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Review

Revolutionizing Renewable Resources: Cutting-Edge Trends and Future Prospects in the Valorization of Oligosaccharides

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Abstract: Lignocellulosic wastes, primarily from agricultural by-products, are a renewable resource increasingly used in the sustainable production of oligosaccharides, significantly contributing to the growing bioeconomy. This innovative utilization of biological resources aligns with the global shift towards sustainable development, focusing on creating products such as food, feed, and bioenergy from renewable sources. Oligosaccharides, specialized carbohydrates, are synthesized either chemically or more eco-friendly, biologically. Biological synthesis often involves enzymes or whole-cell systems to transform lignocellulosic wastes into these valuable sugars. As functional food supplements, oligosaccharides play a crucial role in human and animal health. They serve as prebiotics, indigestible components that promote the proliferation of beneficial gut microbiota, especially within the colon. This positive impact on gut flora is essential for boosting the immune system and regulating physiological functions. Important prebiotics, including galactooligosaccharides (GOS), xylooligosaccharides (XOS), fructooligosaccharides (FOS), mannan-oligosaccharides (MOS), and isomaltooligosaccharides (IMOS), are produced through methods involving enzymes or the use of whole cells, with agricultural waste as substrates. Recent advancements focus on refining these biological processes for oligosaccharide synthesis using lignocellulosic substrates, emphasizing the principles of a circular bioeconomy, which promotes resource reuse and recycling. This review highlights the potential and challenges in the biological synthesis of oligosaccharides from renewable resources. It underscores the need for innovation in process optimization and commercialization strategies to fully exploit lignocellulosic wastes. This approach not only contributes to sustainable product development, but also opens new avenues for the profitable and environmentally friendly utilization of agricultural residues, marking a significant step forward in the bio-based industry.

Keywords: agricultural by-products; prebiotics; lignocellulosic wastes

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1. Introduction

Recently, there has been a significant rise in consumer interest towards foods that are not only safe and nutritious, but also promote longevity and overall health improvement. This trend has steered the food industry towards the development of 'functional foods', especially prebiotics. Prebiotics, primarily indigestible oligosaccharides, are acknowledged

for their health benefits and their role in disease prevention [1]. They enhance gut health, mainly by supporting beneficial bacterial colonies in the intestinal tract, thereby offering a range of health benefits including improved digestion and immune system performance [2]. In response, the food industry is now focusing on more efficient and sustainable methods for oligosaccharide production, aligning with consumer demand. Biotechnological innovations have been crucial in this area, particularly for the eco-friendly production of high-value compounds. A notable approach involves converting lignocellulosic agricultural waste into oligosaccharides, either through microbial fermentation [3] or enzymatic hydrolysis using microorganisms or enzymes [4]. Furthermore, the fields of probiotics and prebiotics merge environmental and societal studies. The Food and Agricultural Organization defines prebiotics as nonviable food components that enhance health by modulating the gut microbiome. To qualify as a prebiotic, a substance must not be a drug or organism, must demonstrate measurable health benefits, and positively affect the host's gut microbiota [5,6]. However, prebiotic effects can vary depending on the specific strains and the unique gut environments of individuals. Prebiotic sources are varied, including fruits, vegetables, and processed foods such as yogurt, cereals, and bread [7,8], which not only foster beneficial gut microbes, but also contribute to cholesterol reduction, antioxidant activity, immune strengthening, and better mineral absorption. This study also explores the role of cellulose, Earth's most abundant natural polysaccharide. Cellulose derivatives, such as carboxymethyl cellulose (CMC), are utilized as encapsulating agents but face limitations due to water solubility and sensitivity to gastric enzymes and pH. An innovative approach involves modifying natural cellulose to increase its resistance to various factors through an eco-friendly process, such as mild oxidation by TEMPO (2, 2, 6, 6-Tetramethylpiperidine-1-oxyl radical) [9], thereby enhancing cellulose's efficacy as a carrier for nondigestible prebiotic supplements. In conclusion, this study offers an exhaustive review of oligosaccharide biosynthesis, focusing on whole-cell and enzyme-mediated methods that utilize renewable resources and industrial effluents, while highlighting the challenges and limitations in the prebiotic and oligosaccharide synthesis arena. The conversion of agricultural by-products into valuable prebiotics outlines the steps from collecting waste materials such as husks and leaves to processing them into prebiotics, which support beneficial microorganisms. The diagram may show the treatments—mechanical, chemical, or biological—used to break down these by-products for gut bacteria nourishment, highlighting important chemical and enzymatic processes or fermentation involved in the transformation (Figure 1).

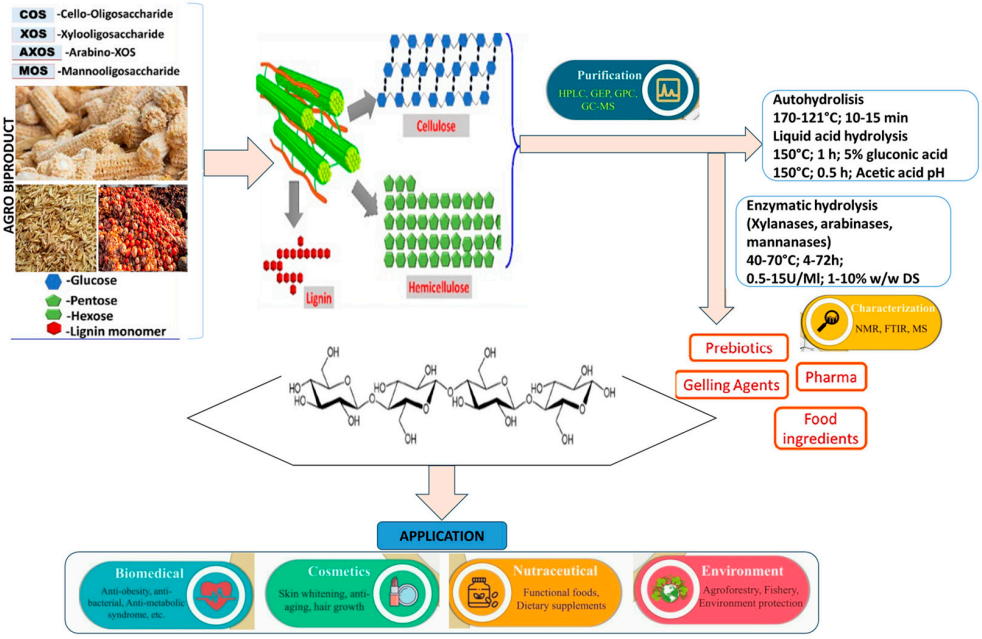

Figure 1. Visual summary of the conversion of agricultural by-products into valuable prebiotics.

Furthermore, the graphical abstract would likely include the potential applications of the resulting functional prebiotics. This could involve their use in dietary supplements, food and beverage products, animal feeds, or even in the pharmaceutical industry for the development of health-promoting products.

1.1. Global Scenario and Market Potential Delves Commercial Prospects of the Prebiotics Market

The global prebiotics market has experienced growth, primarily due to increasing concerns about obesity and a subsequent emphasis on dietary fiber-rich foods. Although natural sources such as fruits and vegetables are beneficial, they often do not meet the average dietary fiber requirements, presenting a significant opportunity for the development of functional foods enhanced with prebiotics (Figure 2) [2]. Among the various prebiotic components, galactooligosaccharides (GOS), fructooligosaccharides (FOS), and inulin stand out as the leading substances in the global market [1,2]. As of 2016, the market was valued at approximately USD 3.34 billion, with projections suggesting a compound annual growth rate (CAGR) of 10.0% up to the year 2025. This growth indicates a significant expansion within the forecast period [10]. The food and beverage sector is the primary consumer of prebiotics, accounting for a whopping 82% of the market share. Following this sector is the animal feed segment, which had a market value of USD 281.9 million in 2015 [11], highlighting the diverse applications and increasing demand for prebiotics across different industries.

The production of oligosaccharides, a key element in the prebiotic market, is led by major companies including Beneo Orfati SA, Danone, Abbott Nutrition, Roquette America Inc., Campina Domo, and Clasado, Ltd. Notably, Danisczo-DuPont has made considerable progress in this area by launching polydextrose under the brand name Litesse, known for its health benefits [12]. Clasado, Ltd., has introduced innovative technology for the synthesis of galactooligosaccharides (GOS), establishing a new standard in the industry. Leading firms such as FrieslandCampina, Danone, and Campina Domo are also focusing on GOS production, indicating a robust and dynamic market [13]. This expanding market reflects the growing awareness and demand for dietary solutions aimed at addressing health issues, such as obesity, with functional foods playing a crucial role [14].

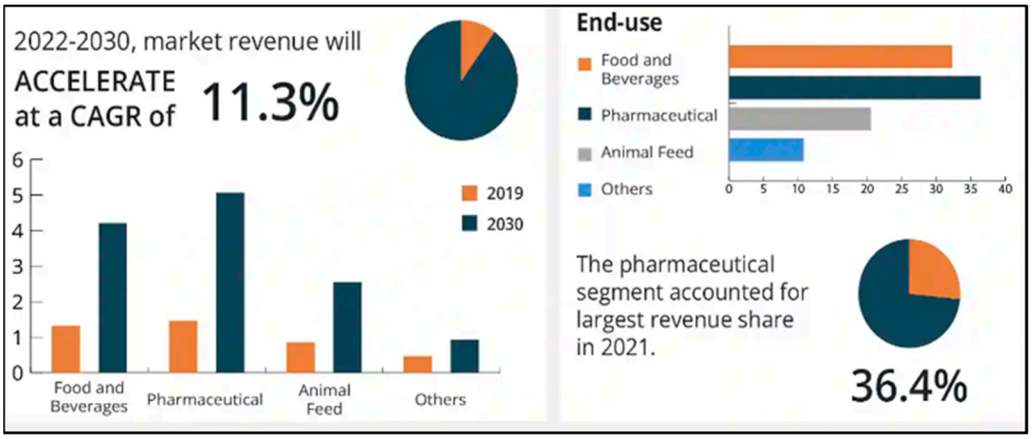

Figure 2. Prebiotics market size, share, trends, by ingredients (fructo-oligosaccharide, galacto-oligosaccharides), by product (dietary supplements, dairy food), by source (roots and grains, vegetables), by end-use, and by region forecast to 2030.

1.2. Autohydrolysis Processes: Principles and Applications

Autohydrolysis, an advanced hydrothermal treatment, utilizes pressurized water for biomass processing and becomes effective when the water temperature surpasses 120 ± 0.2 °C. This increase in temperature leads to enhanced ionization and a notable rise in hydronium ions (H3O+) [15]. At 250 ± 0.1 °C, the concentration of hydronium ions is significantly higher—23.3-times more—than at 25 ± 0.5 °C. The boosted ionization

aids in breaking down biomass components such as hemicelluloses and pectin through depolymerization [16]. During depolymerization, the release of acetic and uronic acids is key, further increasing the production of H3O+ ions, making them more effective than water alone [16]. Autohydrolysis excels in producing oligosaccharides, allowing control over monomers and degradation products by adjusting reaction time and temperature. Initially designed for separating lignocellulosic biomass by dissolving hemicellulose, it is also effective in depolymerizing pectin [16]. The process involves several steps: proton migration to the solid interface, chemical adsorption, interaction between the proton and surface polysaccharides, cleavage and release of oligosaccharides, and the diffusion of these oligomers into the surrounding liquid. The speed of this process is mainly dictated by the chemical reaction phase [17] (Figure 3).

