (Na ₂ S + Na ₂ CO ₃)	Yields: hexoses 100.0% (% cellulose), ethanol yield of 40.1% (% cellulose)	Gao et al. (2021)
Poplar	<i>Combination</i> : hydrothermal pretreatment (HP) and acid hydro-tropic pretreatment (AHP)	6.64% of XOS, 46.8% of fermentable sugars (44.2% g of glucose and 2.6% of xylose), and 10.35% of lignin nanoparticles	Zhu et al. (2022)
Acacia wood	<i>Combination</i> : lime (calcium hydroxide) treatment (LT) (T, 70.9 °C, t, 23.5) and hydrothermal treatment (HT) (T, 200 °C; t, 10 min) <i>Acid</i> : 2.75% H ₂ SO ₄ (T, 127.14 °C; t, 74.13 min)	Glucose yields, 73.5%	Lee and Yu (2021)
Durian peel	<i>Combination</i> : ball mill-assisted alkaline peroxide pretreatment	53% reducing sugars	(Panakkal et al., 2021)
Corn Stover		Yields: 69.65% XOS, 20.55% xylose, 68.94% glucose, and 21.15% gluco-oligosaccharides	(Zhang et al., 2021)

to be stored at room temperature for longer periods than standard enzymes. Furthermore, the high-temperature process should avoid microbial contamination during hydrolysis and reduce the risk of contamination and enzyme activity loss during processing. Thermostable enzymes also enable more stable, rapid, and efficient reactions (Giovannoni et al., 2020; Vasconcellos et al., 2015).

Thermostable enzymes are known for their remarkable thermostability, defined as their ability to maintain their functionality and activity under high-temperature conditions without a significant decline in activity over a long period. In particular, thermostable enzymes are essential for efficient lignocellulosic bioconversion and are recommended to withstand hydrolysis temperatures above 50 to 80 °C. Enzymes from hyperthermophilic bacteria, which exhibit optimal conditions ranging from 80 to 110 °C, can be applied. However, it is important to carefully consider the energy and hydrolysis time required, as higher temperatures require higher energy consumption. Moreover, the application of higher temperatures in the process may lead to the degradation or loss of thermolabile compounds (e.g., volatile compound, plant pigment, etc.) (Linares-Pasten et al., 2014; Patel et al., 2019).

Several thermophilic microorganisms have demonstrated their capability to produce thermostable enzymes that can be utilized for the bioconversion of lignocellulosic biomass. For instance, EG can be produced by *Aspergillus terreus* RWY and *Thermobifidia fusca* UPMC 901, with optimal active conditions of 50–60 °C and 4.0–6.0 (Sharma et al., 2014; Zainudin et al., 2019). In the same environment, *Aspergillus terreus* RWY can produce CBH and xylanases (Sharma et al., 2014). Xylanases from *Aspergillus fumigatus* JCM 10253 are stable at 50 °C for 144 h. *Aspergillus fumigatus* JCM 10253 and *Geobacillus* sp. HTA426 also show cellulase activity at 50–60 °C (Saroj et al., 2018). Endoglucanase and endoxylosidase activities are essential for COS and XOS production (Barbarosa et al., 2020; Cano et al., 2020).

Multi-step Process and Enzymatic Hydrolysis Condition to Produce Cello- and Xylo-oligosaccharides

Enzymatic hydrolysis of lignocellulosic material can produce useful compounds like xylo- and cello-oligosaccharides. Both of these items have several health benefits, including prebiotic and antidiabetic properties (Lin et al., 2016; J. ; Zhong et al., 2020). The production of cello- and xylo-oligosaccharides from lignocellulosic biomass can be carried out in a multi-step process. Firstly, the pretreatment can be applied to the lignocellulosic biomass to destroy the lignin stealth, reducing the crystallinity of

cellulose, and also break down the hemicellulose (Baruah et al., 2018).

In pretreatment I, hydrothermal pretreatment can be used to break down the crystalline structure of the lignocellulosic complex and to degrade hemicellulose. There has been no significant reduction in total lignin at this point, but there has been a change in the soluble and insoluble lignin fractions. At this point, XOS is also being produced (Xue Chen et al., 2016; Marcondes et al., 2020; Santo et al., 2018).

Under ideal circumstances, hydrothermal pretreatment has been shown to generate XOS in the range of 21.3 to 80.40% with minimal production of byproducts (Liqin Qin et al., 2022; Wang et al., 2021a, b; Zhang et al., 2022). The temperature range used for hydrothermal pretreatment varies from 150 to 215 °C, with a duration of 5.5 min to 2 h. The most commonly used temperature for hydrothermal pretreatment is around 160 °C, which takes into account energy consumption and prevents further degradation of compounds that could occur at higher temperatures (Capetti et al., 2023; Neto et al., 2020; Xiong et al., 2023). The determination of optimal conditions is dependent on the specific type of biomass being utilized (Table 4).

For instance, Marcondes et al. (2020) carried out hydrothermal pretreatment under optimum conditions at 182 °C for 5.5 min in the absence of an external catalyst to produce $\pm 43.61\%$ XOS and $\pm 2.26\%$ xylose. The XOS and xylose produced from this step accumulated in the liquid fraction, while the solid fraction still consists of cellulose, xylan, and lignin. In another recent study, Xiong et al. (2023) produced XOS from bamboo by using hydrothermal pretreatment at 160 °C and 120 min with the addition of 0.2 M formic acid, resulting in approximately 68.04% of initial xylan present.

Santo et al. (2018) observed physical changes in the lignocellulosic structure during hydrothermal pretreatment. The hydrothermal pretreatment separated the fibers on the bundle surface from the others, but there was no significant difference in total lignin. Images from field emission scanning electron microscopy (FESEM) and confocal laser scanning microscopy (CLSM) show that lignin is redistributed and that no lignin accumulates on the surface. This is due to variations in the soluble and insoluble lignin fractions (Fig. 1).

The solid phase is recommended to undergo delignification (pretreatment II) after hydrothermal pretreatment to eliminate lignin and obtain a cellulose-rich solid with a minimal amount of lignin. This process aims to enhance enzymatic hydrolysis by reducing inhibitors (Lei Qin et al., 2016; Sun et al., 2016a, b; Zoghliami & Paës, 2019). Alkaline pretreatment is a preferable method to eliminate a significant amount of lignin without producing unwanted inhibitor byproducts (Martín et al., 2022; Jinyu Tan et al., 2021).

Table 3 Potential microorganisms for the production of thermostable enzymes

No	Microbes	Fermentation condition	Enzymes	Enzyme activity	Enzyme optimum condition		Ref
					pH	Temperature	
1	<i>Aspergillus terreus</i> RWY	Solid state; 45 °C for optimal production of enzyme	<i>Cellulases</i> and <i>Xylanases</i>	<i>Cellulases</i> (Filter paper cellulase (FP) (11.3 ± 0.65 U/g-ds), endoglucanase (EG) (103 ± 6.4 U/g-ds), β-glucosidase (BGL) (122.5 ± 8.7 U/g-ds), cellobiohydrolase (CBH) (10.3 ± 0.66 U/g-ds)), <i>Xylanases</i> (xylanase (872 ± 22.5 U/g-ds), β-xylosidase (22.1 ± 0.75 U/g-ds), α-L-arabino-furanosidase (126.4 ± 8.4 U/g-ds), and xylan esterase (907 ± 15.5 U/g-ds))	4.0–6.0	50–60 °C	Sharma et al. (2014)
2	<i>Geobacillus</i> sp. HTA426	Isolated from hot spring district; optimal enzyme production at 60 °C for 72 h	<i>Cellulase</i>	CMCase activity, 103.67 U/mL	7.0	Optimum 60 °C; stable for 5 h incubation at range 50–70 °C	Potprommanee et al. (2017)
3	<i>Aspergillus fumigatus</i> JCM 10,253	Isolated from Warangal district, India. Solid-state fermentation (SSF) at 50 °C for 7 days	<i>Extracellular lignocellulolytic enzyme isolate</i>	CMCase (26.2 IU/mL), FPase (18.2 IU/mL), β-glucosidase (0.87 IU/mL), and xylanase (2.6 IU/mL) (incubation time of 144 h at 50 °C)	–	60 °C for crude cellulase and 50 °C for FPase, β-glucosidase, and xylanase	Saroj et al. (2018)
4	<i>Thermobifidia fusca</i> UPMC 901	Isolated from composted oil palm empty fruit; fermented on modified tryptic soy medium at 50 °C for 24 h	<i>Endoglucanase</i>	CMCase: 0.9 U/mL (pH 5; T: 60 °C);	5.0	Thermal stability at 70 °C (t: 24 h) and 50–60 °C (t: 144 h)	Zainudin et al. (2019)

Table 4 Hydrothermal pretreatment strategy for producing XOS

No	Biomass	Temperature	Time	Other condition	Efficiency	Ref
1	Sugarcane bagasse	182 °C	5.5 min	<i>Solid/liquid ratio</i> , 1:11 (w/v); <i>agitation speed</i> , 120 rpm in 8L-Parr Reactor	43.61% XOS and 2.26% xylose	Marcondes et al. (2020)
2	Sugarcane bagasse	160 °C	30 min	<i>Solid/liquid ratio</i> , 1:10 (g/mL); <i>pressure</i> , 7 Bar	79.2% cellulose, 5.2% hemicellulose, and 10.9% lignin	Santo et al. (2018)
3	Jiuzao	181.5 °C	20 min	<i>Solid/liquid ratio</i> , 1:13.6 (g/mL); <i>particle size</i> , > 20 mesh	21.3% XOS	Qin et al. (2022)
4	Banana pseudostem pulp	Up to 150 °C	–	<i>Solid/liquid ratio</i> , 1:11 (w/v); non-isothermal condition	47.39% glucan, 10.43% xylan, 4.54% arabinan, and 14.41% Klason lignin; enzymatic digestibility 78.62%	Díaz et al. (2021)
5	Corn stover	215 °C	1.5 h	<i>Solid/liquid ratio</i> , 1:20 (w/v); combined with mechanical pretreatment (ball-mill and ultrasound)	80.40% XOS (26.97% for total xylotetraose, xylotri-ose, and xylobiose)	Zhang et al. (2022)
6	Chinese hickory shell	160 °C	2 h	<i>Solid/liquid ratio</i> , 1:10 (w/v)	55.3% XOS with the low formation of by-product	Wang et al. (2021a, b)
7	Corn cob	160 °C	60 min	<i>Solid/liquid ratio</i> , 1:10 (w/v); <i>pressure</i> , 73 Psi; in 1L-Parr Reactor	16.7% xylan and 30% lignin	Capetti et al. (2023)
8	Bamboo	160 °C	120 min	<i>Solid/liquid ratio</i> , 1:10 (w/v); addition of 0.2 M formic acid	68.04% XOS from initial xylan	Xiong et al. (2023)
9	<i>Eucalyptus</i> by-product	161 °C	65 min	<i>Solid loading</i> , 10% (w/v); <i>agitation speed</i> , 4 rpm in 1.5 L steel reactor	60 mg XOS/g <i>Eucalyptus</i> by-product	Neto et al. (2020)
10	Poplar	170 °C	50 min	<i>Solid/liquid ratio</i> , 1:10 (w/v)	Delignification: 24.9%; yield, 35.4% XOS (DP 2–6) and 17.6% xylans (DP > 6)	Su et al. (2021)

Under optimal conditions, alkaline pretreatment can also recover a specific amount of xylan (35–89.4%) and lignin (14.2–92.6%) (Capetti et al., 2023; Sun et al., 2016a, b; Wang et al., 2021a, b). The liquid residue obtained from this process contains a significant amount of lignin, which can be utilized to manufacture various materials such as adhesives, composites, and carbon fibers. Additionally, the solid fraction that is rich in cellulose can be utilized as a substrate for the production of COS (Capetti et al., 2023; Neto et al., 2020; Sun et al., 2016a, b). The yield obtained from alkaline pretreatment may differ based on the type of biomass used and the specific conditions employed during the pretreatment process (Table 5).

For instance, a combination of alkaline sulphonation and steam pretreatment was applied by Chu et al. (2018) to eliminate approximately 69.37% lignin. Khat et al. (2018) found that the optimal conditions for alkaline pretreatment to recover XOS were 12–18% alkaline concentration, 110–120 °C temperature, and 37.5–40 min of steaming time. This process significantly increases cellulose accessibility

(Ávila-Lara et al., 2015; Chu et al., 2018; Yuansah et al., 2019) (Fig. 2).