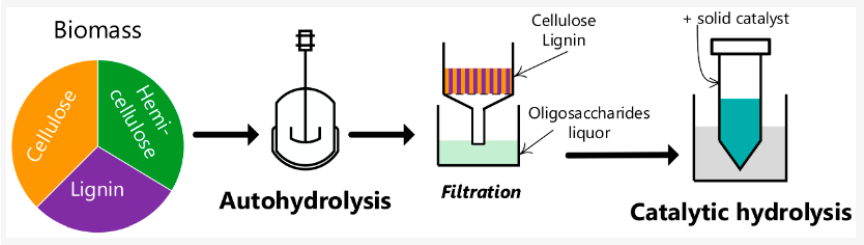

Figure 3. Combining autohydrolysis with catalytic hydrolysis to enhance the value of hemicellulose production.

Key factors influencing the autohydrolysis process include particle size, liquid-to-solid ratio (LSR), temperature/time, and pH. Particle size directly affects the surface area and porosity, with smaller particles enhancing these properties. However, the energy required for milling to achieve smaller particle sizes necessitates a careful balance [18]. The LSR, generally within 8–10 g water/g dry material, impacts the concentration of acetic and uronic acids, thereby influencing autohydrolysis efficiency and its energy demands [18]. The intertwined relationship of temperature and time is encapsulated in the severity factor, a crucial parameter that significantly affects the autohydrolysis process. Maintaining pH above 4.0 during autohydrolysis aids in selectively producing oligomers while minimizing the breakdown into monomers and degradation products [19]. The severity factor, essential for comparing diverse experimental conditions, was initially developed for pulping processes. It integrates the reaction kinetics and the temperature's effect on the rate constant, accommodating variations such as nonisothermal temperature profiles or low pH levels. The treatment temperature profile can be either isothermal, where the temperature remains constant, or nonisothermal, with the temperature decreasing after reaching a peak. Generally, higher temperatures combined with shorter reaction times lead to a higher yield of pentoses and fewer degradation products [19]. Additionally, the reaction time and temperature influence the molecular weight distribution of oligosaccharides. Autohydrolysis can be performed in various reactor configurations, including batch, semicontinuous, or continuous, with batch reactors most commonly used [20]. This process offers multiple advantages over other treatments, such as reduced chemical usage due to the natural increase in acetic acid concentration from acetyl groups in biomass, efficient solubilization of hemicelluloses and pectin, the generation of valuable by-products (both solid and liquid), and lower capital costs due to decreased corrosion potential [20].

1.3. Utilization of Lignocellulosic Waste Residues: Approaches and Applications

The use of autohydrolysis, a refined hydrothermal treatment, has significantly evolved in the processing of lignocellulosic waste residues. Initially, in the 1970s and 1980s, it was chiefly used for the delignification of various wood types [21]. However, during the 1990s, its application broadened, serving as a pretreatment to improve the accessibility of cellulose for its conversion into simple sugars for biofuel or chemical production. This era also marked an increased interest in autohydrolysis for producing xylooligosaccharides

(XOS), valued for their health benefits [22,23]. In 1999, a key study focusing on oligosaccharide production through autohydrolysis investigated the kinetics of breaking down hemicelluloses, especially xylan, into XOS and xylose, along with the examination of xylose degradation by-products [24]. This research established the groundwork for a sustainable method of valorizing hemicelluloses. Further studies extended autohydrolysis to various materials, including wood, agricultural, and food wastes, with food wastes offering a notable opportunity due to their high volume [25]. These wastes are divided into two categories: those produced during raw material conditioning, and those resulting from material processing or consumption. The former category, particularly materials high in xylan, yields liquors abundant in XOS when subjected to autohydrolysis [26].

The autohydrolysis process also accounts for the hydrolysis susceptibility of arabinan, often present in lignocellulosic residues [27]. Optimal conditions for producing xylooligosaccharides (XOS) and arabinooligosaccharides (AROS) differ, reflecting the unique requirements for each type of oligosaccharide [28,29]. Research indicates similar rates of xylan to XOS conversion across various materials, such as corncobs and rice husks, under comparable conditions [30]. However, the degree of polymerization (DP) for these oligosaccharides is frequently not detailed. For materials such as almond shells, intensifying the process severity results in XOS with a lower DP. While this leads to lower oligosaccharide concentrations, incorporating enzymatic hydrolysis and purification steps can improve yields [31]. Occasionally, small quantities of galactooligosaccharides (GAOS) and glucooligosaccharides (GOS) are produced, with their concentration and DP affected by the severity factor. This underscores the significance of precise control over temperature and time to achieve desired product specifications [32]. Comparative analysis shows that different materials necessitate distinct severity factors for optimal XOS production. For example, brewer's spent grains require a different optimal severity factor compared to other substrates, showcasing faster arabinan degradation relative to xylan [33]. Advances in this domain include strategies such as the preliminary removal of starch from grains, enhancing the prebiotic qualities of the resultant mixture. Additionally, employing partial enzymatic hydrolysis to reduce the average molecular weight of oligosaccharides further improves their functional properties [34].

The research by Gullón and colleagues highlighted that chestnut shells, when processed under relatively low severity factors, exhibited higher polysaccharide solubilization compared to other residues such as hazelnut shells. This study illuminates the distinct solubilization behaviors of various agricultural residues [34]. Under optimal conditions, this led to the significant production of xylooligosaccharides (XOS) and glucooligosaccharides (GOS), while yielding lower concentrations of arabinooligosaccharides (AROS) and antioxidant compounds. In contrast, Rico et al. [35] found that achieving similar yields of XOS required higher severity factors, which resulted in reduced concentrations of GOS, AROS, and antioxidants. Vine shoots were also identified as promising raw materials for XOS and GOS production, with notable amounts produced under specific severity conditions. Studies on these materials showed that increased severity levels resulted in a narrower molecular weight distribution of the hemicellulosic fraction, indicating a more consistent product [36]. The varied use of autohydrolysis across different lignocellulosic waste residues underscores its adaptability and effectiveness in producing valuable oligosaccharides and other beneficial compounds.

The study on lignocellulosic waste residues subjected to autohydrolysis, as presented in Table 1, focuses on materials rich in polysaccharides other than xylan, such as pectin and arabinan. These are predominantly found in various fruit peels and agricultural by-products, making them highly susceptible to hydrolytic degradation [37,38]. Notably, agricultural residues such as orange peels, lemon peels, sugar beet pulp, apple pomace, passion fruit peels, and olive by-products are characterized by their high pectin content. In contrast, sugarcane bagasse is mainly composed of xylan, and coconut meal consists largely of mannan. These compositional differences are crucial for determining the potential applications of these agricultural by-products [39].

Research into agricultural residues such as orange peels, lemon peels, and sugar beet pulp has pinpointed optimal autohydrolysis conditions at a temperature of 160 °C and a severity factor of 2.5. These conditions have been identified to maximize the production of specific oligosaccharides, thus optimizing the process's efficiency for these particular materials [40]. For example, sugar beet pulp under these conditions yields a high concentration of AROS (13 g/L), while orange and lemon peels produce significant amounts of pectooligosaccharides. However, it is essential to note that increasing the temperature beyond 160 °C leads to a reduction in acetyl substituents in oligosaccharides, resulting in higher production of monosaccharides and degradation products. This highlights a critical consideration for the thermal treatment of these substances [40]. Additionally, continuous flow reactor operations for autohydrolysis of sugar beet pulp have been explored, achieving similar AROS production levels, but with a significantly reduced residence time.

The autohydrolysis process applied to apple pomace and citrus wastes, particularly for pectin extraction, has been explored with notable outcomes. In the case of apple pomace, researchers achieved a high yield of pectin that possessed a lower molecular weight than pectin extracted through conventional methods, by applying a temperature of 150 °C for 5 min. This method paves the way for additional research focused on producing pectic oligosaccharides from apple pomace [41]. Similarly, passion fruit peels, which are primarily composed of pectin and cellulose, have been demonstrated to produce significant quantities of oligosaccharides under conditions analogous to those used for orange peels [40]. Interestingly, it is suggested that the glucan obtained from these peels likely results from starch hydrolysis rather than cellulose, with hemicelluloses showing greater resistance to hydrolysis compared to pectin [41].

Table 1. Overview of autohydrolysis for raw material conditioning wastes, highlighting its effectiveness, optimal conditions, and benefits across treatment scenarios.

SNo	Reactor	Condition	Treatment	Product	Residue	Year	Reference
1	316 stainless steel pressure reactor of 1 L capacity, featuring water circulation capability and temperature regulation via PID control.	Temperature of 200 °C for 20 min heating time, followed by a 5 min holding period. Liquid to Solid Ratio (LSR) is 10, with a severity factor (H) of 3.94.	Autohydrolysis isothermal.	High degree of polymerization xylooligosaccharides (XOS) at a concentration of 0.077 g per gram, alongside low degree of polymerization XOS at a concentration of 0.033 g per gram.	Almond shells	2019	[21]
2	A 1.5 L capacity reactor, constructed from 5100 series stainless steel, known as a Parr reactor, features temperature regulation via a precision PID-controlled system.	The process involves reaching a temperature of 200 °C, with a Liquid to Solid Ratio (LSR) set at 8, and a severity factor (H) calculated to be 4.01.	Autohydrolysis is performed under isothermal conditions, implying that the process is carried out at a constant temperature throughout. This consistent temperature is a fundamental characteristic of the procedure.	Xylooligosaccharides (XOS) present at a concentration of 0.10 g per gram, and galactooligosaccharides (GOS) at a concentration of 0.069 g per gram.	Vine shoots	2016	[22]

Table 1. *Cont.*

SNo	Reactor	Condition	Treatment	Product	Residue	Year	Reference
3	A high-pressure reactor, model BR-300, features a 0.6 L capacity stainless steel tank equipped with a heating block for temperature control. It includes a paddle agitator for mixing contents and utilizes tap water for cooling, which is circulated through an internal coil.	The procedure entails heating to 190 °C for a duration of 5 min, followed by maintaining this temperature for an additional 5 min holding period. The Liquid to Solid Ratio (LSR) is set at 10, with the agitator speed at 300 revolutions per min (r.p.m.), and a severity factor (H) calculated at 3.92.	Autohydrolysis is performed under isothermal conditions, indicating that the process is maintained at a constant temperature throughout its duration.	Xylooligosaccharides (XOS) are measured at a concentration of 0.10 g per gram.	Hazelnut shells	2017	[23]
4	A stainless steel Parr reactor, with a volume of 3.75 L, is equipped with two Rushton turbines for mixing. It is heated using external fabric mantles and features cooling through internal stainless steel loops. The temperature within the reactor is regulated using a PID-controlled system for precise thermal management.	The process involves heating to a temperature of 202 °C over a period of 39 min, with the Liquid to Solid Ratio (LSR) established at 8.	The process of autohydrolysis is performed in a nonisothermal manner, which means that it involves changing temperatures throughout the operation.	Xylooligosaccharides (XOS) are found at a concentration of 0.20 g per gram, while arabinooligosaccharides (AROS) are present at a concentration of 0.016 g per gram.	Corncob	2002	[23]
5	A Parr reactor, designed for conducting chemical reactions under controlled conditions.	The procedure involves elevating the temperature to 212 °C and maintaining this heat for a duration of 45 min, with a Liquid to Solid Ratio (LSR) set at 8.	Autohydrolysis is performed using a nonisothermal approach, indicating that the process involves changing temperatures rather than a constant temperature throughout.	Xylooligosaccharides (XOS) are recorded at a concentration of 0.10 g per gram, while galactooligosaccharides (GOS) are observed at a concentration of 0.027 g per gram.	Rice husks	2004	[24]
6	A Parr reactor constructed from stainless steel, designed for performing chemical reactions with precision and durability.	The protocol requires heating to a temperature of 202 °C for a total of 39 min, utilizing a Liquid to Solid Ratio (LSR) of 8.	Autohydrolysis is a non-isothermal process, indicating that it does not maintain a constant temperature throughout the duration of the reaction. Instead, the temperature changes as the reaction progresses.	Xylooligosaccharides (XOS) are detected at a concentration of 0.18 g per gram.	Barley husks	2004	[25]

Table 1. *Cont.*

SNo	Reactor	Condition	Treatment	Product	Residue	Year	Reference
7	A 1.5 L capacity reactor, crafted from stainless steel and equipped with a Parr PID controller, is utilized for precise temperature regulation during reactions.	The process settings include reaching a temperature of 180 °C, applying a Liquid to Solid Ratio (LSR) of 8, and achieving a severity factor (H) of 3.08.	The method of autohydrolysis is performed under nonisothermal conditions, implying that it involves temperature variations throughout the process.	Xylooligosaccharides (XOS) are present at a concentration of 0.057 g per gram, while galactooligosaccharides (GOS) are measured at a concentration of 0.054 g per gram.	Chestnut shells	2018	[26]
8	A 0.6 L capacity reactor, made of stainless steel and identified as the Parr 4842 model, is designed for conducting various chemical processes.	The experimental setup involves heating to a temperature of 210 °C, maintaining a Liquid to Solid Ratio (LSR) of 8, and achieving a severity factor (H) of 4.09.	Autohydrolysis is carried out under nonisothermal conditions, signifying that the process involves varying temperatures over time.	Xylooligosaccharides (XOS) are identified at a concentration of 0.061 g per gram.	Peanut shells	2018	[27]
9	A stainless steel reactor with a capacity of 0.6 L is employed for the process. The stirring is achieved using two four-blade turbine impellers, and electric heating is utilized to maintain the required temperature. Cooling is facilitated through an internal loop that circulates water, ensuring precise temperature control throughout the reaction.	The experimental conditions involve maintaining a precise temperature of 200 ± 0.2 °C for a duration of 10 min during the holding phase. Additionally, a Liquid to Solid Ratio (LSR) of 10 is applied throughout this process. These specific parameters are critical for ensuring the reproducibility and success of the experiment, allowing for accurate data collection and analysis.	Autohydrolysis Isothermal.	In this context, it's noteworthy that the concentration of low degree of polymerization xylooligosaccharides (Low DP-XOS) is determined to be 0.12 g per gram. This measurement provides valuable information about the composition of the sample and its suitability for various applications.	Sugarcane bagasse	2018	[42]
10	A stainless steel vessel with a volume of 0.12 L is utilized for the experimental setup. The vessel's heating is accomplished using an aluminum block heater, which is meticulously regulated through a PID (proportional-integral-derivative) controller to maintain precise temperature conditions. To counteract the generated heat, the vessel is efficiently cooled by a continuous flow of tap water, ensuring the stability and control of the entire system.	The experimental procedure comprises two distinct phases, starting with heating the system to a temperature of 275 °C, followed by a subsequent cooling phase that extends the total duration to 14.5 min. Throughout this process, a Liquid to Solid Ratio (LSR) of 10 is maintained, and a severity factor (H) of 4.52 is calculated. These specific conditions are meticulously selected and are instrumental in achieving the desired results and gaining insights into the behavior of the materials involved in the experiment.	Autohydrolysis nonisothermal.	In the context provided, the concentration of mannooligosaccharides (MANOS) is reported at 0.23 g per gram. This specific measurement is crucial for understanding the composition of the analyzed sample and its potential applications.	Coconut meal	2014	[43]

Table 1. *Cont.*

SNo	Reactor	Condition	Treatment	Product	Residue	Year	Reference
11	A stainless steel vessel with a volume of 0.125 L is employed for the experimental setup. The vessel's heating is achieved through the use of an aluminum block heater, while effective cooling is ensured by the continuous flow of tap water, maintaining the desired temperature conditions throughout the process.	The procedure entails heating the system to a specific temperature of 175 °C and maintaining this temperature for a duration of 5.5 min. During this process, a Liquid to Solid Ratio (LSR) of 16 is employed, and a severity factor (H) of 2.21 is calculated, which are key parameters influencing the outcomes of the operation. These precise conditions are crucial for achieving the intended results and understanding the behavior of the materials involved in the experiment.	Autohydrolysis nonisothermal.	In the given context, the concentrations of specific oligosaccharides are observed, with polysaccharides (POS) measured at a concentration of 0.14 g per gram, and galactooligosaccharides (GOS) recorded at a concentration of 0.051 g per gram. These values are significant in characterizing the composition of the analyzed sample.	Passion fruit peel	2017	[44]
12	A Parr reactor constructed from stainless steel, which is widely recognized for its durability and corrosion resistance, is utilized for various chemical processes and experiments.	The experimental setup involves maintaining a temperature of 195 °C, while utilizing a Liquid to Solid Ratio (LSR) of 8. Additionally, a severity factor (H) of 3.65 is calculated for the process. These specific conditions are carefully selected and crucial for achieving the desired outcomes and understanding the behavior of the materials involved in this operation.	Autohydrolysis nonisothermal.	The analysis reveals the presence of specific oligosaccharides, with xylooligosaccharides (XOS) measured at a concentration of 0.12 g per gram, galactooligosaccharides (GOS) at 0.040 g per gram, and arabinooligosaccharides (AROS) at 0.032 g per gram. These concentrations are pivotal in characterizing the composition of the sample and assessing its potential applications.	Brewery's spent grains	2015	[45]
13	The experimental setup includes a reactor vessel with a capacity of 0.05 L, constructed from SUS316 stainless steel, known for its resistance to corrosion. To achieve the desired temperature, the reactor is heated within a molten salt bath, ensuring precise temperature control during the process. Furthermore, to rapidly cool the reactor down to 50 °C in less than 3 min, a water bath cooling system is employed, allowing for efficient temperature management.	The experimental procedure involves a multi-step process, starting with heating to a temperature of 160 °C for 5 min. Subsequently, there is a 2 min holding period at this temperature, followed by a 3 min cooling phase. Throughout this process, a Liquid to Solid Ratio (LSR) of 8 is maintained. These precise time and temperature intervals, along with the LSR, are critical factors that contribute to the successful execution of the procedure and the desired results.	Autohydrolysis isothermal.	In the context provided, it is crucial to note that the concentration of Arabinooligosaccharides (AROS) is determined to be 0.15 g per gram. This specific measurement play a significant role in characterizing the composition of the analyzed sample and assessing its potential uses or applications.	Beet fiber (beet pulp)	2013	[46]

Table 1. *Cont.*

SNo	Reactor	Condition	Treatment	Product	Residue	Year	Reference
14	A stainless steel reactor with a substantial capacity of 100 L, known for its durability and versatility in accommodating large-scale chemical processes and reactions.	The operation entails maintaining a temperature of 170 °C for a duration of 15 min while utilizing saturated steam as the heating medium. These specific conditions are carefully chosen and play a significant role in the successful execution of the process at hand.	Steam processing, isothermal.	The analysis includes the identification of polysaccharides (POS), xylooligosaccharides (XOS), and galactooligosaccharides (GOS), although there is no quantitative information available regarding their respective yields in the given context. These components are essential to characterize the composition of the sample, even though the precise quantities are not provided.	Alperujo	2012	[47]
15	The equipment used is an autoclave with a working volume of 0.5 L, specifically designed for conducting controlled experiments and reactions. To monitor the conditions within the reactor, precise measurements of temperature and pressure are obtained using a thermocouple and a pressure gauge, respectively, ensuring accurate data collection and control during the experiments.	The procedure involves maintaining a temperature of 150 °C for a specific holding period of 5 min. During this time, a high Liquid to Solid Ratio (LSR) of 30 is employed, which is a critical factor in the success of the process. These controlled conditions are essential for achieving the desired outcomes in this particular operation.	Autohydrolysis Isothermal.	In the context provided, it is noteworthy that the concentration of Polysaccharides (POS) is quantified at 0.17 g per gram. This specific measurement is crucial for understanding the composition of the analyzed sample and its potential applications in various processes or industries.	Citrus peel, apple pomace	2014	[48]
16	The experimental setup comprises a 3.75 L stainless steel Parr reactor, featuring the integration of two four-blade turbine impellers, which play a crucial role in achieving thorough mixing during chemical processes. The heating system is powered by electricity, allowing for accurate temperature control within the reactor. Furthermore, the system incorporates an internal cooling loop, which effectively dissipates excess heat generated during the reactions, ensuring stable and controlled conditions throughout the experimentation.	The experimental parameters consist of maintaining a temperature of 160 °C, employing a Liquid to Solid Ratio (LSR) of 12. Additionally, a severity factor (H) of 2.46 and an agitator speed of 150 revolutions per min (r.p.m.) are meticulously selected. These precise conditions play a crucial role in ensuring the success and reproducibility of the process being conducted.	Autohydrolysis Nonisothermal.	The analysis indicates that the sample contains polysaccharides (POS) at a concentration of 0.20 g per gram and arabinooligosaccharides (AROS) at a concentration of 0.076 g per gram. These measurements are essential for characterizing the composition of the sample and assessing its suitability for specific applications or processes GALOS (0.066 g/g).	Orange peel	2010	[49]

Table 1. *Cont.*

SNo	Reactor	Condition	Treatment	Product	Residue	Year	Reference
17	A Parr reactor, crafted from durable stainless steel and boasting a substantial 3.75 L capacity, serves as the primary vessel for conducting various chemical processes and reactions, making it an essential tool in the field of research and experimentation.	The process involves maintaining a temperature of 160 °C while utilizing a Liquid to Solid Ratio (LSR) of 12. Additionally, it incorporates a severity factor (H) of 2.51 and an agitator speed of 150 revolutions per min (r.p.m.). These specific conditions are critical for achieving the desired outcomes in the given procedure.	Autohydrolysis Nonisothermal.	In the context provided, it's important to note that the sample comprises polysaccharides (POS) with a concentration of 0.25 g per gram, arabinooligosaccharides (AROS) at a concentration of 0.068 g per gram, and galactooligosaccharides (GALOS) measured at 0.026 g per gram. These specific measurements play a pivotal role in characterizing the composition of the sample and evaluating its potential applications in various industries and processes.	Lemon peel	2013	[50]

Note: H is the severity factor (Log (R_0)).

Sugarcane bagasse, another significant agricultural by-product, has been examined for its potential in generating low degree of polymerization (DP) xylooligosaccharides, achieving optimal results at 200 °C with a 10 min reaction time [33]. Furthermore, the study on coconut meal, which is abundant in mannan polymers, under optimal conditions (a severity factor of 4.5) resulted in the production of oligosaccharides, predominantly within a DP range of 2 to 6 [39]. These investigations underscore the versatility of autohydrolysis in valorizing various lignocellulosic wastes into valuable oligosaccharides and enhancing our understanding of process optimization for different biomass sources.