Sun et al. (2016a, b) investigated the alkaline treatment of rice straw to extract lignin and obtain cellulose-rich substrates. The treatment involved using 1.5% aqueous NaOH at a temperature of 80 °C for a duration of 3 h with a solid-to-liquid ratio of 1:10 (g/mL). As a result, they obtained post-treated substrates that were rich in cellulose. The lignin yield was 14.2 g, which had a purity of 99.2%, and 30.86 g of cellulose-rich solid was obtained per 100 g of rice straw.

Delignified cellulose-rich solids can be hydrolyzed at this stage to produce COS using thermostable enzymes. The residual xylan may still be present in the solids, leading to the formation of XOS. Thermostable enzymes are preferred because they reduce the risk of contamination, are more stable for longer hydrolysis times, and may result in a lower hydrolysis charge (Giovannoni et al., 2020). Endoglucanase isolated from *Thermobifidia fusca* UPMC901 has been reported to be active for up to 144 h at a temperature of 50–60 °C without loss of activity (Zainudin et al., 2019).

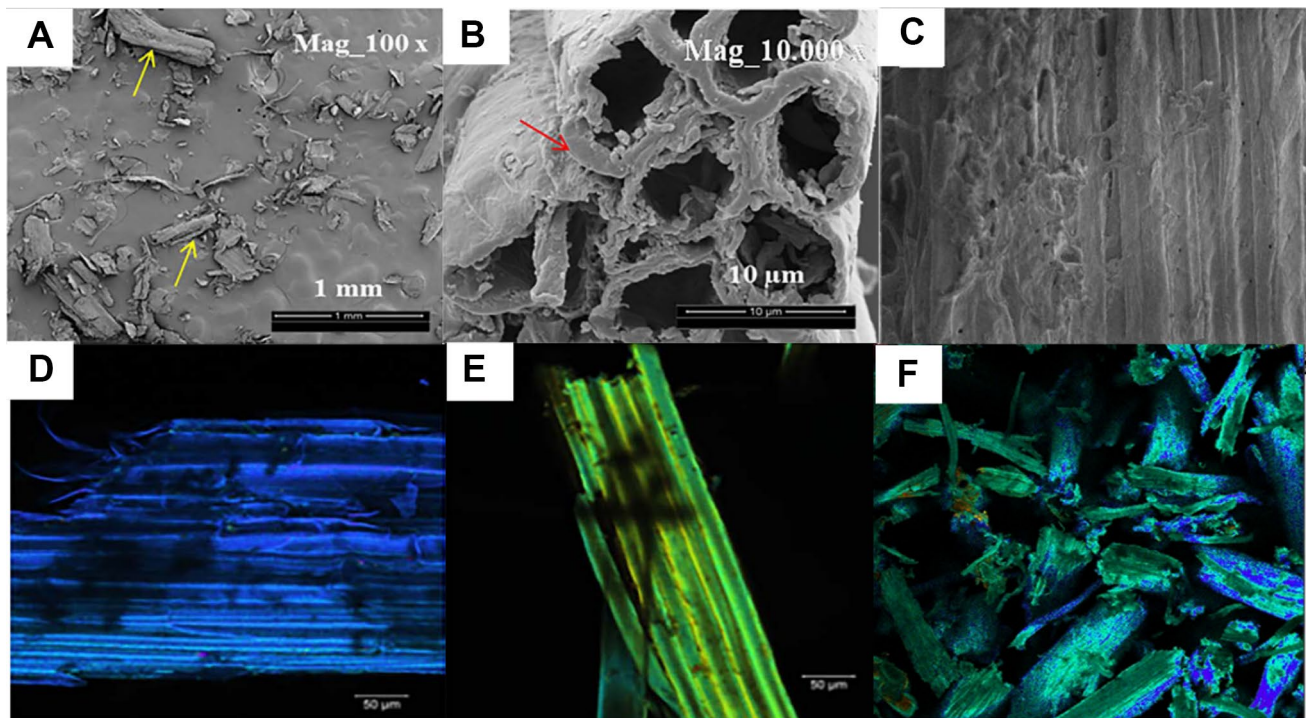


Fig. 1 SEM imaging of **a** untreated lignocellulose structure; **b** hydrothermal pretreatment, in which fibers become more separated from the surface of the fiber bundle; and **c** alkaline pretreatment, in which lignin is dissolved and pores are increased. CLSM imaging of **d**

untreated lignocellulose structure; **e** hydrothermal-pretreated lignocellulose; and **f** alkaline-pretreated lignocellulose (Ávila-Lara et al., 2015; Santo et al., 2018)

Various strategies can be employed to achieve optimal yields of COS, and these may vary depending on the biomass characteristics and pretreatment methods used before entering this stage. Some approaches involve using enzymes simultaneously, taking advantage of their synergistic effects, while others adopt a sequential approach, taking into account potential product inhibition (Barbarosa et al., 2020; Chu et al., 2014; Díaz et al., 2021; Kendrick et al., 2022) (Table 6).

For example, Ávila and Goldbeck (2022) added thermostable enzymes simultaneously, including endo-1,4- β -glucanase (GH12) from *Aspergillus niger*, exo-1,4- β -glucanase (GH5) from *Trichoderma longibrachiatum*, and feruloyl esterase (CE1) from *Clostridium thermocellum*, to hydrolyze sugarcane straw, resulting in a COS yield of 67.60 mg/g of substrate and 4.17 mg of glucose/g of substrate. Similarly, Chu et al. (2014) utilized cellulase C2730 to hydrolyze sugarcane straw and obtained a COS yield of 51.78%. Another method involves stepwise enzymatic hydrolysis, as demonstrated by Li et al. (2019), who separated the products after bioconversion carried out sequentially by xylanase and cellulase, resulting in a total conversion of cellulose of 91.51%. Separation of products is crucial in this process, as the hydrolyzate of xylanase conversions, such as XOS and its derivatives, may inhibit the cellulases.

Therefore, removing the product from the system allows the cellulase activity to proceed unhindered.

The performance of cellulase mixtures can also be improved by separating β -glucosidase (BG) from EG and CBH. The process can be accomplished by using thermostable cellulases and carrying out the “adsorption–separation” method. This involves separating the β -glucosidases from the EGs and CBHs at a temperature of 10 °C and a pH of 7.0. The retention of CMCase is improved, while the retention of β -glucosidase is decreased by adjusting the pH close to the *pI* of the BGs, which is 8.7. The BGs can be separated and accumulated in the supernatant during centrifugation. Once β -glucosidase is separated from cellulases, the process can be performed at elevated temperatures up to 50–60 °C to optimize the activity of endoglucanase. The hydrolysis process involves three stages and can improve the total COS yield accumulated in each step by approximately 51.78%, with hydrolysis reaching approximately 75.56%. This method was reported by Chu et al. (2014) (Fig. 3).

This multi-step process using thermostable enzymes has the potential to improve the yield of COS and XOS production, decrease the use of enzyme concentration, have a quick hydrolysis time, and eliminate the risk of contamination. Furthermore, the gradual separation of products in the system in a multi-step process minimizes the inhibition of

Table 5 Alkaline treatment strategy for producing XOS and cellulose-rich solid

No	Biomass	Alkaline concentration	Time	Other condition	Efficiency	Ref
1	Sugarcane straw	24% (w/v) KOH	180 min	Solid loading, 8%; T, 35 °C	Hemicellulose solubility, 82.18%; delignification, 91.18%	Ávila and Goldbeck (2022)
2	Corn cob	1% (w/v) NaOH	40 min	<i>Solid/liquid ratio</i> , 1:10 (w/v); T, 121 °C	35% xylan and 8.3% lignin	Capetti et al. (2023)
3	Rice husk	12% (w/v) NaOH	30 min	<i>Solid/liquid ratio</i> , 1:10 (w/v); T, 133.69 °C	54.49% of xylan recovered	Khat-udomkiri et al. (2020)
4	Sugarcane bagasse	1% (w/v) NaOH	1 h	<i>Solid/liquid ratio</i> , 1:20 (w/v); T, 100 °C; ω , 80 rpm	Partially solubilized lignin from 40 to 19% and residual hemicellulose is not detected	Marcondes et al. (2020)
5	Sugarcane bagasse	1% (w/v) NaOH	6 h	<i>Solid/liquid ratio</i> , 1:20 (w/w); T, 70 °C; addition of 1.5% H ₂ O ₂	Klason lignin removal, 84.45%; cellulose recovery, 91.91%; xylan recovery, 61.87% (low loss of valuable polysaccharides)	Li et al. (2019)
6	Corn stalk	8% (w/v) NaClO ₂	2 h	<i>Solid/liquid ratio</i> , 1:5 (w/v); T, 70 °C	Delignification, 92.6%; residual lignin, 2%; hemicellulose, 83.3%	Wang et al. (2021a, b)
7	Corn stover	2% (w/v) NaOH in methanol	1 h	<i>Solid/liquid ratio</i> , 1:2 (w/w); T, 80 °C	Delignification, > 70%; glucan recovery, 97.4%; xylan recovery, 89.4%	Gong et al. (2020)
8	<i>Miscanthus sinensis</i>	0.9% (w/v) NaOH	10 min	<i>Solid loading</i> , 10% (w/v); T, 121 °C; addition of 1% surfactant PEG 2000	Lignin significantly decreased in pre-treated <i>Michantus</i> , 15.2%	Cheng et al. (2022)
9	<i>Nothofagus pumilio</i> sawdust	2% (w/v) NaOH	20 min	<i>Solid/liquid ratio</i> , 1:20 (w/v); T, 121 °C; P, 1 atm	Delignification, 87%; xylan recovery, 62%	Lehuedé et al. (2023)
10	Rice straw	1.5% (w/v) NaOH	3 h	<i>Solid/liquid ratio</i> , 1:15 (w/v); T: 80 °C;	Yields, 14.20 g lignin/100 g rice straw (liquid) and 30.8 g cellulose/100 g rice straw (solid)	(Sun et al., 2016a, b)

enzyme action due to product accumulation, allowing for a higher total yield of the desired product (Chu et al., 2014; Patel et al., 2019). Chu et al. (2014) developed a strategy for COS production using a cellulase enzyme-deficient BG by performing the “adsorption–separation” method and multi-step enzymatic hydrolysis repeated three times. The product from each stage is separated from the process to avoid enzyme inhibition. The hydrolysis scheme gradually hydrolyzed the substrate over 6 h, 6 h, and 12 h, yielding COS of 20.40%, 15.53%, and 15.85%, respectively, for a total yield of 51.78%.

Because a large portion of COS will be produced through enzymatic processes, cellulase inhibitors such as xylan and its derivatives must be separated at each stage to achieve optimal yields. This product can be made using

a multi-step enzymatic hydrolysis process. Unlike COS, XOS will accumulate during pretreatment stages I, II, and the first stage of enzymatic hydrolysis (Fig. 4). Product separation at each stage of multi-step enzymatic hydrolysis keeps XOS from being hydrolyzed further to the monosaccharide xylose. Several studies from Hao et al. (2022) and Zhang et al. (2021) used xylanase–cellulase enzymes in multi-step enzymatic hydrolysis. According to the findings, XOS was only detected in the first step of enzymatic hydrolysis. Further hydrolysis revealed only monosaccharide accumulation (Table 6). In order to separate the soluble oligosaccharide-rich products from multiple stages, various purification methods were employed to collect and isolate COS, XOS, sugars, and other compounds such as phenolic compounds and lignin derivatives (Fig. 4).

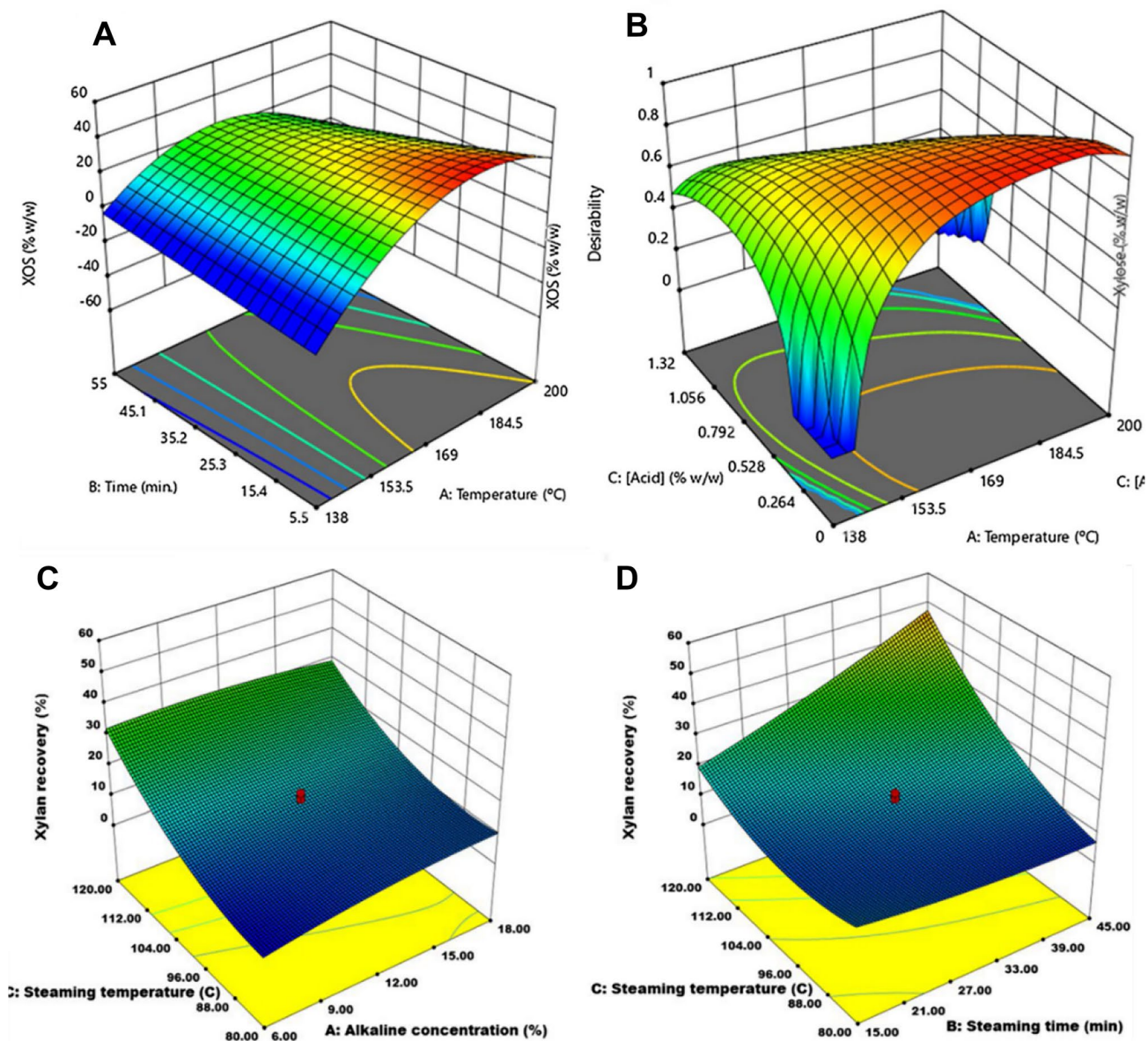


Fig. 2 Hydrothermal pretreatment response surface for XOS yields **a** in the absence of acid, **b** for XOS maximum and xylose minimum yields. The alkaline pretreatment response surface is affected by **c** the

effect of steam temperature and NaOH concentration and **d** steaming temperature and time (Khat et al., 2018; Marcondes et al., 2020)

Purification Strategies for XOS and COS

The purification of XOS and COS products is a crucial step in producing high-quality and pure products, as impurities can negatively impact their quality. The success of this stage can determine the selling value of the product. Strategies such as activated carbon, filtration, and columns are commonly used to remove color, protein, and salt, and collect XOS, COS, sugar, phenolic, and lignin (Jiang et al., 2021; Kim et al., 2022; Wang et al., 2021a, b; Wijaya et al., 2020) (Table 7).

For instance, Jiang et al. (2021) utilized multi-stage purification to purify products obtained from the enzymatic hydrolysis of bamboo. They achieved a decolorization efficiency of 53.3% from activated carbon, 77.7% for deproteinization, and a sugar loss of 2% from ion exchange. Furthermore, they attained an 80% desalination rate, a 7.1% sugar loss rate, and an 85.7% decolorization rate from ion exchange. Ultrafiltration resulted in an XOS recovery rate of 93.3%, with 9.4% of sugar loss and a decrease in monosaccharides from 14.6 to 6.7%.

Table 6 Enzymatic hydrolysis strategies for COS and XOS production

No	Substrate	Enzymes	Specification	Hydrolysis condition	Efficiency	Ref
1	Jiuzao	Xylanase	XynAR expressed in recombinant <i>E. coli</i>	T, 60 °C; Ph 5; enzyme loading, 15 U/g; t, 2 h	XOS yields, 34.2%	Qin et al. (2022)
2	Corn stover	Xylanase	Xylanase from <i>Trichoderma viride</i> ; 4529 U/g (Aladdin)	[S]: 2% (w/v, g/mL) pretreated corn stover; [E]: Xylanase 100 IU/g substrate; pH 5; T: 50 °C; ω, 800 rpm; t, 24 h	Xylan conversion, 84% (Including XOS, xylotri-ose, xylobiose, and xylose as products); and 28.57% cellulose conversion	Zhang et al. (2021)
3	Sugarcane straw	Cellulase Processive Endoglucanase Endoglucanase Lytic polysaccharide monoxy- genases (LPMO) Cellobiose dehydrogenase (CDH)	Cellulase from <i>Trichoderma reesei</i> ; 238 FPU/g (Sigma-Aldrich) CaCel9R from <i>Clostridium thermocellum</i> F7 CeCel9M from <i>Clostridium cellulolyticum</i> ATCC 35319 TtCel61A from <i>Trichoderma reesei</i> NcCDH1a from <i>Neurospora crassa</i> OR74A	[E]: Cellulase 25 FPU/g substrate; pH 5; T: 50 °C; ω: 800 rpm; t: 72 h Enzyme loading (All enzymes), 10 U/g substrate; [S]: 1% (w/v); pH 5; T: 50 °C; ω, 800; t, 48 h; additives: lactose and copper	62.49% cellulose conversion and 6.18% xylan conversion 60.49 mg COS/gram-pretreated sugarcane straw	Barbarosa et al., 2020)
4	Banana Pseudostem pulp	Cellulase	Cellulase from <i>Trichoderma reesei</i> (BG activity 19 U/mL; EG activity 2138 U/g; 54 FPU/g (Sigma-Aldrich)	5% (w/v) db substrate loading; enzyme loading: 1 FPU/g db; pH 4.8; T: 50 °C; ω, 150; t, 12 h; additives: sodium azide (0.02% w/v) (antimicrobial)	27.87% COS and 10.62% glucan	Díaz et al. (2021)
5	Asparagus fiber	Cellulase	Cellulast® CCN03187 from <i>trichoderma reesei</i> ; 11.7 µkat/g (Novozymes)	Solid loading, 25 g/L; [E]: 700 nkat/g substrate; T, 50 °C; ω, 900; t, 7 h	<i>Lab-scale</i> : total COS, 37.2 g/100 g cellulose; glu-cose, 8.14 g/100 g; cellobi-ose, 25.3 g/100 g; cellotri-ose, 2.53 g/100 g; and cellodextrin (DP > 4), 1.2 g/100 g <i>Bench-scale</i> : total COS, 44.6 g/100 g cellulose; glu-cose, 10.9 g/100 g; cellobi-ose, 30 g/100 g; cellotri-ose, 3.3 g/100 g; and cellodextrin (DP > 4), 1 g/100 g	Siccama et al. (2022)

Table 6 (continued)

No	Substrate	Enzymes	Specification	Hydrolysis condition	Efficiency	Ref
6	Michantus	Processive endoglucanase Classic endoglucanase	TtCel9a from <i>Thermobifida fusca</i> expressed in <i>P. pastoris</i> NRRL 11,430 CcCel9m from <i>Clostridium cellulolyticum</i> ATCC 35319 expressed in <i>P. pastoris</i> NRRL 11430	Enzyme loading, 150 CMCase U/g solid for TtCel9a, CcCel9m, and OsCel7a(-105) and 5 mg/g solid for TrCel61a; substrate loading, 1% (w/v); T, 50 °C; pH 5.5; t, 72 h	Conversion of glucan to COS with DP 2–5, 90%	Kendrick et al. (2022)
7	Corncob	Cellobiohydrolase (CBH) Lytic polysaccharide monoxygenases (LPMO)	OsCel7a(-105) from <i>Orphiomyces</i> sp. In 02 expressed in <i>P. pastoris</i> NRRL 11,430 TtCel61a from <i>Trichoderma reesei</i> expressed in <i>P. pastoris</i> NRRL 11,430	Enzyme loading, 10,000 nkat/g DM; pH 5; T, 50 °C; t, 24 h	Yield, 58.3 g/1 kg corncob	Hao et al. (2022)
8	Corncob	Xylanase Cellulase Cellulase	Xylanase from <i>Nonomurata flexuosa</i> (GH1) expressed in <i>Trichoderma reesei</i> ; 11,726 nkat/mL Cellic CTec2; 123 FPU (Novozymes) Cellulase C2730 (Sigma); FPase 101.37 FPU; CMCCase (EG 805.59 U/g) and EG 38.20 U/g	Enzyme loading, 10 mg/g DM; pH 5; T, 50 °C; t, 72 h Substrate loading, 5%; enzyme loading, 15 FPU/g cellulose; pH 4.8; T, 50 °C; ω, 150 rpm; t, 6+6+12 h (product separated every stage)	Yield, 79.1% glucose and 22.3% xylose Total COS, 51.78%	Chu et al. (2014)
9	Rice husk	Xylanase	1,4-β-Xylanase Pentopan™ MonoBG (Novozymes)	Enzyme loading, 6.25 mg/g xylan; pH 6; T, 50 °C; t, 72 h	Yields, 17.5 mg XOS/mL xylan	Khat et al. (2018)
10	Corncob	Xylanase	PxXyn10A (GH10) from <i>Xanthomonas axonopodis</i> + PxXyn11B (GH11) from <i>Geobacillus stearothermophilus</i>	Substrate loading, 2% (w/v) solid fraction; enzyme loading, 125 μg/mL (GH10:GH11 ratio = 2:1); pH 6; T, 50 °C; ω, 150 rpm; t, 48 h	41–59 mg XOS/g initial substrate	Capetti et al. (2023)
		Xylanase	PxXyn10A (GH10) from <i>Xanthomonas axonopodis</i> + MetXyn (GH11) from <i>Trichoderma reesei</i>	Substrate loading, 38% (v/v) liquid fraction; enzyme loading, 125 μg/mL (GH10:GH11 ratio = 1:1); pH 6; T, 50 °C; ω, 150 rpm; t, 48 h	73.5 mg XOS/g initial biomass	

Table 6 (continued)

No	Substrate	Enzymes	Specification	Hydrolysis condition	Efficiency	Ref
11	Sugarcane straw	Combination	Endo-1,4- β -glucanase (GH12) from <i>Aspergillus niger</i> + Exo-1,4- β -glucanase (GH5) from <i>Trichoderma longibrachiatum</i> + feruloyl Esterase (CE1) from <i>Clostridium thermocellum</i>	Substrate loading, 0.1% (w/v); enzyme loading, 1.75:1:1.34 (mg/g) (GH12:GH5:CE1); pH 5; T, 50 °C; ω : 1000 rpm; t, 48 h	67.60 mg COS/g substrate and 4.17 mg glucose/g	Ávila and Goldbeck (2022)
12	Sugarcane bagasse	Combination	Xylanase (SUNSON Industry Group Co., Ltd) and Cellulase (Imperial Jade Bio-Technology Co., Ltd)	Substrate loading, 2% (w/v); enzyme loading: xylanase, 300 IU/g; cellulase, 25 FPU/g; pH 5; T, 50 °C; ω , 800 rpm; additives, sodium azide (0.02% w/v)	Cellulose conversion: 91.51%	Li et al. (2019)

In a recent investigation, Kim et al. (2022) conducted a multi-step purification process that involved peroxidase-mediated polymerization to remove 50.2% of the phenolic compounds. After that, diafiltration was used to eliminate 72.8% of the phenolic compounds while only incurring an XOS loss of 4.1%. The study also concluded that activated carbon is not a suitable method for XOS purification since it tends to absorb more xylotri- and xylobiose than xylose.