In research concerning autohydrolysis, the resultant liquors typically undergo steps of purification and concentration, while the remaining solid mass is further processed to separate cellulose and lignin fractions [23]. The methodologies adopted in these studies vary significantly; some focus on optimizing the autohydrolysis conditions to directly produce oligosaccharides with the preferred degree of polymerization (DP) in a single step. In contrast, other studies adopt a fractionation approach, aiming to initially extract high DP oligosaccharides, which are then subjected to selective processing through chemical or enzymatic methods to achieve the desired specifications. Regardless of the approach taken, a purification step is often essential to ensure the produced oligosaccharides meet the necessary quality and purity standards [25]. This reflects the importance of both the process strategy and the subsequent purification in achieving high-quality oligosaccharide outputs from autohydrolysis.

The susceptibility of different polymers to autohydrolysis generally follows the sequence of galactooligosaccharides (GALOS) being less susceptible than xylooligosaccharides (XOS), which in turn are less susceptible than mannan oligosaccharides (MANNOS). Notably, operational variables such as particle size or agitation speed, crucial for understanding mass transfer in dynamic processes, have not been thoroughly investigated in this context. The majority of these studies employ batch operations, with time and temperature identified as the main factors for optimization.

It is important to recognize the dual functionality of autohydrolysis, as it proves efficient not only in generating oligosaccharides, but also in producing a solid feedstock conducive to further fractionation processes. This dual capability renders autohydrolysis a valuable tool for diverse applications in the production of bio-based materials (Table 1). Specifically, the process improves the accessibility of cellulose for enzymatic action, thereby facilitating the transformation of sugars obtained via enzymatic hydrolysis into valuable products through catalysis and bioprocessing [25]. This multifaceted utility of autohydrolysis underscores its significance in the valorization of biomass, enhancing both the yield of oligosaccharides and the efficiency of producing bio-based products (Table 1).

2. Functional Properties of Polymeric Oligosaccharides

2.1. The Role and Benefits of Galactooligosaccharides (GOS) in Human and Animal Health

Galactooligosaccharides (GOS), including oligogalactosyl lactose and transgalactooligosaccharides, are prebiotics pivotal for promoting gut health, especially by fostering the growth of beneficial *Bifidobacteria*. Predominantly extracted from cow's milk and soybeans, GOS is prominently featured in human milk during the early stages of lactation. GOS comprises a glucose molecule linked to a series of galactose units, generally ranging from two to ten in number. These units can form either alpha (α) or beta (β) configurations, depending on the type of linkage between the galactose and glucose molecules [51]. Alpha GOS, with a terminal sucrose moiety, is prevalent in human and bovine milk, as well as in seeds and pulses, while beta GOS is derived from lactose. Categorized into the raffinose and melibiose families based on their specific linkages, GOS offers versatile applications due to its unique attributes. Its moisture retention capability and ability to reduce the freezing point make it a valuable food industry additive, aiding in preserving freshness and texture [52]. Additionally, GOS serves as a sweetening agent with approximately 30–60% of the sweetness of sucrose, making it suitable for a variety of food, feed, and pharmaceutical products [52]. Beyond its nutritional value, GOS is crucial in dietary supplements aimed at infants and the elderly, supporting gut health and overall wellness [53]. In medical contexts, lactulose, a derivative of GOS, is utilized to manage conditions such as hyperammonemia and portosystemic encephalopathy [54]. Importantly, GOS supplementation in infants has been linked to lower risks of allergic conditions, such as eczema, underscoring its potential in allergy prevention. This multifunctionality of GOS, spanning food quality improvement, health benefits, and medical uses, underscores its importance across the food and healthcare industries.

2.2. Exploring the Health Impacts of Xylo-Oligosaccharides (XOS) in Human and Animal Nutrition

Xylo-oligosaccharides (XOS) represent a distinctive group of nondigestible oligosaccharides known for their resistance to human digestion and significant probiotic effects. Primarily supporting *Bifidobacteria* and certain Lactobacilli, XOS offer more than just probiotic benefits; they also play potential roles in managing diabetes, exhibit antioxidant and antibacterial activities, contribute to immune system modulation, and may help reduce the risk of colon cancer [55]. Their applications are diverse, extending from human nutrition to animal feed and even cosmetics, illustrating their wide-ranging utility. Notably, XOS have a significant impact on blood sugar control and digestive health, as they are proven to influence insulin secretion and offer laxative benefits that support gastrointestinal function [56].

In specialized products such as detoxification jellies, nutritional supplements for cancer patients, and weight loss foods, xylo-oligosaccharides (XOS) prove their versatility. Moreover, as a noncariogenic sweetener, XOS is ideal for low-calorie diets, offering sweetness without promoting tooth decay. XOS is associated with numerous health benefits, including enhanced calcium absorption, improved lipid metabolism, cardiovascular protection, and anti-inflammatory and antiallergic effects [57]. Its regular intake has been linked to a healthier gut microbiota in older adults. While XOS offers various benefits across

multiple health and nutritional applications, caution is recommended when using it to treat constipation in pregnant women due to the possibility of side effects.

Chemically, XOS consist of xylose units linked by β-(1–4)-xylosidic bonds, granting them resistance to acidic environments and high temperatures. These molecules are derived from the hydrolysis of xylan, a major component of lignocellulosic biomass, with their molecular formula expressed as C5nH8n + 2O4n + 1 for n values ranging from 2 to 6. This range illustrates the diversity in their molecular structures and properties, offering insight into XOS's chemical composition and origins. Although XOS are naturally present in various foods such as fruits, vegetables, and honey, the concentrations in these sources are typically too low to yield substantial probiotic effects [58,59]. Consequently, dietary supplementation with XOS is crucial for exploiting their full health benefits, highlighting their increasing significance across the food, nutrition, healthcare, and cosmetics industries (Figure 4).

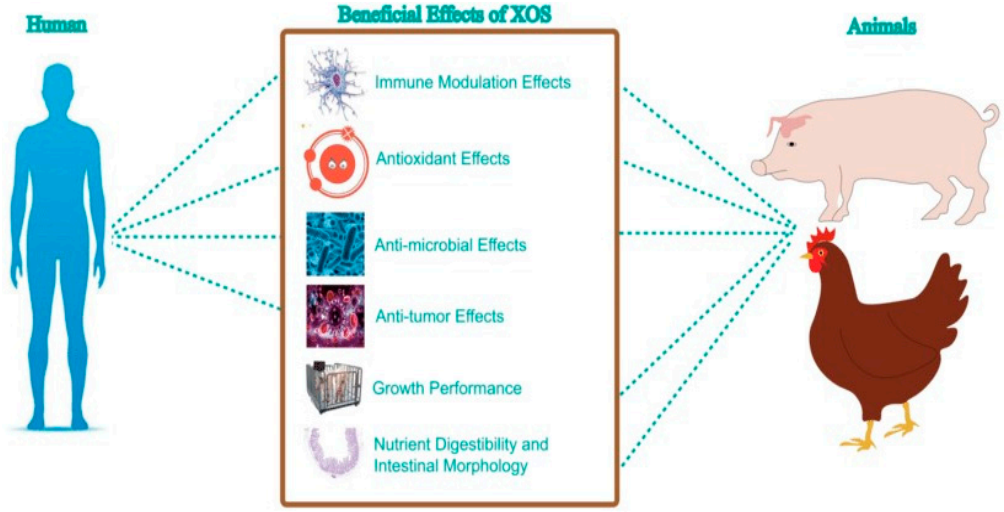

Figure 4. Functional health effect of xylooligosacharide towards humans and animals.

2.3. Investigating the Nutritional and Health Benefits of Fructo-Oligosaccharides (FOS) in Humans and Animals

Fructooligosaccharides (FOS) are a group of oligosaccharides composed of short chains of fructose molecules. The main constituents of FOS are 1-kestose, nystose, and fructofuranosylnystose [60]. These molecules feature one to three fructosyl units attached to the β-2,1 position of a sucrose molecule, a structure that prevents digestion by human enzymes and allows them to reach the colon intact. In the food industry, the functional properties of FOS are highly valued. It serves as a stabilizer, enhancing the texture and consistency of various food products. Additionally, FOS acts as a bulking agent, increasing food volume without substantially raising caloric content [61]. FOS is also utilized in sweetener production due to its natural sweet taste, making it a suitable sugar substitute, especially for those reducing sugar intake.

The health benefits and physiological effects of FOS on human health are extensive. A key advantage of FOS is its prebiotic nature, supporting the growth and metabolic activity of beneficial bacteria such as *Bifidobacteria* in the human gut. This prebiotic action is crucial for promoting a healthier and more balanced gut microbiome, leading to enhanced digestive health and overall well-being. Such effects contribute to improved gut health and digestion, aiding in alleviating conditions such as constipation [62]. Beyond gut health, FOS is explored for its ability to regulate blood sugar levels and its potential in diabetes prevention or management. With a low caloric value of 1.5 kcal/g, FOS is an appealing choice for calorie-conscious consumers. Additionally, its noncariogenic property means it does not promote tooth decay, addressing a common issue associated with sugar intake [63].

Previous studies have underscored the capacity of Fructooligosaccharides (FOS) to bolster the body's defenses against certain pathogenic bacteria in the intestines, thereby

enhancing intestinal health [64]. Moreover, dietary FOS supplementation has been associated with the inhibition of precancerous lesions in animal models, alongside promoting the proliferation of beneficial gut bacteria and lowering cecal pH, suggesting potential roles in cancer prevention [65]. An essential benefit of FOS is its positive impact on mineral absorption. Research indicates that animals supplemented with FOS show increased absorption of key minerals such as calcium and magnesium, resulting in improved bone density [66]. This property of FOS is particularly valuable for individuals at risk of osteoporosis.

FOS has received widespread recognition and regulatory approval as a food additive in various countries, including the United States, Japan, and some European Union nations. This approval underlines FOS's safety, versatility, and utility in diverse food products and dietary contexts [67]. The recommended daily intake of FOS is established at 0.8 g per kilogram of body weight, a guideline aimed at optimizing health benefits while mitigating potential adverse effects. Thus, FOS emerges as a versatile and beneficial element within the food and health industries. Their unique structure and functional qualities render them apt for multiple uses, whereas their health benefits, particularly concerning gut health and mineral absorption, underscore their significance as a dietary supplement.

2.4. Assessing the Role of Mannan-Oligosaccharides (MOS) in Enhancing Human and Animal Health and Nutrition

Mannan-oligosaccharides (MOS), sourced from the cell walls of yeasts and fungi, play an increasingly prominent role in animal nutrition and health. In the poultry sector, MOS is a favored feed additive, recognized for its capacity to bolster gut health and immune responses in chickens and other birds [68]. This enhancement of intestinal health is partially credited to MOS's ability to adhere to pathogenic bacteria, preventing these pathogens from attaching to the intestinal lining and causing infections. The benefits of MOS, however, extend beyond poultry to other monogastric animals, including pigs and sheep. One of the standout properties of MOS is its antioxidative capability. It has been demonstrated to mitigate oxidative stress in animals by neutralizing free radicals and curtailing lipid peroxidation, which can otherwise result in cellular damage [69].