Bioactivities of Cello- and Xylo-Oligosaccharides

COS can act as prebiotics by promoting the growth of good microbiota in the gut, such as *Clostridium butyricum*, *Lactococcus lactis*, *Lactobacillus paracasei*, and *Lactobacillus rhamnosus*, whereas XOS stimulates the bacterial groups bifidobacilli and lactobacilli (Khat-udomkiri et al., 2020; Zhong et al., 2020).

Zhong et al. (2020) conducted a study that illustrated the prebiotic effect of COS with DP 3–6. The result showed a loss of the insoluble fraction by ≤ 10 mol% and an increase in the cell density for *C. butyricum*, *L. lactis*, *L. paracasei*, and *L. rhamnosus*, up to 4.1-fold. In another study, Lin et al. (2016) demonstrated that XOS supplementation in humans significantly increased the growth of *Bifidobacterium* and lactobacilli. Specifically, *Bifidobacterium* spp. increased by more than 10 Log CFU/g feces, and *Lactobacillus* spp. increased by more than 8 Log CFU/g feces.

The gut microbiota can produce SCFAs through the fermentation of undigested or partially digested polysaccharides in the large intestine, particularly COS and XOS. In a study by Takagi et al. (2016), prebiotics were used to stimulate the *Bifidobacterium* group of bacteria in the human gut, and it was found that XOS led to a significant increase in acetate production of approximately 1.23 mM. Another study by Karnaouri et al. (2019) investigated the prebiotic potential of a COS-rich hydrolyzate from spruce against *L. plantarum*. The results showed a significant increase in acetic acid by 0.6% and propionate acid by 0.1%, while butyric acid and formic acid did not experience significant changes.

Most bacteria produce SCFAs through the glycolytic pathway, while some bacteria, such as Bifidobacteria, can produce them through the pentose phosphate pathway. In addition, certain bacteria, such as acetogens, can produce SCFAs via other pathways, such as the Wood–Ljungdahl pathway for acetate (C2) production (Jian Tan et al., 2014). The succinate pathway is the dominant route for the production of fecal propionate (C3), which has been linked to the relative abundance of Bacteroidetes in the gut microbiota. Butyrate producers (e.g., *Faecalibacterium*, *Eubacterium*, and *Roseburia*) predominantly use the butyryl-CoA:acetate CoA-transferase pathway to produce butyrate (C4). Interestingly, only a few anaerobes, such as *Roseburia inulinivorans*

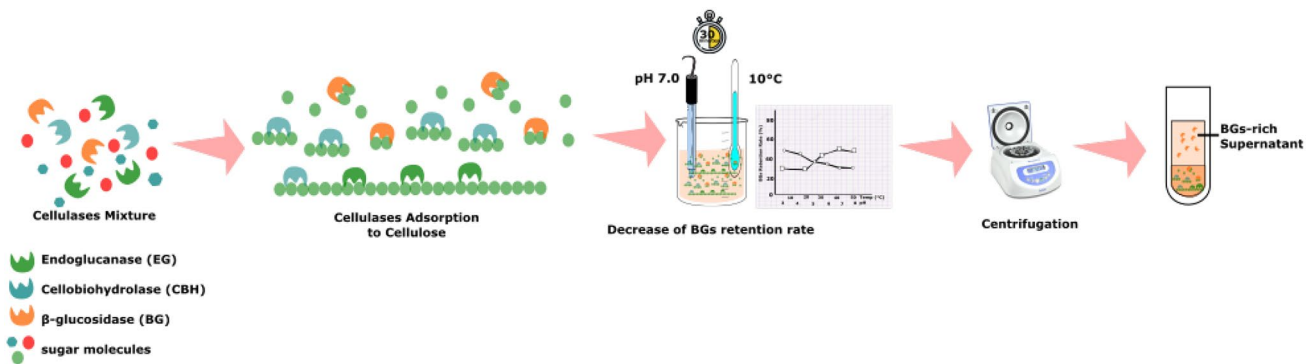


Fig. 3 Schematic diagram of adsorption–separation method developed by Chu et al. (2014)

and *Coprococcus catus*, can produce both SCFAs (Ríos-Covián et al., 2016).

COS and XOS fermentation by gut microbiota lead to an increase of SCFAs in the human intestine, which play an important anti-inflammatory role by increasing immune cell chemotaxis and acting as an antimicrobial by disrupting osmotic and pH balance (Jian Tan et al., 2014). XOS also has antidiabetic activity in type 2 diabetic rats by regulating blood glucose levels and increasing glucose tolerance (Khat-udomkiri et al., 2020) (Fig. 5).

Khat-udomkiri et al. (2020) conducted a study to investigate the potential antihyperglycemic effect of XOS derived from rice husk. The study findings indicated that XOS reduced the cecal pH in diabetic and control rats. Additionally, XOS supplementation increased the levels of SCFAs in the cecal content of normal and diabetic rats, particularly propionate and butyrate. These increases in AMPK phosphorylation and GLUT4 protein expression lead to improved glucose uptake and reduced triglyceride concentration. According to the findings, the modulation of endotoxemia

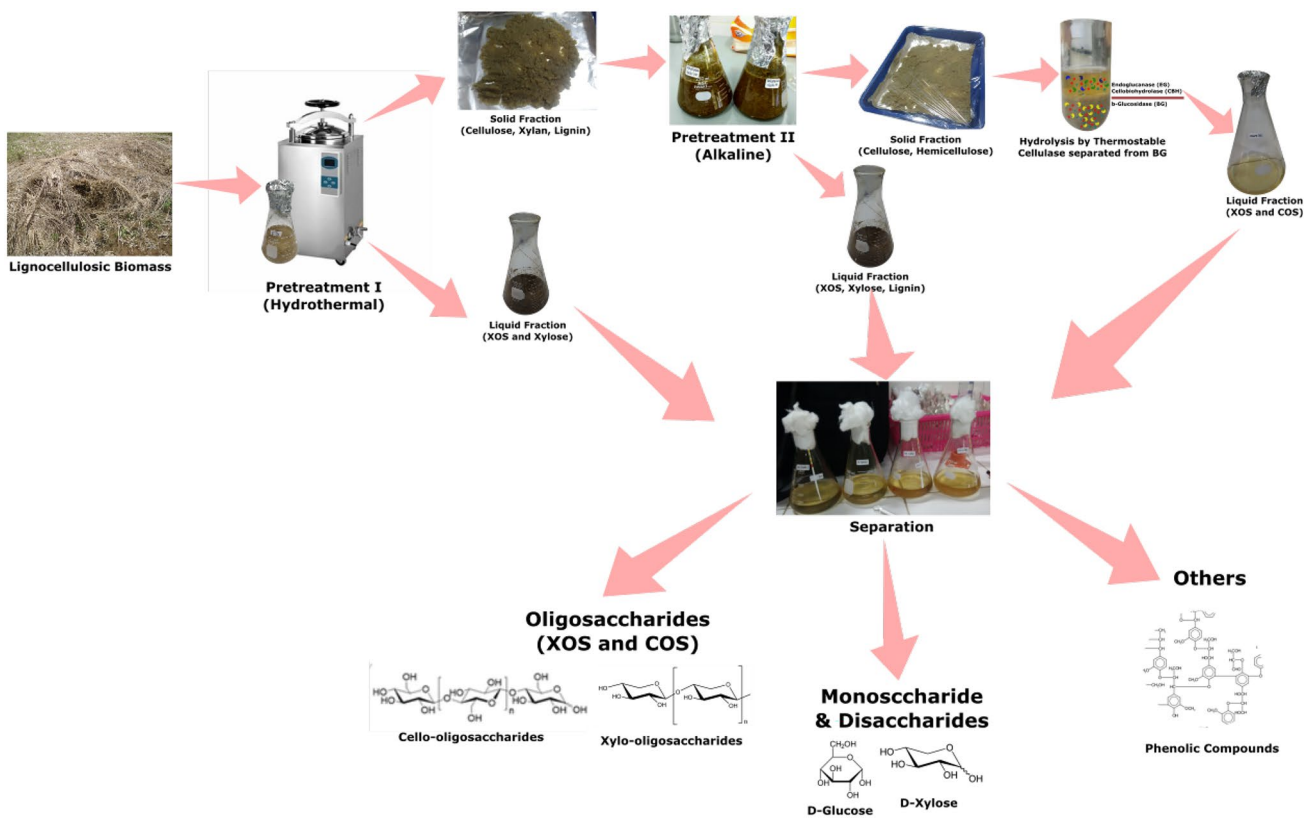


Fig. 4 Schematic diagram of multi-step enzymatic hydrolysis strategy for COS and XOS production

Table 7 Potential purification strategies for XOS and COS

Biomass	Purification strategy	Purification efficiency	Ref
Bamboo	Activated carbon (0.5%)	Decolorization, 53.3%	Jiang et al. (2021)
	Ultrafiltration (10 kDa filter)	Sugar loss rate, 2%; deproteinization, 77.7%	
	Ion exchange (amberlite, 732 strong acid cation exchange resin 0.096 (v/v) and amberlite FPA53 anion exchange resin, 0.090 (v/v))	Desalination rate, 80%; sugar loss rate, 7.1%	
	Nanofiltration (100–300 Da concentration ratio, 9.1)	XOS recovery, 93.3%; sugar loss rate, 9.4%; monosaccharide decrease from 14.6% to 6.7%	
Palm oil empty fruit bunch	Peroxidase-mediated polymerization (peroxidase 40 U/mL and 0.1% H ₂ O ₂ at pH 7; T, 25 °C)	Phenolic compound removal rate, 50.2%	Kim et al. (2022)
	Diafiltration with ultrafiltration membrane MW limit 1 kDa and Diameter, 4 cm using stirred cell	Phenolic compound removal rate, 72.8%; XOS loss rate, 4.1%	
	Nanofiltration NTR-7450	Recovery rate, 42.4%; maximum value of xylobiose, 90.1%	Wijaya et al. (2020)
Corn stalk	Activated carbon powder (4% (w/v))	XOS purity, 96.3%; XOS yield, 77.4%; color value, 814	Wang et al. (2021a, b)
<i>Northofagus pumilio</i> sawdust	Stirred ultrafiltration cell (Ultracel 10 kDa, 200 rpm)	NaOH pretreatment residue removal, 2%; extracted xylan, 62.5% (no loss during pretreatment)	Lehuedé et al. (2023)

and improvement of gut dysbiosis could be the primary mechanism underlying XOS's antihyperglycemic effect.

Market Potential and Commercial Availability of XOS and COS

The market for prebiotics, including XOS and COS, is expanding due to the growing awareness of gut health and the benefits of a healthy gut microbiome. According to Amorim et al. (2019), the global prebiotics market is expected to reach US\$7.37 billion by 2023, with a compound annual growth rate of 10.4% from 2020 to 2025. The market potential for XOS and COS is specifically driven

by their prebiotic properties, which can improve digestion, enhance immune function, and promote the growth of beneficial gut bacteria (Khat-udomkiri et al., 2020; Jian Tan et al., 2014; Zhong et al., 2020).

XOS is available in various commercially viable forms, including powders, supplements, prebiotic foods, and drinks, owing to their production from various sources, such as agricultural waste, forestry waste, and food processing waste. However, the high cost associated with COS production has resulted in a limited and highly specialized market, primarily for analytical applications. Some common trade names for XOS and COS, including xylobiose, xylotriose, xylotetraose, xylopentaose, xylohexaose, cellobiose, cellotriose, cellotetraose, cellopentaose, and cellohexaose, were provided

Fig. 5 Bioactivity potential of COS and XOS

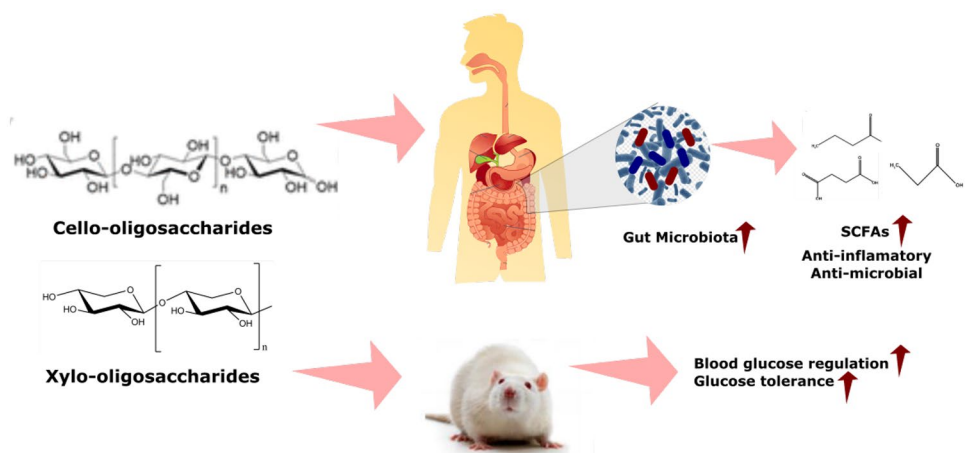


Table 8 Prices of commercial XOS and COS

Product	Provider	Purity	Price
Xylobiose	Megazyme, Ireland	> 95%	US\$3.91/mg
Xylobiose	Sigma-Aldrich, US	≥ 90%	US\$22.52/mg
Xylotriose	Megazyme, Ireland	> 90%	US\$3.91/mg
Xylotetraose	Megazyme, Ireland	> 95%	US\$6.52/mg
Xylopentaose	Megazyme, Ireland	> 95%	US\$16.06/mg
Xylohexaose	Megazyme, Ireland	> 95%	US\$21.27/mg
Cellobiose	Sigma-Aldrich, USA	≥ 98%	US\$6.51/g
Cellotriose	Megazyme, Ireland	> 90%	US\$3.96/mg
Cellotetraose	Megazyme, Ireland	> 95%	US\$4.19/mg
Cellotetraose	Sigma-Aldrich, USA	≥ 85%	US\$70.61/mg
Cellopentaose	Megazyme, Ireland	> 95%	US\$8.40/mg
Cellohexaose	Megazyme, Ireland	> 95%	US\$18.82/mg

by Megazyme and Sigma-Aldrich (Barbarosa et al., 2020; Chen et al., 2021) (Table 8).

The market price of XOS and COS can vary depending on the source material, degree of purity, and production process. According to Lan et al. (2021), the minimum selling price of XOS ranges from US\$3430 to 7500/MT for the baseline case with 80% XOS content, US\$4030 to 8970/MT for 90% content, and US\$4840 to 10,640/MT for 95% content. Barbarosa et al. (2020) conducted a techno-economic analysis of COS production and found that the unit production cost of cellopentaose varies between US\$0.40 and 1.15/mg. A recent study by Xiong et al. (2023) estimated revenue of US\$118.86 from the production of XOS, glucose, and lignin by enzymatic processing of 100 kg of waste bamboo, accounting for the cost of enzymes used during the process.

Challenges and Future Work

Pretreatment By-product Recovery During Production of XOS and COS

In the production process of XOS and COS, lignocellulose releases several phenolic compounds. Phenolic compounds released during the pretreatment process can be lignin derivatives or plant secondary metabolites. Vanillin is one of the valuable phenolics formed from lignin compounds

that are discarded during the COS and XOS manufacturing processes. To generate the vanillin from the lignin, the liquid fraction from the pretreatment II process (Fig. 4) was collected and fermented in an orbital shaker using submerged fermentation (SmF). The vanillin compound was extracted in ethyl acetate for 160 min from the fermented products. The vanillin was separated from other phenolic compounds, then centrifuged and filtered. The presence of vanillin in the filtrate and residue can be confirmed and quantified using LC–ESI–MS/MS. Thin-layer chromatography (TLC) was used for further purification (Harshvardhan et al., 2017; Nurika et al., 2020) (Fig. 6).

Separation of β -glucosidase Enzymes Using “Separation–Adsorption” Method in High Temperature and Continuous System for COS Production

To increase the amount of COS produced, the BG enzyme must be separated from the other cellulase enzymes (EG and CBH). Separating enzymes generally has a high production cost, so the adsorption–separation method developed by (Chu et al., 2014) is an alternative to separating BG from EG and CBH. The BG enzyme is separated by utilizing its isoelectric point, and decreasing the temperature results in a decrease in the retention rate of BG. When centrifuged, BG will leave the solid–liquid system and accumulate in the supernatant, while the solid residue consists of EG, CBH, and a trace of BG. Each time the product is separated from the substrate, the BG remains are gradually released in the multi-step enzymatic hydrolysis system.

To increase production capacity on a larger scale, thermostable cellulases can be induced from thermophilic microbes and used in the process (Yuansah et al., 2019). An adsorption–separation method can be used to separate thermostable cellulase enzymes from BGs. The thermostable cellulase-deficient BGs can be immobilized in a matrix for continuous system bioreactors (Chu et al., 2014; Pino et al., 2018) (Fig. 7). The stability of immobilized thermostable enzymes will be an advantage in large-scale production, particularly in avoiding contamination and optimizing production yields. Furthermore, the enzymes used in the process can be reused, and the enzymes do not mix with the product or require further separation.

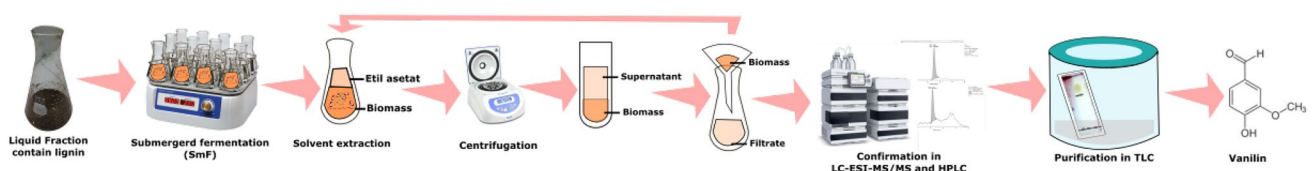


Fig. 6 Schematic diagram of vanillin recovery from the post-pretreatment liquid fraction in XOS and COS production

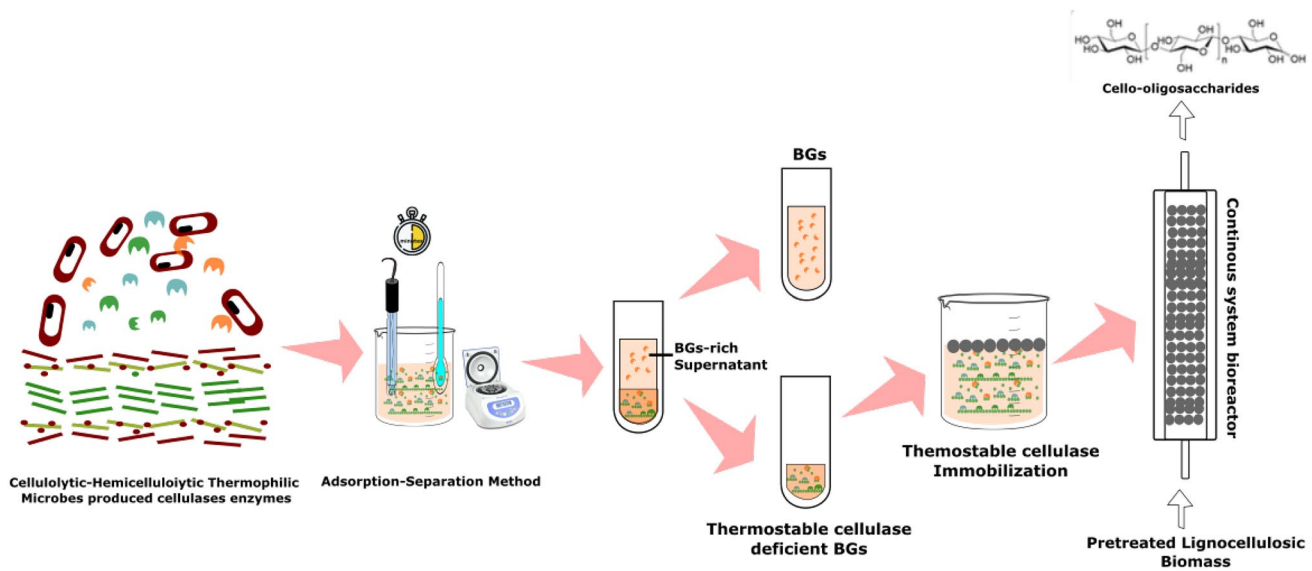


Fig. 7 Schematic diagram of adsorption–separation methods to produce thermostable, cellulase-deficient BGs and their application in continuous system bioreactor

Conclusions

Based on the discussion thus far, it is possible to conclude that lignocellulosic biomass is one of the natural sources that can be converted into a variety of functional compounds, such as XOS and COS. As previously stated, because the complex structure of lignocellulose makes it difficult to convert enzymatically and results in a slow reaction and low yield, it is critical to understand the inhibitors on the lignocellulosic source to determine strategies for carrying out enzymatic hydrolysis for COS and XOS production. Thus, it is envisaged that additional studies will be conducted to establish a more efficient technique for the production of valuable products from lignocellulose.

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Declarations

Ethics Approval and Consent to Participate Not applicable.

Consent for Publication Not applicable.

Competing Interests The authors declare no competing interests.

References

- Aleixandre, A., Gil, J. V., Sineiro, J., & Rosell, C. M. (2022). Understanding phenolic acids inhibition of α -amylase and α -glucosidase and influence of reaction conditions. *Food Chemistry*, 372. <https://doi.org/10.1016/j.foodchem.2021.131231>
- Amorim, C., Silvério, S. C., Prather, K. L. J., & Rodrigues, L. R. (2019). From lignocellulosic Residues to market: Production and commercial potential of xylooligosaccharides. *Biotechnology Advances*, 37(7), 107397. <https://doi.org/10.1016/j.biotechadv.2019.05.003>
- Antczak, A., Szadkowski, J., Szadkowska, D., & Zawadzki, J. (2022). Assessment of the effectiveness of liquid hot water and steam explosion pretreatments of fast-growing poplar (*Populus trichocarpa*) wood. *Wood Science and Technology*, 56(1), 87–109. <https://doi.org/10.1007/s00226-021-01350-1>
- Ávila-lara, A. I., Camberos-flores, J. N., & Mendoza-pérez, J. A. (2015). Optimization of Alkaline and Dilute Acid Pretreatment of Agave Bagasse by Response Surface Methodology, 3(September), 1–10. <https://doi.org/10.3389/fbioe.2015.00146>
- Ávila, P. F., & Goldbeck, R. (2022). Fractionating process of lignocellulosic biomass for the enzymatic production of short chain cello-oligosaccharides. *Industrial Crops and Products*, 178(80). <https://doi.org/10.1016/j.indcrop.2022.114671>
- B.Brenellia, L., Bhatia, R., Djajadid, D. T., Thygesend, L. G., Rabeloe, S. C., Leak, D. J., et al. (2022). Xylo-oligosaccharides, fermentable sugars, and bioenergy production from sugarcane straw using steam explosion pretreatment at pilot-scale. *Bioresource Technology*. <https://doi.org/10.1016/j.biortech.2022.127093>
- Barbarosa, F. C., Kendrick, E., Beatriz, L., Silvano, H., Maria, G., Sarita, C., et al. (2020). Optimization of cello-oligosaccharides production by enzymatic hydrolysis of hydrothermally pretreated sugarcane straw using cellulolytic and oxidative enzymes. *Biomass and Bioenergy*, 141(August). <https://doi.org/10.1016/j.biombioe.2020.105697>
- Baruah, J., Nath, B. K., Sharma, R., Kumar, S., Deka, R. C., Baruah, D. C., & Kalita, E. (2018). Recent trends in the pretreatment of lignocellulosic biomass for value-added products. *Frontiers in*