Mannan-oligosaccharides (MOS) act as prebiotics due to their resistance to digestion in the upper gastrointestinal tract of animals, enabling them to arrive intact in the colon. There, MOS promotes the growth of beneficial gut bacteria, especially *Bifidobacteria*, which are essential for optimal gut health, digestion, and immune system enhancement. Research demonstrates that animals consuming MOS-supplemented diets show notable improvements in growth rates, feed conversion ratios, and disease resistance, even under stress-inducing circumstances such as weaning or pathogen exposure [70]. The production of MOS represents a sustainable method of valorizing agricultural waste, through the enzymatic hydrolysis of such waste to extract valuable MOS, showcasing an eco-friendly strategy to convert underused resources into high-value products. Beyond gut health and immunity, recent studies suggest MOS's potential anticarcinogenic effects, particularly against colon cancer, opening new research pathways for its use as a functional ingredient in animal feeds and potentially in human diets for disease prevention and health enhancement [71]. Therefore, mannan-oligosaccharides are highlighted as a pivotal element in animal nutrition, delivering a spectrum of health benefits, from gut health and immune support to antioxidant and possible anticarcinogenic properties, with their production from sustainable sources further boosting their value in the feed industry and possibly for human nutrition.

3. Synthesis of Oligosaccharides from Lignocellulosic Biomass

3.1. Approaches to Oligosaccharide Production: Cell-Free and Whole Cell-Mediated Biosynthesis

The production of galactooligosaccharides (GOS) encompasses sophisticated chemical and enzymatic procedures. Initially, researchers investigated a chemical synthesis approach, which utilized mineral acids to act on monosaccharides. Despite this method's potential, it faced significant hurdles regarding scalability and the specificity of the resulting products.

These challenges rendered the chemical synthesis approach impractical for large-scale production [72].

3.1.1. Production of Galactooligosaccharides (GOS) Using Lignocellulosic Biomass

The synthesis of galactooligosaccharides (GOS) through chemical methods, particularly via reversion involving the treatment of monosaccharides with mineral acids, encountered significant challenges. The lack of specificity in the products generated was not suited for large-scale production, and environmental concerns about the use of harsh chemicals further limited its feasibility within the food industry [5,73]. These obstacles highlighted the necessity for more efficient and environmentally sustainable alternatives. As a result, the research focus shifted towards enzymatic synthesis methods, particularly employing enzymes such as β-galactosidase (E.C. 3.2.1.23) and α-galactosidase (E.C. 3.2.1.22), marking a commitment to greener and more effective production techniques. These enzymes are proficient in catalyzing the hydrolysis of β-galactosides into monosaccharides. A notable advancement was made with β-galactosidase from *Aspergillus oryzae*, which showed exceptional ability to convert lactose into GOS under specific conditions, representing a significant step forward in GOS production [74] (Table 2).

Innovative approaches for continuous galactooligosaccharides (GOS) production have been explored, focusing on enhancing the process's efficiency and scalability. One notable method involves immobilizing β-galactosidase enzymes on various substrates, coupled with nanofiltration fractionation using cellulose acetate membranes. These techniques not only improve the production efficiency of GOS, but also present viable options for scaling up the manufacturing process [75]. Specifically, the use of chitosan as a substrate for immobilizing β-galactosidase has shown to significantly increase the yield of GOS, rendering the production process more economical and efficient compared to conventional methods [76]. Additionally, whole cell transformation techniques employing *Kluyveromyces marxianus*, followed by enrichment with *Saccharomyces cerevisiae*, have proven effective in enhancing both the purity and yield of GOS [77]. This strategy underscores the potential of utilizing living cells as biocatalysts in GOS production, offering an environmentally friendly and efficient alternative to chemical synthesis methods.

The extraction and utilization of β-galactosidase enzymes for galactooligosaccharides (GOS) production have been central to various research projects, with significant breakthroughs reported. Yang et al. demonstrated a remarkable advancement by using β-galactosidase from *Thermotoga naphthophila RKU-10*, a thermophilic enzyme showing a 100% catalytic efficiency. This enzyme was used to convert lactose from processed milk waste into GOS, achieving a high yield of 23.28 g per L per h at 75 °C and pH 6.5 [78].

Moreover, the enzyme β-glucosidase TN0602, characterized by its deep and narrow catalytic pocket, has been crucial for the efficient conversion of lactose into GOS. In another study, Hackenhaar et al. utilized whey permeate, a dairy industry by-product, for GOS production using a commercial β-glucosidase enzyme from *Kluyveromyces lactis*. This process resulted in an 89.27% lactose conversion rate and a GOS yield of 25 g per 100 g of lactose, with a specific productivity of 51 g of GOS per gram of enzyme per h, showcasing the process's high efficiency [79].

Kittibunchakul et al. focused on finding cost-effective sources for GOS production, crucial for the prebiotic industry's sustainability. They employed β-glucosidase immobilized on chitosan-coated magnetic nanoparticles, achieving a notable GOS yield of 17% mol/mol from 2.34 M lactulose after a 36 h reaction period. This method represents a promising and economically viable approach for GOS production, offering a significant contribution to the advancement and sustainability of the prebiotic sector [80].

Extensive research underscores that the synthesis of galactooligosaccharides (GOS) is predominantly facilitated by enzymes derived from fungal and bacterial origins, utilizing lactose as the substrate. Arsov et al. conducted a noteworthy study using an enzyme from *Limosilactobacillus reuteri*, achieving a GOS production yield of 38% from lactose. The primary GOS compounds produced included allolactose, D-Galp-(1 → 6)-D-Gal,

among others [81]. Delgado-Fernandez et al. investigated the properties of recombinant β-galactosidase from Lactiplant.

Table 2. Summary of GOS production methods, β-galactosidase sources, yields, and processes, with related studies to improve production and understand synthesis factors.

SNo	Source of β-Galactosidase	Yield	Process	Reference
1	*L. acidophilus* ATCC 4356, a specific strain known for its enzymatic activity. This strain is employed to catalyze various biochemical reactions, including the conversion of lactose into valuable galactooligosaccharides (GOS), making it a crucial component in the production of prebiotic compounds.	86 g/L	The process involves the immobilization of enzymes on a methacrylic polymer carrier, which serves as a stable and effective support system for the enzymes, facilitating their catalytic activity and enabling various applications in biotechnology and industrial processes.	[82,83]
2	*T. naphthophila* RKU-10	23.28 g/L/h	Enzyme.	[84,85]
3	*A. niger*	35%	Enzyme.	[86,87]
4	*K. lactis*	21 g/L	In this innovative approach, bead-immobilized β-galactosidase is employed in conjunction with nanofiltration for the fractionation of sugar mixtures. The use of cellulose acetate membranes enhances the efficiency and selectivity of the fractionation process, making it a valuable technique in the production and purification of specific sugar compounds, including oligosaccharides.	[88,89]
5	*A. oryzae*	29 g/100 g of lactose	Fermentation in 50% (*w*/*w*) lactose monohydrate.	[90]
6	*T. thermophillus*	34%	Immobilization on to insoluble carrier Eupergit C.	[91]
7	*A. oryzae*	39.30%	Packed bed reactor.	[92]
8	*B. circulans*	44%	Enzyme.	[92]

3.1.2. Generating Xylooligosaccharides (XOS) from Lignocellulosic Biomass: Techniques and Advancements

The trend in research increasingly emphasizes transforming agricultural by-products into valuable prebiotics, particularly focusing on xylooligosaccharides (XOS). Samanta et al. [93] conducted a significant study utilizing pigeon pea stalks (*Cajanus cajan*), which were processed with sodium hydroxide and steam to extract an impressive 96% of xylan content. This extracted xylan was then treated with xylanase enzyme (11.01 U), resulting in the production of xylobiose (0.502 mg/mL) at an optimized temperature of 48.11 ± 1 °C and pH of 4.91 over a duration of 15.65 h. Additionally, this process yielded xylotriose (0.204 mg/mL) at 39.29 °C and pH 5.44 using 3.23 U of xylanase over 15.26 h. This approach exemplifies the potential of utilizing enzymatic treatments on agricultural residues to efficiently produce XOS, showcasing the viability of converting underutilized biomass into significant prebiotic compounds.

In a separate study, Bian et al. [94] focused on sugarcane bagasse, treating it with potassium hydroxide and then applying crude xylanase produced by *Pichia stipitis*. This approach resulted in the extraction of xylan-rich hemicelluloses and XOS, with an impressive yield of 31.8% (5.29 mg/mL) of XOS after 12 h. The XOS composition primarily included xylobiose, xylotriose, and xylotetraose, along with smaller amounts of xylopentose and

xylohexose, verified through NMR and FT-IR analysis. These XOS also featured Araf and 4-O-Me-α-d-GlcpA residues.

Peng et al. [95] highlighted the isolation of a thermostable endoxylanase from *Streptomyces thermovulgaris* (*S. thermovulgaris*) *TISTR1948*, utilized for producing xylooligosaccharides (XOS) from corncob. Operating at an optimal condition of 53.80 ± 0.4 °C and pH 6.17, the enzyme successfully transformed 752.15 mg/g of KOH-treated hemicellulose into 162.97 mg/g of XOS, demonstrating its efficiency for high-yield XOS production. This is indicative of the broad and inventive strategies employed in generating XOS from agricultural by-products, emphasizing their importance in the food industry. Samanta et al. [96] achieved the production of 1.9 mg/mL of xylobiose by hydrolyzing xylan extracted from corn husks using xylanase (5.7 U/mL) at 44 ± 3 °C and pH 5.8 over 17.5 h. Similarly, Jnawali et al. [97] successfully extracted 93% of xylan from brown coconut husk using a 20% sodium hydroxide (NaOH) and steam treatment. Postextraction, the xylan was treated with xylanase, yielding significant amounts of xylobioses.

Moreover, Zhang et al. [98] explored genetic engineering by inserting an alkali-tolerant xylanase gene from *Bacillus subtilis* (*B. subtilis*) Lucky9 into *E. coli BL21*. The expressed recombinant xylanase, with a molecular weight of 21 KDa, was efficient in converting xylan from beech wood and corncob into xylobiose and xylotriose. Notably, this enzyme retained 60% of its activity after 2 h at a high temperature of 60 ± 3 °C. These diverse approaches underscore the potential of various agricultural residues as sources for XOS production. The use of different substrates, such as pigeon pea stalks and coconut husks, alongside various enzymatic and chemical processes, showcases the versatility and effectiveness of these methods in producing valuable prebiotics from plant biomass.

Corn cobs, recognized for their cost-effectiveness, have been leveraged for xylooligosaccharides (XOS) production. A steam processing technique facilitates the extraction of 90% xylan from the corn cobs, which is then transformed into xylobiose (X2) and xylotriose (X3) using commercial xylanase enzymes. Optimization research, such as the work conducted by Samanta et al. [98], has significantly improved the yields of these oligosaccharides by fine-tuning variables such as temperature, pH, and enzyme concentration.

Sugarcane bagasse is another agricultural by-product processed to extract xylan, achieving an 80% recovery rate through a 12% sodium hydroxide w/v and steam treatment. XOS2 and XOS3 are synthesized from the obtained xylan. Advanced optimization methodologies, including response surface methodology (RSM) and ridge analysis, have been applied to maximize xylobiose production from the xylan extracted from sugarcane bagasse [99].

Wheat bran, an abundant agricultural by-product, offers a promising substrate for industrial-scale production of xylooligosaccharides (XOS). Utilizing a method that includes pretreatment, microwave-assisted enzymatic hydrolysis, and purification, a range of XOS types such as X4, X3, and X2 have been successfully produced, with their identification confirmed through HPLC analysis [100]. This process exemplifies the innovative approaches being developed for XOS production, underscoring the ability of these methods to convert various plant biomass sources into valuable prebiotic compounds efficiently.