- Energy Research*, 6(DEC), 1–19. <https://doi.org/10.3389/fenrg.2018.00141>
- Broekaert, W. F., Courtin, C. M., Verbeke, K., Wiele, T. V., De, V., & W., & Delcour, J. A. (2011). Prebiotic and other health-related effects of cereal-derived arabinoxylans, arabinoxylan-oligosaccharides and XOS. *Critical Reviews in Food Science and Nutrition*, 51, 178–194. <https://doi.org/10.1080/10408390903044768>
- Cano, M. E., García-Martin, A., Morales, P. C., Wojtusik, M., Santos, V. E., Kovensky, J., & Ladero, M. (2020). Production of oligosaccharides from agrofood wastes. *Fermentation*, 6(1), 1–27. <https://doi.org/10.3390/fermentation6010031>
- Capetti, C. C. de M., Pellegrini, V. O. A., Espirito Santo, M. C., Cortez, A. A., Falvo, M., Curvelo, A. A. da S., et al. (2023). Enzymatic production of xylooligosaccharides from corn cobs: Assessment of two different pretreatment strategies. *Carbohydrate Polymers*, 299(July 2022), 120174. <https://doi.org/10.1016/j.carbpol.2022.120174>
- Chen, X., Zhai, R., Li, Y., Yuan, X., Liu, Z. H., & Jin, M. (2020). Understanding the structural characteristics of water-soluble phenolic compounds from four pretreatments of corn stover and their inhibitory effects on enzymatic hydrolysis and fermentation. *Biotechnology for Biofuels*, 13(1), 1–13. <https://doi.org/10.1186/s13068-020-01686-z>
- Chen, X., Li, H., Sun, S., Cao, X., & Sun, R. (2016). Effect of hydrothermal pretreatment on the structural changes of alkaline ethanol lignin from wheat straw. *Scientific Reports*, 6(September), 1–9. <https://doi.org/10.1038/srep39354>
- Chen, Y., Xie, Y., Ajuwon, K. M., Zhong, R., Li, T., Chen, L., et al. (2021). Xylo-oligosaccharides, preparation and application to human and animal health: A review. *Frontiers in Nutrition*, 8(September), 1–10. <https://doi.org/10.3389/fnut.2021.731930>
- Cheng, X., Luo, Y., Gao, Y., Li, S., Xu, C., Tang, S., et al. (2022). Surfactant-assisted alkaline pretreatment and enzymatic hydrolysis of *Miscanthus sinensis* for enhancing sugar recovery with a reduced enzyme loading. *Frontiers in Bioengineering and Biotechnology*, 10(July), 1–9. <https://doi.org/10.3389/fbioe.2022.918126>
- Chu, Q., Li, X., Xu, Y., Wang, Z., Huang, J., Yu, S., & Yong, Q. (2014). Functional cellobio-oligosaccharides production from the corncob residues of xylo-oligosaccharides manufacture. *Process Biochemistry*, 49(8), 1217–1222. <https://doi.org/10.1016/j.procbio.2014.05.007>
- Chu, Q., Song, K., Bu, Q., Hu, J., Li, F., Wang, J., et al. (2018). Two-stage pretreatment with alkaline sulphonation and steam treatment of *Eucalyptus woody* biomass to enhance its enzymatic digestibility for bioethanol production. *Energy Conversion and Management*, 175(September), 236–245. <https://doi.org/10.1016/j.enconman.2018.08.100>
- Collins, S. R., Wellner, N., Martinez Bordonado, I., Harper, A. L., Miller, C. N., Bancroft, I., & Waldron, K. W. (2014). Variation in the chemical composition of wheat straw: The role of tissue ratio and composition. *Biotechnology for Biofuels*, 7(1), 1–14. <https://doi.org/10.1186/s13068-014-0121-y>
- de Souza, A. P., Leite, D. C. C., Pattathil, S., Hahn, M. G., & Buckeridge, M. S. (2013). Composition and structure of sugarcane cell wall polysaccharides: Implications for second-generation bioethanol production. *Bioenergy Research*, 6(2), 564–579. <https://doi.org/10.1007/s12155-012-9268-1>
- Desseaux, V., Stocker, P., Brouant, P., & Ajandouz, E. H. (2018). The mechanisms of alpha-amylase inhibition by flavan-3-ols and the possible impacts of drinking green tea on starch digestion. *Journal of Food Science*, 83(11), 2858–2865. <https://doi.org/10.1111/1750-3841.14353>
- Díaz, S., Ortega, Z., Benítez, A. N., Marrero, M. D., Carvalheiro, F., & Duarte, L. C. (2021). Oligosaccharides production by enzymatic hydrolysis of banana pseudostem pulp. *Biomass Conversion and Biorefinery*, (0123456789). <https://doi.org/10.1007/s13399-021-02033-4>
- Dionisi, D., Anderson, J. A., Aulenta, F., & Paton, G. (2014). The potential of microbial processes for lignocellulosic biomass conversion to ethanol : A review, (October). <https://doi.org/10.1002/jctb.4544>
- Feng, J., & Kong, F. (2022). Enzyme inhibitory activities of phenolic compounds in pecan and the effect on starch digestion. *International Journal of Biological Macromolecules*, 220, 117–123. <https://doi.org/10.1016/j.ijbiomac.2022.08.045>
- Gallage, N. J., & Møller, B. L. (2015). Vanillin – bioconversion and bioengineering of the most popular plant flavor and its de novo biosynthesis in the vanilla orchid. *Molecular Plant*, 8(1), 40–57. <https://doi.org/10.1016/j.molp.2014.11.008>
- Gao, H., Wang, Y., Yang, Q., Peng, H., Li, Y., Zhan, D., et al. (2021). Combined steam explosion and optimized green-liquor pretreatments are effective for complete saccharification to maximize bioethanol production by reducing lignocellulose recalcitrance in one-year-old bamboo. *Renewable Energy*, 175, 1069–1079. <https://doi.org/10.1016/j.renene.2021.05.016>
- Giovannoni, M., Gramegna, G., Benedetti, M., & Mattei, B. (2020). Industrial use of cell wall degrading enzymes: The fine Line between production strategy and economic feasibility. *Frontiers in Bioengineering and Biotechnology*, 8(April), 1–20. <https://doi.org/10.3389/fbioe.2020.00356>
- Gong, Z., Wang, X., Yuan, W., Wang, Y., Zhou, W., Wang, G., & Liu, Y. (2020). Fed - batch enzymatic hydrolysis of alkaline organosolv - pretreated corn stover facilitating high concentrations and yields of fermentable sugars for microbial lipid production. *Biotechnology for Biofuels*, 1–15. <https://doi.org/10.1186/s13068-019-1639-9>
- González-Bautista, E., Santana-Morales, J. C., Ríos-Fránquez, F. J., Poggi-Varaldo, H. M., Ramos-Valdivia, A. C., Cristiani-Urbina, E., & Ponce-Noyola, T. (2017). Phenolic compounds inhibit cellulase and xylanase activities of *Cellulomonas flavigena* PR-22 during saccharification of sugarcane bagasse. *Fuel*, 196, 32–35. <https://doi.org/10.1016/j.fuel.2017.01.080>
- Guilherme, A. A., Dantas, P. V. F., Santos, E. S., Fernandes, F. A. N., & Macedo, G. R. (2015). Evaluation of composition, characterization and enzymatic hydrolysis of pretreated sugar cane bagasse. *Brazilian Journal of Chemical Engineering*, 32(1), 23–33. <https://doi.org/10.1590/0104-6632.20150321s00003146>
- Hao, X., Xu, F., & Zhang, J. (2022). Effect of pretreatments on production of xylooligosaccharides and monosaccharides from corncob by a two-step hydrolysis. *Carbohydrate Polymers*, 285(July 2021), 119217. <https://doi.org/10.1016/j.carbpol.2022.119217>
- Harshvardhan, K., Suri, M., Goswami, A., & Goswami, T. (2017). Biological approach for the production of vanillin from lignocellulosic biomass (*Bambusa tulda*). *Journal of Cleaner Production*, 149, 485–490. <https://doi.org/10.1016/j.jclepro.2017.02.125>
- Hidayatullah, I. M., Setiadi, T., Tri Ari Penia Kresnowati, M., & Boopathy, R. (2020). Xylanase inhibition by the derivatives of lignocellulosic material. *Bioresource Technology*, 300 (November 2019), 122740. <https://doi.org/10.1016/j.biortech.2020.122740>
- Hijosa-Valsero, M., Paniagua-García, A. I., & Díez-Antolínez, R. (2017). Biobutanol production from apple pomace: The importance of pretreatment methods on the fermentability of lignocellulosic agro-food wastes. *Applied Microbiology and Biotechnology*, 101(21), 8041–8052. <https://doi.org/10.1007/s00253-017-8522-z>
- Hsieh, C. C., Cannella, D., Jørgensen, H., Felby, C., & Thygesen, L. G. (2014). Cellulase inhibition by high concentrations of monosaccharides. *Journal of Agricultural and Food Chemistry*, 62, 3800–3805.
- Hu, B., Zhu, S., Fang, S., Huo, M., Li, Y., Yu, Y., & Zhu, M. (2016). Optimization and scale-up of enzymatic hydrolysis of wood pulp

- for cellulosic sugar production. *BioResources*, 11(3), 7242–7257. <https://doi.org/10.15376/biores.11.3.7242-7257>
- Hu, Z., Wang, Y., Liu, J., Li, Y., Wang, Y., Huang, J., et al. (2021). Integrated NIRS and QTL assays reveal minor mannose and galactose as contrast lignocellulose factors for biomass enzymatic saccharification in rice. *Biotechnology for Biofuels*, 14(1), 1–13. <https://doi.org/10.1186/s13068-021-01987-x>
- Huang, C., Lai, C., Wu, X., Huang, Y., He, J., Huang, C., et al. (2017). An integrated process to produce bio-ethanol and xylooligosaccharides rich in xylobiose and xylotriose from high ash content waste wheat straw. *Bioresource Technology*, 241, 228–235. <https://doi.org/10.1016/j.biortech.2017.05.109>
- Husin, H., Ibrahim, M. F., Kamal Bahrin, E., & Abd-Aziz, S. (2019). Simultaneous saccharification and fermentation of sago ham-pas into biobutanol by *Clostridium acetobutylicum* ATCC 824. *Energy Science and Engineering*, 7(1), 66–75. <https://doi.org/10.1002/ese3.226>
- Itelima, J., Ogbonna, A., Pandukur, S., Egbere, J., & Salami, A. (2013). Simultaneous saccharification and fermentation of corn cobs to bio-ethanol by co-culture of *Aspergillus niger* and *Saccharomyces cerevisiae*. *International Journal of Environmental Science and Development*, 4(2), 239–242. <https://doi.org/10.7763/ijesd.2013.v4.343>
- Jiang, Y., Wang, X., Wu, Z., Xu, J., Hu, L., & Lin, L. (2021). Purification of xylooligosaccharides from bamboo with non-organic solvent to prepare food grade functional sugars. *Results in Chemistry*, 3, 100153. <https://doi.org/10.1016/j.rechem.2021.100153>
- Jönsson, L. J., Alriksson, B., & Nilvebrant, N. O. (2013). Bioconversion of lignocellulose: Inhibitors and detoxification. *Biotechnology for Biofuels*, 6(1), 1–10. <https://doi.org/10.1186/1754-6834-6-16>
- Jönsson, L. J., & Martín, C. (2016). Pretreatment of lignocellulose: Formation of inhibitory by-products and strategies for minimizing their effects. *Bioresource Technology*, 199, 103–112. <https://doi.org/10.1016/j.biortech.2015.10.009>
- Karnaouri, A., Matsakas, L., Krikigianni, E., Rova, U., & Christakopoulos, P. (2019). Valorization of waste forest biomass toward the production of cello-oligosaccharides with potential prebiotic activity by utilizing customized enzyme cocktails. *Biotechnology for Biofuels*, 12(1), 1–19. <https://doi.org/10.1186/s13068-019-1628-z>
- Kendrick, E. G., Bhatia, R., Barbosa, F. C., Goldbeck, R., Gallagher, J. A., & Leak, D. J. (2022). Enzymatic generation of short chain cello-oligosaccharides from *Miscanthus* using different pretreatments. *Bioresource Technology*, 358(May), 127399. <https://doi.org/10.1016/j.biortech.2022.127399>
- Khat-udomkiri, N., Toejing, P., Sirilun, S., Chaiyasut, C., & Lailerd, N. (2020). Antihyperglycemic effect of rice husk derived xylooligosaccharides in high-fat diet and low-dose streptozotocin-induced type 2 diabetic rat model. *Food Science and Nutrition*, 8(1), 428–444. <https://doi.org/10.1002/fsn3.1327>
- Khat, N., Sivamaruthi, B. S., Sirilun, S., & Lailerd, N. (2018). Optimization of alkaline pretreatment and enzymatic hydrolysis for the extraction of xylooligosaccharide from rice husk. *AMB Express*, 8(115), 1–10. <https://doi.org/10.1186/s13568-018-0645-9>
- Kim, D., Yu, J. H., Hong, K. S., Jung, C. D., Kim, H., Kim, J., & Myung, S. (2022). Green production of low-molecular-weight xylooligosaccharides from oil palm empty fruit bunch via integrated enzymatic polymerization and membrane separation for purification. *Separation and Purification Technology*, 293(December 2021), 121084. <https://doi.org/10.1016/j.seppur.2022.121084>
- Kim, S. B., Lee, S. J., Lee, J. H., Jung, Y. R., Thapa, L. P., Kim, J. S., et al. (2013). Pretreatment of rice straw with combined process using dilute sulfuric acid and aqueous ammonia. *Biotechnology for Biofuels*, 6(1), 1–11. <https://doi.org/10.1186/1754-6834-6-109>
- Kocabas, D. S., Kole, M., & Yagci, S. (2020). Development and optimization of hemicellulose extraction bioprocess from poppy (*Papaver somniferum* L.) stalks assisted by instant controlled pressure drop (DIC) pretreatment. *Biocatalysis and Agricultural Biotechnology*, 29. <https://doi.org/10.1016/j.bcab.2020.101793>
- Kont, R., Kurašin, M., Teugjas, H., & Väljamäe, P. (2013). Strong cellulase inhibitors from the hydrothermal pretreatment of wheat straw. *Biotechnology for Biofuels*, 6(1), 1–14. <https://doi.org/10.1186/1754-6834-6-135>
- Lan, K., Xu, Y., Kim, H., Ham, C., Kelley, S. S., & Park, S. (2021). Techno-economic analysis of producing xylo-oligosaccharides and cellulose microfibrils from lignocellulosic biomass. *Bioresource Technology*, 340(August 2021), 125726. <https://doi.org/10.1016/j.biortech.2021.125726>
- Lee, I., & Yu, J. (2021). Design of hydrothermal and subsequent lime pretreatment for fermentable sugar and bioethanol production from acacia wood. *Renewable Energy*, 174, 170–177. <https://doi.org/10.1016/j.renene.2021.04.064>
- Lehuedé, L., Henríquez, C., Carú, C., Córdova, A., Mendonça, R. T., & Salazar, O. (2023). Xylan extraction from hardwoods by alkaline pretreatment for xylooligosaccharide production: A detailed fractionation analysis. *Carbohydrate Polymers*, 302 (November 2022). <https://doi.org/10.1016/j.carbpol.2022.120381>
- Li, A. L., Hou, X. D., Lin, K. P., Zhang, X., & Fu, M. H. (2018). Rice straw pretreatment using deep eutectic solvents with different constituents molar ratios: Biomass fractionation, polysaccharides enzymatic digestion and solvent reuse. *Journal of Bioscience and Bioengineering*, 126(3), 346–354. <https://doi.org/10.1016/j.jbiosc.2018.03.011>
- Li, H., Chen, X., Xiong, L., Luo, M., & Chen, X. (2019). Stepwise enzymatic hydrolysis of alkaline oxidation treated sugarcane bagasse for the co-production of functional xylo-oligosaccharides and fermentable sugars. *Bioresource Technology*, 275(2), 345–351. <https://doi.org/10.1016/j.biortech.2018.12.063>
- Li, M., Cheng, Y. L., Fu, N., Li, D., Adhikari, B., & Chen, X. D. (2014). Isolation and characterization of corncob cellulose fibers using microwave-assisted chemical treatments. *International Journal of Food Engineering*, 10(3), 427–436. <https://doi.org/10.1515/ijfe-2014-0052>
- Lin, S. H., Chou, L. M., Chien, Y. W., Chang, J. S., & Lin, C. I. (2016). Prebiotic effects of xylooligosaccharides on the improvement of microbiota balance in human subjects. *Gastroenterology Research and Practice*, 2016. <https://doi.org/10.1155/2016/5789232>
- Linares-Pasten, J., Andersson, M., & Karlsson, E. (2014). Thermostable glycoside hydrolases in biorefinery technologies. *Current Biotechnology*, 3(1), 26–44. <https://doi.org/10.2174/22115501113026660041>
- Liu, J., Liu, Y., He, X., Teng, B., & Mcrae, J. M. (2021). Valonea tannin: Tyrosinase inhibition activity, structural elucidation and insights into the inhibition mechanism. *Molecules*, 26(2747), 1–18. <https://doi.org/10.3390/molecules26092747>
- Lu, F., Rodriguez-Garcia, J., Van Damme, I., Westwood, N. J., Shaw, L., Robinson, J. S., et al. (2018). Valorisation strategies for cocoa pod husk and its fractions. *Current Opinion in Green and Sustainable Chemistry*, 14, 80–88. <https://doi.org/10.1016/j.cogsc.2018.07.007>
- Maheshwari, N. V. (2018). Agro-industrial lignocellulosic waste: An alternative to unravel the future bioenergy. In A. Kumar, S. Ogita, & Y.-Y. Yau (Eds.), *Biofuels: Greenhouse Gas Mitigation and Global Warming* (pp. 291–305). Springer.
- Majewska, M. P., Miltko, R., Beźceki, G., Kędzierska, A., & Kowalik, B. (2022). Comparison of the effect of synthetic (tannic acid) or natural (oak bark extract) hydrolysable tannins addition on fatty acid profile in the rumen of sheep.
- Marcondes, W. F., Milagres, A. M. F., & Arantes, V. (2020). Co-production of xylo-oligosaccharides, xylose and cellulose

- nano fi brils from sugarcane bagasse. *Journal of Biotechnology*, 321(June), 35–47. <https://doi.org/10.1016/j.jbiotec.2020.07.001>
- Martín, C., Dixit, P., Momayez, F., & Jönsson, L. J. (2022). Hydrothermal pretreatment of lignocellulosic feedstocks to facilitate biochemical conversion. *Frontiers in Bioengineering and Biotechnology*, 10(February), 1–17. <https://doi.org/10.3389/fbioe.2022.846592>
- Mathibe, B. N., Malgas, S., Radosavljevic, L., Kumar, V., Shukla, P., & Pletschke, B. I. (2020). Lignocellulosic pretreatment-mediated phenolic by-products generation and their effect on the inhibition of an endo-1,4- β -xylanase from *Thermomyces lanuginosus* VAPS-24. *Biotech*, 10(8), 1–11. <https://doi.org/10.1007/s13205-020-02343-w>
- Messaoudi, Y., Smichi, N., Allaf, T., Allaf, K., & Gargouri, M. (2015). Effect of instant controlled pressure drop pretreatment of lignocellulosic wastes on enzymatic saccharification and ethanol production. *Industrial Crops & Products*, 77, 910–919. <https://doi.org/10.1016/j.indcrop.2015.09.074>
- Mhlongo, S. I., den Haan, R., Viljoen-Bloom, M., & van Zyl, W. H. (2015). Lignocellulosic hydrolysate inhibitors selectively inhibit/deactivate cellulase performance. *Enzyme and Microbial Technology*, 81, 16–22. <https://doi.org/10.1016/j.enzmictec.2015.07.005>
- Moser, M., Agemans, A., & Caers, W. (2014). Production and bioactivity of oligosaccharides from chicory roots. *Food Oligosaccharides: Production, Analysis and Bioactivity*, 9781118426, 55–75. <https://doi.org/10.1002/9781118817360.ch4>
- Mugaranja, K. P., & Kulal, A. (2020). Alpha glucosidase inhibition activity of phenolic fraction from *Simarouba glauca*: An in-vitro, in-silico and kinetic study. *Heliyon*, 6(7), e04392. <https://doi.org/10.1016/j.heliyon.2020.e04392>
- Neto, F. S. P. P., Roldán, I. U. M., Galán, J. P. M., Monti, R., de Oliveira, S. C., & Masarin, F. (2020). Model-based optimization of xylooligosaccharides production by hydrothermal pretreatment of Eucalyptus by-product. *Industrial Crops and Products*, 154(October). <https://doi.org/10.1016/j.indcrop.2020.112707>
- Nurika, I., Suhartini, S., Azizah, N., & Barker, G. C. (2020). Extraction of vanillin following bioconversion of rice straw and its optimization by response surface methodology. *Molecules*, 25(6031), 1–19.
- Panakkal, E. J., Cheenkachorn, K., Gundupalli, M. P., Kitiborwornkul, N., & Sriariyanun, M. (2021). Impact of sulfuric acid pretreatment of durian peel on the production of fermentable sugar and ethanol. *Journal of the Indian Chemical Society*, 98(12). <https://doi.org/10.1016/j.jics.2021.100264>
- Patel, A. K., Singhania, R. R., Sim, S. J., & Pandey, A. (2019). Thermostable cellulases: current status and perspectives. *Bioresource Technology*, 279(November 2018), 385–392. <https://doi.org/10.1016/j.biortech.2019.01.049>
- Pino, M. S., Rodríguez-Jasso, R. M., Michelin, M., Flores-Gallegos, A. C., Morales-Rodríguez, R., Teixeira, J. A., & Ruiz, H. A. (2018). Bioreactor design for enzymatic hydrolysis of biomass under the biorefinery concept. *Chemical Engineering Journal*, 347(March), 119–136. <https://doi.org/10.1016/j.cej.2018.04.057>
- Pinyo, J., Luangpituksa, P., Suphantharika, M., Hansawasdi, C., & Wongsagonup, R. (2016). Effect of enzymatic pretreatment on the extraction yield and physicochemical properties of sago starch. *Starch/staerke*, 68(1–2), 47–56. <https://doi.org/10.1002/star.201500185>
- Pointner, M., Kuttner, P., Obrlik, T., Jäger, A., & Kahr, H. (2014). Composition of corncobs as a substrate for fermentation of biofuels. *Agronomy Research*, 12(2), 391–396.
- Potprommanee, L., Wang, X., Han, Y., Nyobe, D., Peng, P., Huang, Q., et al. (2017). Characterization of a thermophilic cellulase from *Geobacillus* sp. HTA426, an efficient cellulase-producer on alkali pretreated of lignocellulosic biomass, 1–16.
- Qin, L., Li, W. C., Liu, L., Zhu, J. Q., Li, X., Li, B. Z., & Yuan, Y. J. (2016). Inhibition of lignin-derived phenolic compounds to cellulase. *Biotechnology for Biofuels*, 9(1), 1–10. <https://doi.org/10.1186/s13068-016-0485-2>
- Qin, Liqin, Ma, J., Tian, H., Ma, Y., Wu, Q., Cheng, S., & Fan, G. (2022). Production of xylooligosaccharides from Jiuzao by autohydrolysis coupled with enzymatic hydrolysis using a thermostable xylanase. *Foods*, 11(17). <https://doi.org/10.3390/foods11172663>
- Rabemanolontsoa, H., & Saka, S. (2013). Comparative study on chemical composition of various biomass species. *RSC Advances*, 3(12), 3946–3956. <https://doi.org/10.1039/c3ra22958k>
- Rai, S. N., Birla, H., Singh, S. S., Zahra, W., Patil, R. R., Jadhav, J. P., et al. (2017). *Mucuna Pruriens* Protects against MPTP Intoxicated Neuroinflammation in Parkinson's Disease through NF- κ B/pAKT Signaling Pathways, 9 (December), 1–14. <https://doi.org/10.3389/fnagi.2017.00421>
- Ríos-Covián, D., Ruas-Madiedo, P., Margolles, A., Gueimonde, M., De los Reyes-Gavilán, C. G., & Salazar, N. (2016). Intestinal short chain fatty acids and their link with diet and human health. *Frontiers in Microbiology*, 7(FEB), 1–9. <https://doi.org/10.3389/fmicb.2016.00185>
- Santo, M. E., Rezende, C. A., Bernardinelli, O. D., Pereira, N., Curvelo, A. A. S., DeAzevedo, E. R., et al. (2018). Structural and compositional changes in sugarcane bagasse subjected to hydrothermal and organosolv pretreatments and their impacts on enzymatic hydrolysis. *Industrial Crops and Products*, 113(December 2017), 64–74. <https://doi.org/10.1016/j.indcrop.2018.01.014>
- Saroj, P., Manasa, P., & Narasimhulu, K. (2018). Characterization of thermophilic fungi producing extracellular lignocellulolytic enzymes for lignocellulosic hydrolysis under solid - state fermentation.
- Saville, B. A., & Saville, S. (2018). Xylooligosaccharides and arabinoxylooligosaccharides and their application as prebiotics, 5(3), 121–130.
- Sharma, R., Kocher, G. S., Bhogal, R. S., & Oberoi, H. S. (2014). Cellulolytic and xylanolytic enzymes from thermophilic *Aspergillus terreus* RWY. *Journal of Basic Microbiology*, 54(12), 1367–1377. <https://doi.org/10.1002/jobm.201400187>
- Sharma, S., Vaid, S., Bhat, B., Singh, S., & Bajaj, B. K. (2019). Thermostable enzymes for industrial biotechnology. In *Advances in Enzyme Technology* (pp. 469–495). Elsevier B.V. <https://doi.org/10.1016/B978-0-444-64114-4.00017-0>
- Shrivastava, B., Jain, K. K., Kalra, A., & Kuhad, R. C. (2014). Bio-processing of wheat straw into nutritionally rich and digested cattle feed. *Scientific Reports*, 4, 1–9. <https://doi.org/10.1038/srep06360>
- Siccama, J. W., Oudejans, R., Zhang, L., Kabel, M. A., & Schutyser, M. A. I. (2022). Steering the formation of cellobiose and oligosaccharides during enzymatic hydrolysis of asparagus fibre. *Lwt*, 160(November 2021), 113273. <https://doi.org/10.1016/j.lwt.2022.113273>
- da Silva, J. C., de Oliveira, R. C., da Neto, A., & S., Pimentel, V. C., & Santos, A. de A. dos. (2015). Extraction, addition and characterization of hemicelluloses from corn cobs to development of paper properties. *Procedia Materials Science*, 8, 793–801. <https://doi.org/10.1016/j.mspro.2015.04.137>
- Solarte-toro, J. C., Romero-garcía, J. M., & Martínez-patiño, J. C. (2019). Acid pretreatment of lignocellulosic biomass for energy vectors production: A review focused on operational conditions and techno-economic assessment for bioethanol production. *Renewable and Sustainable Energy Reviews*, 107(February), 587–601. <https://doi.org/10.1016/j.rser.2019.02.024>