The field of agricultural by-product utilization for value-added compound production is gaining significant attention. XOS, recognized for their prebiotic properties, are among the compounds being derived from such agro-residues. For instance, Chapla et al. [101] showed that the β-xylanase enzyme from *Aspergillus foetidus* (*A. foetidus*) could produce XOS with a yield of 6.73 ± 0.23 mg/mL after an 8 h incubation at 45 °C, using corncob as the substrate. This process resulted in xylobiose and xylotriose as the main XOS forms. Additionally, De Menezes et al. [102] explored the capability of Pleurotus species, specifically *Pleurotus* sp. *BCCB068* and *Pleurotus tailandia*, in degrading the xylan component in oats, pointing to the industrial potential of these strains in transforming hemicellulosic agro-residual wastes into beneficial products. These studies collectively highlight the broad spectrum of substrates and methodologies being applied in the field, demonstrating the

vast potential for sustainable and efficient production of XOS and other prebiotics from agricultural by-products.

Dhiman et al. [103] took an innovative approach by immobilizing the crude enzyme from Pholiota adipose on silicon oxide nanoparticles. This technique allowed for the enzyme to be reused up to 17 cycles, showcasing its potential for efficient and sustainable industrial use. In a distinct method, da Silva Menezes et al. [104] utilized the *Aspergillus brasiliensis BLf1* (*A. brasiliensis*) strain to produce xylooligosaccharides (XOS) using rice husk as the substrate, achieving significant enzyme activity of 183.5 U/g substrate through solid-state fermentation. This approach proved particularly adept at processing the solid nature of rice husk, underscoring its effectiveness for XOS production from this agricultural by-product. Adsul et al. [105] documented a successful process for xylan hydrolysis using xylanase from *Streptomyces matensis*, applied to beech wood-derived xylan. This enzymatic treatment yielded xylobiose and xylotriose as primary XOS products, demonstrating the method's efficacy for generating XOS from wood-based sources.

Furthermore, the anaerobic fermentation of oat spelt xylan by *Butyrivibrio fibrisolvens* (*B. fibrisolvens*) *H17c*, known for xylan degradation, primarily produced xylobiose [105]. Another study by Adsul et al. found *Pseudozyma hubeiensis* (*P. hubeiensis*) *NCIM 3574* capable of producing xylanase, which efficiently converted xylan to XOS with varying degrees of polymerization (3–7 DP). Narisetty et al. [106] explored using corncob and wheat bran as substrates for producing xylanase via *Pichia stipitis* (*P. stipites*) through solid-state fermentation, highlighting the potential of diverse agricultural residues as sources for xylanase production and subsequent XOS generation. The hydrolysis of xylan by this enzyme resulted in an impressive 92% yield of XOS, including xylotetroase, xylotriose, and xylobiose. These studies collectively emphasize the versatility and potential of different agricultural residues as substrates for xylanase production and XOS generation, offering innovative and sustainable approaches for valorizing agricultural by-products into valuable prebiotic compounds.

Metagenomic techniques have been leveraged to identify and harness xylanase-encoding genes for XOS production, as seen in the work of Sun et al., who isolated a xylanase gene and expressed it in *Bacillus megaterium* (*B. megaterium*) *MS941*. This enzyme efficiently produced XOS with degrees of polymerization ranging from 2 to 4, showcasing the utility of metagenomic approaches in identifying novel enzymes for biotechnological applications [107]. Additionally, crude xylanase from *B. subtilis*, lacking β-xylosidase activity, proved effective in transforming sugarcane bagasse into pure forms of xylobiose, xylotriose, and xylotetraose, serving as prebiotic sources for promoting the growth of *Bifidobacterial strains* and the production of beneficial fatty acids [108].

Pigeon pea (*Cajanus cajan*) stalks have been explored for XOS production, with treatments using sodium hydroxide and steam achieving high xylan recovery rates. Subsequent enzymatic hydrolysis with xylanase under optimized conditions yielded xylobiose and xylotriose, demonstrating the potential of agricultural residues as sources for XOS [96]. The hydrolysis of sugarcane bagasse, following potassium hydroxide treatment and crude xylanase application from *Pichia stipitis*, also led to substantial XOS production [107]. Furthermore, a thermostable endoxylanase from *Streptomyces thermovulgaris* TISTR1948 was utilized to generate XOS from corncob, highlighting the effectiveness of thermostable enzymes in processing biomass for XOS production [109].

These varied studies underline the broad spectrum of agricultural residues as viable feedstocks for XOS production. Through the use of specific xylanases, optimization of processing conditions, and innovative production methods, the efficiency of XOS generation has been significantly enhanced. These advancements not only contribute to the valorization of agro-residual biomass, but also open up promising avenues for the application of XOS in the food and pharmaceutical sectors, marking a significant stride in the sustainable production of valuable prebiotic compounds (Table 3).

Table 3. Consolidated data on enzymatic XOS production, including enzyme sources, yields, xylan origins, and studies, crucial for refining production processes and understanding XOS production nuances for various applications.

SNo	Source of Enzyme	Yield	Source of Xylan	Reference
1	*B. megaterium MS941*	Enhancement 80%	Corn cob	[110]
2	*S. thermovulgaris*	162.97 mg/g	Corn cob	[111]
3	*P. stipites*	31.80%	Sugarcane bagasse	[112,113]
4	Enzyme applied in the production of xylooligosaccharides (XOS) was sourced from *B. subtilis*	A yield of 3.2 g of xylooligosaccharides (XOS) was obtained from 50 g of wheat bran	Wheat bran	[114]
5	*B. subtilis*	xylobiose 68.48 mg/g	Sugarcane bagasse	[108]
6	*T. viridae*	xylobiose 96%	Pigeon pea	[114]
7	*T.a viridae*	xylobiose 0.502 mg/mL	Pigeon pea	[115]
8	*T. viridae*	xylobiose 1.208 mg/mL	Corn cob	[116,117]

3.1.3. Production of Fructooligosaccharides (FOS) from Lignocellulosic Biomass: Methods and Progress

Fructooligosaccharides (FOS) are primarily obtained from plant-based materials, either through direct extraction or by enzymatic synthesis from common substrates such as sucrose or inulin. Singh et al. [118] detailed that the major enzymes used in FOS production are fructosyltransferase (E.C.2.4.1.99) for deriving FOS from biomass-derived sucrose and endoinulinase (E.C.3.2.1.7) for inulin-based FOS production. Aside from inulin and sucrose, alternative sources such as date biomass, maple syrup, and chicory inulin have been explored for FOS production.

Narisetty investigated the efficacy of commercial fructosyltransferase across various substrates, finding sucrose as the kinetically superior substrate for FOS generation [119]. Silva et al. [120] observed that inulinase from *Aspergillus niger* (*A. niger*) and *K. marxianus NRRL Y 7571* could effectively produce FOS from inulin, with specific yields of kestose (GF2), nystose (GF3), and fructosyl nystose (GF4) when treated with A. niger inulinase.

Further research by Nascimento et al. [121] revealed that *Penicillium citreonigrum* (*P. citreonigrum*) produces β-fructofuranosidase (EC 3.2.1.26), capable of converting sucrose to FOS with enhanced activity in the presence of Cu4+ ions, resulting in significant kestose production. Smaali et al. [122] took advantage of date by-products as a novel source for sucrose to synthesize FOS using β-fructofuranosidase (Ffase) from *Aspergillus awamori* (*A. awamori*) *NBRC4033*, achieving notable yields and demonstrating the efficiency of using agro-residue biomasses for FOS production.

These studies collectively emphasize the diverse strategies and resources available for FOS production, showcasing the adaptability of methods in utilizing various plant biomass sources for generating valuable prebiotic compounds efficiently and sustainably. The examples provided illustrate the potential of employing different agro-residues and immobilized enzymes for the industrial production of FOS, presenting a sustainable and efficient approach, as detailed across the summarized findings in these research efforts.

Transfructosylation, an enzymatic method for synthesizing fructooligosaccharides (FOS) from sucrose, has seen diverse approaches for improving yield and efficiency. Using endoinulinase in a biphasic butyl acetate/buffer system, a significant 60.2% FOS production was achieved, with nystose being the primary end-product from sucrose [123]. Maple syrup at 15°Bx served as a substrate for FOS production, with kestose as the main product. Increasing maple syrup concentration to 66°Bx enhanced the production of nystose and fructosyl-nystose [123]. Díez-Municio et al. [124] discovered that inulosucrase from *Lactobacillus gasseri* (*L. gasseri*) *DSM 20,604* could produce FOS and maltosylfructosides (MFOS) from sucrose and combinations of sucrose and maltose.

An innovative two-phase system combined levansucrase (from *Bacillus amyloliquefaciens*) with endo-inulinase (from *A. niger*) for short-chain FOS (scFOS) and oligolevans production, utilizing sucrose. This setup allowed levansucrase to create levans, while endo-inulinase controlled molecule size, with 6-kestose being the primary scFOS [125]. Immobilizing levansucrase enhanced levan production over scFOS, improving cost-effectiveness [125].

Soliman et al. [126] explored immobilizing inulinase from *A. niger* on polyurethane foam, using a pressurized liquefied petroleum gas (LPG) system for FOS synthesis from sucrose, achieving a 31% total FOS yield, including GF2, GF3, and GF4. Bersaneti et al. [127] revealed that levansucrase from *B. subtilis natto* could produce FOS and levan simultaneously, yielding 41.3 g/L of FOS and 86.9 g/L of levan in a bioreactor from a 350 g/L sucrose concentration. Huang et al. [128] reported *Aspergillus aculeatus M105* producing extracellular fructosyltransferase (FTase), achieving FOS yields of 67.54% and 65.47% (*w*/*w*) for extracellular and immobilized enzyme activities, respectively, with notable hydrolytic activity. These studies illustrate the ongoing innovation in FOS production, emphasizing various substrates, enzymes, and methods to enhance yield and application possibilities in the food and pharmaceutical industries.

Peña-Cardeña and colleagues, in publication [129], successfully synthesized fructooligosaccharides (FOS) of low molecular weight, including compounds such as 1-kestose, 6-kestose, neokestose, nystose, and f-nystose, by employing a specialized version of the enzyme inulosucrase, named IslA4, which was derived from *Leuconostoc citreum*, using sucrose as the substrate. Following a distinct methodology, Kralj and their team, documented in reference [130], demonstrated that a pair of fructosyltransferases labeled WDG185 are capable of producing FOS directly from inulin. Within this duo, the enzyme β-fructofuranosidase (FosA) is responsible for the creation of short-chain fructooligosaccharides ranging from GF_2 to GF_4, while inulosucrase (InuO) facilitates the production of inulooligosaccharides, with a range extending from GF_2 to GF_{24}, from sucrose. This process effectively mimics the natural synthesis of inulin found in plants. These studies highlight the versatility and potential of various enzymatic processes for producing FOS, a prebiotic component, from sucrose. The focus on innovative methods and substrates emphasizes the evolving nature of this field, offering new opportunities for the sustainable production of FOS (Figure 5).