- Sorrenti, V., Ali, S., Mancini, L., Davinelli, S., Paoli, A., & Scapagnini, G. (2020). Cocoa polyphenols and gut microbiota interplay: Bioavailability, prebiotic effect, and impact on human health. *Nutrients*, *12*(7), 1–16. <https://doi.org/10.3390/nu12071908>
- Stamogiannou, I., Van Camp, J., Smaghe, G., Van de Walle, D., Dewettinck, K., & Raes, K. (2021). Impact of phenolic compound as activators or inhibitors on the enzymatic hydrolysis of cellulose. *International Journal of Biological Macromolecules*, *186*(June), 174–180. <https://doi.org/10.1016/j.ijbiomac.2021.07.052>
- Su, Y., Fang, L., Wang, P., Lai, C., Huang, C., Ling, Z., et al. (2021). Efficient production of xylooligosaccharides rich in xylobiose and xylotriose from poplar by hydrothermal pretreatment coupled with post-enzymatic hydrolysis. *Bioresource Technology*, *342*(July), 125955. <https://doi.org/10.1016/j.biortech.2021.125955>
- Sun, L., Warren, F. J., Netzel, G., & Gidley, M. J. (2016a). 3 or 3'-Galloyl substitution plays an important role in association of catechins and theaflavins with porcine pancreatic α -amylase: The kinetics of inhibition of α -amylase by tea polyphenols. *Journal of Functional Foods*, *26*, 144–156. <https://doi.org/10.1016/j.jff.2016.07.012>
- Sun, S., Chen, W., Tang, J., Wang, B., Cao, X., Sun, S., & Sun, R. C. (2016b). Synergetic effect of dilute acid and alkali treatments on fractional application of rice straw. *Biotechnology for Biofuels*, *9*(1), 1–13. <https://doi.org/10.1186/s13068-016-0632-9>
- Takagi, R., Sasaki, K., Sasaki, D., Fukuda, I., Tanaka, K., Yoshida, K., et al. (2016). A single-batch fermentation system to simulate human colonic microbiota for high-throughput evaluation of prebiotics. *PLoS ONE*, *11*(8), 1–16. <https://doi.org/10.1371/journal.pone.0160533>
- Tan, Jian, McKenzie, C., Potamitis, M., Thorburn, A. N., Mackay, C. R., & Macia, L. (2014). *The role of short-chain fatty acids in health and disease*. *Advances in Immunology* (1st ed., Vol. 121). Elsevier Inc. <https://doi.org/10.1016/B978-0-12-800100-4.00003-9>
- Tan, J., Li, Y., Tan, X., Wu, H., Li, H., & Yang, S. (2021). Advances in pretreatment of straw biomass for sugar production. *Frontiers in Chemistry*, *9*(June), 1–28. <https://doi.org/10.3389/fchem.2021.696030>
- Thangavelu, S. K., Saleh, A., & Nasir, F. (2014). Bioethanol production from sago pith waste using microwave hydrothermal hydrolysis accelerated by carbon dioxide. *Applied Energy*, *128*, 277–283. <https://doi.org/10.1016/j.apenergy.2014.04.076>
- Thite, V. S., & Nerurkar, A. S. (2019). Valorization of sugarcane bagasse by chemical pretreatment and enzyme mediated deconstruction. *Scientific Reports*, *9*(1), 1–14. <https://doi.org/10.1038/s41598-019-52347-7>
- Vasconcellos, V. M., Tardioli, P. W., Giordano, R. L. C., & Farinas, C. S. (2015). Production efficiency versus thermostability of (hemi) cellulolytic enzymatic cocktails from different cultivation systems. *Process Biochemistry*, *50*(11), 1701–1709. <https://doi.org/10.1016/j.procbio.2015.07.011>
- Wang, S., Nie, S., & Zhu, F. (2016). Chemical constituents and health effects of sweet potato. *Food Research International*, *89*, 90–116. <https://doi.org/10.1016/j.foodres.2016.08.032>
- Wang, Y., Yang, Y., Qu, Y., & Zhang, J. (2021a). Selective removal of lignin with sodium chlorite to improve the quality and antioxidant activity of xylo-oligosaccharides from lignocellulosic biomass. *Bioresource Technology*, *337*(June), 125506. <https://doi.org/10.1016/j.biortech.2021.125506>
- Wang, Z. K., Huang, C., Zhong, J. L., Wang, Y., Tang, L., Li, B., et al. (2021b). Valorization of Chinese hickory shell as novel sources for the efficient production of xylooligosaccharides. *Biotechnology for Biofuels*, *14*(226), 1–13. <https://doi.org/10.1186/s13068-021-02076-9>
- Wijaya, H., Sasaki, K., Kahar, P., Rahmani, N., Hermiati, E., Yopi, Y., et al. (2020). High enzymatic recovery and purification of xylooligosaccharides from empty fruit bunch via nanofiltration. *Processes*, *8*(5), 1–9. <https://doi.org/10.3390/PR8050619>
- Xiong, B., Ma, S., Chen, B., Feng, Y., Peng, Z., Tang, X., et al. (2023). Formic acid-facilitated hydrothermal pretreatment of raw biomass for co-producing xylo-oligosaccharides, glucose, and lignin. *Industrial Crops and Products*, *193*(December 2022), 116195. <https://doi.org/10.1016/j.indcrop.2022.116195>
- Yang, J., Summanen, P. H., Henning, S. M., Hsu, M., Lam, H., Huang, J., et al. (2015a). Xylooligosaccharide supplementation alters gut bacteria in both healthy and prediabetic adults: A pilot study. *Frontiers in Physiology*, *6*(Aug), 1–11. <https://doi.org/10.3389/fphys.2015.00216>
- Yang, M., Zhang, J., Kuittinen, S., Vepsäläinen, J., Soininen, P., Keinänen, M., & Pappinen, A. (2015b). Enhanced sugar production from pretreated barley straw by additive xylanase and surfactants in enzymatic hydrolysis for acetone-butanol-ethanol fermentation. *Bioresource Technology*, *189*, 131–137. <https://doi.org/10.1016/j.biortech.2015.04.008>
- Yuansah, S. C. (2019). Potensi Pembuatan Gula Non-Digestible Dari Selulosa dan Hemiselulosa Menggunakan Hidrolisis Enzimatis. *Carnea Journal*, *2*(2), 69–74. <https://doi.org/10.20956/carnea.v2i2.116>
- Yuansah, S. C., Darmawan, Fitriana, N., & Laga, A. (2019). Pengaruh Pretreatment Jerami Padi Pada Produksi Enzim Termostabil Menggunakan Isolat Bakteri Termofilik (The effect of rice straw pretreatment on thermostable enzyme production using thermophilic bacteria isolate). *Canrea Journal*, *2*(1), 13–18. <https://doi.org/10.20956/carnea.v2i1.175>
- Zainudin, M. H. M., Mustapha, N. A., Hassan, M. A., Bahrin, E. K., Tokura, M., Yasueda, H., & Shirai, Y. (2019). A highly thermostable crude endoglucanase produced by a newly isolated *Thermobifida fusca* strain UPMC 901. *Nature Research*, *9*(13526), 1–8. <https://doi.org/10.1038/s41598-019-50126-y>
- Zhang, F., Lan, W., Li, Z., Zhang, A., Tang, B., Wang, H., et al. (2021). Co-production of functional xylo-oligosaccharides and fermentable sugars from corn stover through fast and facile ball mill-assisted alkaline peroxide pretreatment. *Bioresource Technology*, *337*(May), 125327. <https://doi.org/10.1016/j.biortech.2021.125327>
- Zhang, F., Lan, W., Zhang, A., & Liu, C. (2022). Green approach to produce xylo-oligosaccharides and glucose by mechanical-hydrothermal pretreatment. *Bioresource Technology*, *344*(PB), 126298. <https://doi.org/10.1016/j.biortech.2021.126298>
- Zhao, L., Sun, Z., Zhang, C., Nan, J., Ren, N., Lee, D., & Chen, C. (2022). Advances in pretreatment of lignocellulosic biomass for bioenergy production: Challenges and perspectives. *Bioresource Technology*, *343*(October 2021), 126123. <https://doi.org/10.1016/j.biortech.2021.126123>
- Zhong, C., Ukowitz, C., Domig, K. J., & Nidetzky, B. (2020). Short-chain cello-oligosaccharides: Intensification and scale-up of their enzymatic production and selective growth promotion among probiotic bacteria. *Journal of Agricultural and Food Chemistry*, *68*(32), 8557–8567. <https://doi.org/10.1021/acs.jafc.0c02660>
- Zhou, M., Fan, G., Xia, H., Zhang, X., & Teng, C. (2021). Ultrasound-assisted production of xylo-oligosaccharides from alkali-solubilized corncob bran using *Penicillium janthinellum* XAF01 acidic xylanase. *Frontiers in Bioengineering and Biotechnology*, *9*(September), 1–9. <https://doi.org/10.3389/fbioe.2021.755003>
- Zhu, J., Zhang, H., Jiao, N., Xu, G., & Xu, Y. (2022). Fractionation of poplar using hydrothermal and acid hydrolytic pretreatments for co-producing xylooligosaccharides, fermentable sugars, and lignin nanoparticles. *Industrial Crops and Products*. <https://doi.org/10.1016/j.indcrop.2022.114853>

- Zhu, Z., Rezende, C. A., Simister, R., McQueen-Mason, S. J., Macquarrie, D. J., Polikarpov, I., & Gomez, L. D. (2016). Efficient sugar production from sugarcane bagasse by microwave assisted acid and alkali pretreatment. *Biomass and Bioenergy*, 93, 269–278. <https://doi.org/10.1016/j.biombioe.2016.06.017>
- Ziolkowska, A., Debska, B., & Banach-szott, M. (2020). Content of phenolic compounds in meadow isolation method.
- Zoghalmi, A., & Paës, G. (2019). Lignocellulosic biomass: Understanding recalcitrance and predicting hydrolysis. *Frontiers in Chemistry*, 7(December). <https://doi.org/10.3389/fchem.2019.00874>

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