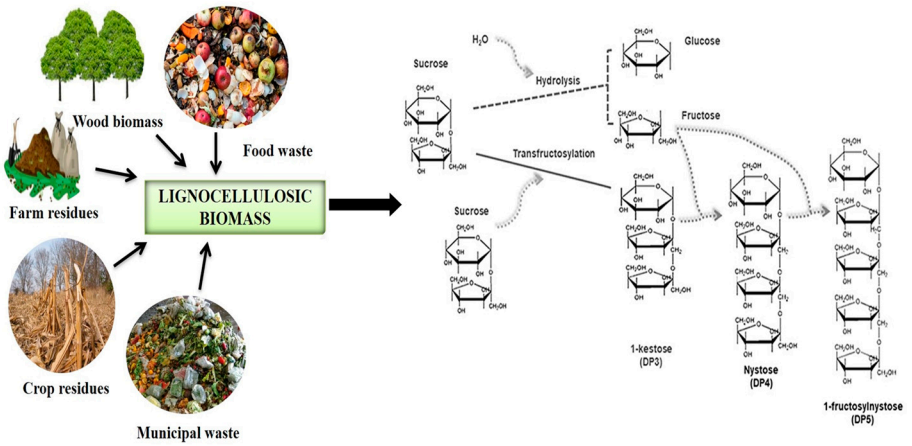

Figure 5. Innovations in producing fructooligosaccharides (FOS) from lignocellulosic biomass with bacterial and fungal enzymes.

The compound 1-kestose, recognized for its dual function as a volume enhancer and a potent sweetener, has undergone extensive investigation regarding its synthesis from inulin for fructooligosaccharide (FOS) production. A particular method of interest employs heat-resistant inulinase for generating solid-state fermentation (SSF) (E.C.2.4.199). In detailed research, a reaction mixture containing 2191 mM sucrose in a 0.1 mM potassium phosphate buffer (pH 8.0), with 0.18g of dry fungal mycelium per 100 mL, was incubated at a temperature of 55 ± 3 °C. This process led to the generation of roughly 644 mM

(equivalent to about 325 g/L) of 1-kestose after 12 h of incubation, achieving a conversion rate of 60% (*w*/*w*) of sucrose to FOS. Chen and Liu, in their study [131], explored the use of *Apostichopus japonicus* TIT-90076, which is noted for producing an enzyme with high transfructosylating activity, β-fructofuranosidase. They pinpointed the enzyme's transfructosylation activity's optimal pH range as 5.0 to 6.0 and the temperature range for peak activity between 55 ± 2 °C and 65 ± 2 °C, with sucrose being identified as the most effective substrate when present at a 25% concentration for maximal enzyme efficiency. Hirayama and coresearchers [132] centered their work on *A. niger ATCC* 20611 for FOS creation, introducing the culture into a fermentation broth with specific nutritional content, incubated at pH 6.0 and 28 ± 2 °C. Their method entailed the incubation of a 50% sucrose solution under specified conditions to yield FOS, with 1-kestoses detected at 8 h and a notable increase in nystose after 72 h of fermentation. Similarly, Patel and associates [133] examined *Fusarium oxysporum* for its fructosyltransferase production capability. These studies collectively illuminate the wide range of microbial sources and experimental conditions conducive for FOS production, particularly 1-kestose, underscoring the diverse approaches to optimizing synthesis parameters such as substrate concentration, temperature, pH, and microbial strains.

A wide array of microbial strains has been explored for their capacity to synthesize fructooligosaccharides (FOS) under tailored growth conditions. Takeda and colleagues, in study [134], enhanced Czapek's medium by adjusting its initial pH to 5.5, which led to a notable increase in fructosyl transferase activity, facilitating FOS synthesis as soon as 8 h. They discovered *Scopuloariopsis brevicaulis N-01* in soil, documenting its ability to produce a substantial 95.6 g/l of 1-kestose, achieving a conversion efficiency of 64.0% against a theoretical maximum of 85%. This efficiency notably surpasses that of *A. niger*, which only managed a 24% conversion rate for 1-kestose. Usami et al. [135] observed that *Penicillium frequentans* WU-1S could induce β-fructofuranosidase activity through fructose transfer, employing a conidial inoculum density of 10^6 per mL and conducting fermentation at 30 °C to reach a peak transfructosylation activity of 5.40 U/mL. Barthomeuf and Pourrat [136] detailed FOS generation from sucrose using crude fructosyltransferases (FTF) derived from *Penicillium rugulosum*, utilizing a process that required a 3-day incubation at 29–30 °C in Czapek's medium enriched with 3% sucrose, 1% NH_4Cl, and 0.75% soya peptone, and spore concentration of 108 per L at pH 5.5. They found that the crude enzyme (culture filtrate), a mix of FTF and glycosidase, swiftly achieved high FOS concentrations (650 g/L in 10 h), outperforming the combined enzyme system of FTF and glucose oxidase, which yielded 363 g/L in 25 h. Similarly, Yun et al. [137] investigated *Aureobasidium pullulans KFCC 10,245* for its fructosyltransferase production, with their fermentation medium comprising 20% sucrose, 1% yeast extract, 0.5% K_2HPO_4, 0.05% $MgSO_4 \cdot 7H_2O$, and 1% $NaNO_3$ at pH 6.5, noting enhanced enzyme activity with a culture medium containing 55% (*w*/*w*) Mg^{2+}. Hayeshi et al. [138] delved into immobilizing free fructosyl transferring enzyme from *Aureobasidium ATCC 20,524* on volcanic ash. Fujita and team [139] explored FOS synthesis through the transfructosylating activity of β-fructofuranosidase (E.C.3.2.1.2.6) from *Arthrobacter* sp. *K1*, which was grown over two days at 30 °C and 110 rpm in a medium with specific nutrient concentrations, followed by fermentation at 37 °C for 25 h with aeration at 6 L/min. These investigations collectively underscore the vast diversity of microbial sources and cultivation parameters deployable for FOS generation, spotlighting the strategic optimization of fermentation conditions such as pH, temperature, and nutrient makeup to maximize FOS yield and production efficiency, crucial for applications in the food and pharmaceutical sectors (Table 4).

Table 4. Summary of enzymatic FOS production, including enzyme sources, yields, processes, and references, serving as a guide to improve production and understand its complexity.

Sno	Source of Enzyme	Yield	Process	Reference
1	*L. gasseri*	45%	Inulosucrase enzyme	[140]
2	*A. awamori*	123 g/L	β-fructofuranosidase enzyme immobilized on chitosan	[140]
3	*B. subtilis*	41 g/L	Levansucrase enzyme	[141]
4	*K. mycesmarxianus*	kestose (12%), nystose (21%)	Inulinase enzyme	[142]
5	*P. citreonigrum*	59 g/L	β-fructofuranosidase enzyme	[143]
6	*A. niger*	60%	Endoinulinase Enzyme	[144]
7	*A. niger*	31%	Inulinase enzyme	[145,146]

3.1.4. Advancements in Extracting Mannooligosaccharides (MOS) from Lignocellulosic Biomass: Techniques and Developments

The production of mannooligosaccharides (MOS) through the enzymatic treatment of agricultural waste, such as copra meal, highlights a significant advancement in the utilization of renewable resources for generating valuable prebiotic compounds. In the study by Sathitkowitchai et al., the team utilized a genetically engineered endo-β-(1,4)-mannanase, derived from *B. subtilis* but expressed in *Escherichia coli*, demonstrating the enzyme's capacity to effectively target and break down mannans into MOS with degrees of polymerization ranging from 4 to 7 [147]. This process emphasizes the enzyme's high stability and activity under specific conditions, showcasing its potential for scalable production.

Ghosh and colleagues adopted a different approach by using endo-mannanase from *Clostridium thermocellum*, also expressed in *E. coli*, for the hydrolysis of both pretreated and defatted copra meal. This method resulted in the production of 40% mannobiose and 18% mannotriose [148], highlighting the efficiency of the enzyme in converting complex carbohydrates into simpler, valuable oligosaccharides.

These studies collectively illustrate the innovative use of enzymatic hydrolysis for MOS production from underutilized agricultural by-products. They underscore the potential of specific microbial enzymes in transforming plant-based mannans into MOS, offering a sustainable pathway for the development of prebiotics with significant health benefits. The research on MOS not only contributes to the field of nutrition and health, but also presents an eco-friendly alternative for waste biomass valorization.

The innovative research by Jian and their team in 2013 demonstrated the use of galactomannan gum from Gleditsia sinensis for the production of MOS using β-mannanase. Achieving a 75.9% yield of MOS with a degree of polymerization ranging from 1 to 5 after 34.1 h at 57.4 ± 2 °C, this study underscores the potential of utilizing diverse substrates and enzymatic processes for MOS synthesis [149]. The emphasis on optimizing reaction conditions such as temperature, pH, and enzyme selection is indicative of the advances in this field, aiming for sustainable and efficient production of MOS from agro-waste biomass.

Furthermore, the work by Cescutti et al. highlighted the role of β-mannosidase in the hydrolytic cleavage of internal β-1,4-glycosidic bonds, facilitating MOS production [150]. The recent trend towards exploiting underused agricultural biomasses, such as konjac glucomannan (KGM) polysaccharides, for oligosaccharide production, especially focusing on the enzymes from fecal bacteria, marks a significant shift in the approach to prebiotic synthesis. This exploration of various microbial enzymes and substrates enriches the potential for creating valuable prebiotics from sustainable sources, pointing towards innovative applications in food and pharmaceutical industries.

The study by Albrecht et al. in 2011 performed a comparative analysis to evaluate the effectiveness of enzymes from fecal bacteria and fungi, specifically endo-β-(1,4)-glucanase (EC 3.2.1.91) and endo-β-(1,4)-mannanase, in catalyzing oligosaccharide formation. The research demonstrated significant differences in substrate specificity between the two

enzyme sources. Fungal enzymes showed targeted activity towards mannose- and glucose-containing linkages in konjac glucomannan polysaccharides, unlike the fecal bacterial enzymes, which lacked such specificity. The fungal enzymes produced a wide array of hydrolysis outcomes, including mannose and various oligosaccharides, showcasing their ability to break down the polysaccharide into smaller molecules effectively. This distinction in enzyme action is crucial for understanding the enzymatic breakdown of complex polysaccharides and the subsequent production of oligosaccharides. The study highlights the importance of selecting specific enzymes for their efficiency in hydrolysis and their impact on the composition of the resulting oligosaccharides. The findings from this research contribute significantly to the field, providing insights into optimizing the enzymatic production of MOS from agricultural biomass for industrial applications.

The exploration of MOS synthesis from various plant-based sources, including the intriguing utilization of galactoglucomannan from pine craft biomass and konjac flour, showcases significant advancements in biotechnology and sustainable resource use. Tenkanen and colleagues harnessed endo-β-D-mannase from *Trichoderma reesei*, effectively targeting pine craft pulp to produce mannooligosaccharides such as mannobiose, mannotrioses, and mannotetroses (Figure 6). The purified oligosaccharides, achieved through size-exclusion, anion exchange, and carbon chromatography, demonstrate the potential of fungal enzymes in the selective breakdown and conversion of complex polysaccharides into valuable prebiotics [151].

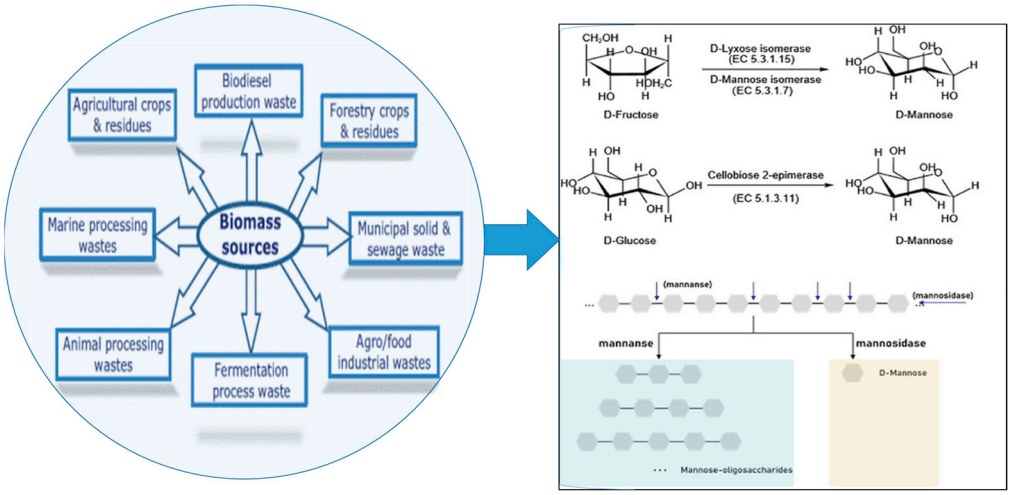

Figure 6. Advances in mannooligosaccharides (MOS) production from lignocellulosic biomass using bacterial and fungal enzymes.

Further extending the scope, Yang et al. [152] isolated endo-1,4-β-mannanase from *Talaromyces cellulolyticus* (*T. cellulolyticus*), which showed notable activity on konjac flour, converting a significant portion of the substrate into MOS with a wide range of polymerization degrees under optimized conditions. This indicates the enzyme's robustness and effectiveness in MOS synthesis from nontraditional substrates [152]. Cao et al. further explored the use of endo-β-mannanase from *Bacillus* sp. *MSJ-5* on konjac flour, achieving a remarkable conversion rate into MOS with specific degrees of polymerization, underlining the enzyme's efficiency and the feasibility of using konjac flour as a rich source for MOS production [152].

These studies collectively illuminate the versatility and potential of employing specialized enzymes for the efficient and sustainable production of MOS from diverse agricultural and plant biomasses. The focus on optimizing enzymatic processes, reaction conditions, and the choice of substrates highlights significant strides towards advancing the enzymatic production of MOS, with broad implications for their use in various industrial applications, particularly in the food and pharmaceutical sectors, where there's a growing demand for naturally derived prebiotic compounds.

The utilization of β-mannanase from *Aspergillus oryzae* for generating MOS from copra meal, as demonstrated by Jana et al. [153], exemplifies the potential of microbial enzymes in transforming agricultural by-products into valuable prebiotic compounds. Similarly, Blibech et al. [154] showed the effective production of MOS from locust bean gum using *Penicillium occitanis* mannanase, further enhanced by immobilizing the enzyme onto chitin. Oda and Tonomura [155] leveraged copra meal for mannase extraction from *Bacillus circulans NT 6.7*, converting mannan-rich waste into MOS, indicating its prebiotic value. *Oda and Tonomura* [155] explored the optimal conditions for MOS synthesis using enzymes from *Trichosporon cutaneum (T. cutaneum) JCM 2947, Trichoderma viridae* (*T. viridae*), finding effective temperature and pH ranges for β-mannanase and β-mannosidase activities.

These studies collectively illuminate the diverse microbial sources and substrates viable for MOS production, emphasizing the need for optimized reaction conditions and specific enzyme selection. This body of research significantly contributes to advancing MOS utilization as a functional ingredient, underscoring the importance of sustainable and renewable biomass sources in various industrial applications (Table 5).

Table 5. Details of enzyme sources and mannan types for MOS production, aiding in method and material selection for specific needs.

SNo	Source of Enzyme	Yield	Source of Mannan	Reference
1	*C. thermocellum*	40% Mannobiose and 18% Mannotriose	Copra meal	[156]
2	*G. sinensis*	29.1 g/L	Galactomannan gum	[157]
3	*T. viridae*	Trimers (27%), Tetramers (6%), and Pentamers (3%)	Konjac glucomannan	[158]
4	*T. cellulolyticus*	71.2%	Konjac flour	[159]
5	*B. subtilis*	8.25%	Copra meal	[160]

3.1.5. Innovations in Producing Isomaltooligosaccharides (IMOs) from Lignocellulosic Biomass: Processes and Technological Progress

The synthesis of isomaltooligosaccharides (IMOs) using enzyme batch systems has been effectively optimized in various research initiatives. Rabelo and colleagues conducted a study utilizing a partially purified dextransucrase enzyme from *Leuconostoc mesenteroides NRRL B-512F*. They identified the optimal conditions for IMO production to be a sucrose concentration of 200 mmol/L and an enzyme activity of 1 U/mL. Under these conditions, they achieved an IMO concentration of 64.42 mmol/L, with a productivity rate of 42.5 mmol/L/h, demonstrating the efficiency of this method and its potential for cost-effective commercial IMO production [161]. The pursuit of alternative sources and methods for IMO production emphasizes the importance of sustainable substrate availability. The use of biomass as a substrate, combined with enzymatic catalysis, highlights a move towards more environmentally friendly production processes. The enzymatic production of IMOs typically involves saccharification and hydrolysis processes, employing a variety of amylase enzymes or combinations thereof, often described as enzyme cocktails. These enzymes saccharify substrates from disaccharides and oligosaccharides to polysaccharides, converting them into simpler sugar units that constitute IMOs [162].

These advancements in IMO synthesis underline the significance of optimizing reaction conditions, such as substrate concentration and enzyme activity, to enhance production efficiency and sustainability. The focus on utilizing renewable resources and developing eco-friendly processes for IMO production reflects the growing demand for sustainable industrial practices, particularly in the food and nutraceutical industries, where IMOs are valued for their functional and health-promoting properties. Utilizing microorganisms instead of isolated enzymes for the production of isomaltooligosaccharides (IMOs) presents a cost-effective and efficient approach. *Aspergillus oryzae TISTR 3102* enzymes were used in a solid-state fermentation process with rice and cassava, leveraging the specific enzymes α-amylase (EC 3.2.1.1) and α-glucosidase (EC 3.2.1.20) for saccharification and hydrolysis,

respectively. This method led to the production of syrups rich in various IMOs, including isomaltose, panose, and isomaltotriose, after 5 days of incubation [163].

Chockchaisawasdee and Poosaran explored a three-step enzymatic process with enzyme cocktails for IMO production from banana flour. This involved initial liquefaction with Termamyl SC, saccharification using Fungamyl 800 L and barley β-amylase, and transglucosidation with Transglucosidase L. The combination of Fungamyl 800 L and barley β-amylase was particularly effective, yielding high levels of IMOs [164]. Saman et al. further scaled up this approach, using a 5 L fermenter and substrates such as nonglutinous rice flour, glutinous rice flour, and cassava starch for IMO production. The process included α-amylase for liquefaction, β-amylase and pullulanase for saccharification, and transglucosidase for transglycosylation, with nonglutinous rice flour yielding the highest amounts of IMOs such as isomaltose, isomaltotriose, and panose [165]. These studies demonstrate the versatility and potential of microbial fermentation and enzyme cocktails for sustainable and efficient IMO production (Figure 7). The optimization of process conditions and enzyme selection is pivotal for the industrial-scale manufacturing of IMOs, highlighting the progress and possibilities in this domain for creating valuable prebiotic oligosaccharides. The adoption of recombinant enzymes to enhance the production of isomaltooligosaccharides (IMOs) represents a significant advancement in biotechnology. Kaulpiboon et al. used a 30% (*w*/*v*) concentration of tapioca starch as a substrate, treated with a combination of pullulanase, a mutated amylomaltase (Y101S), and transglucosidase from *A. niger.* This unique blend of enzymes, especially the synergistic use of both wild-type and mutated amylomaltase with transglucosidase, enabled the efficient production of long-chain IMOs under optimal conditions (pH 7.0 and 40 °C) [166]. Basu et al. introduced a simultaneous saccharification and transglucosylation (SST) method for starch-based IMO production, optimizing enzyme mixture dosages using the Nelder–Mead simplex algorithm. Their approach, especially on substrates such as potato processing waste and broken rice with specific enzyme dosages, significantly boosted IMO production, highlighting the effectiveness of the SST method in enhancing yields [167].

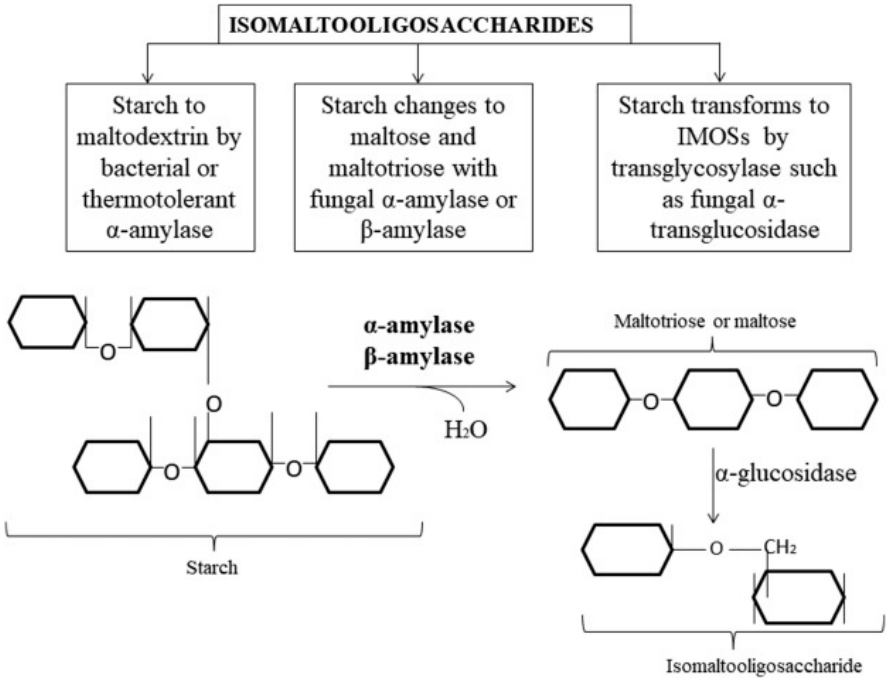

Figure 7. Innovations in producing isomaltooligosaccharides (IMOs) from lignocellulosic biomass using bacterial and fungal enzyme.

Cui et al. [168] explored the synthesis of IMOs from Chinese chestnut starch slurry, employing a sequential process involving heat-stable α-amylase, followed by fungal α-

amylase, β-amylase, pullulanase for saccharification, and α-transglucosidase for transglycosylation. This comprehensive enzymatic process yielded a considerable percentage of chestnut-derived IMOs, demonstrating the method's efficiency and the prebiotic potential of the produced IMOs. These studies underscore the critical role of optimizing enzymatic processes, including temperature, pH, and enzyme dosage, in maximizing IMO yields. The strategic employment of various enzymes, tailored to specific substrates and desired oligosaccharide profiles, signifies the complex yet efficient approach needed for effective IMO production. Such research is pivotal in pushing forward the industrial production of IMOs, focusing on process optimization for enhanced yields, cost-effectiveness, and sustainability. Panose, an isomaltooligosaccharide composed of three glucose units linked by α(1→4) and α(1→6) glycosidic bonds, is recognized for its distinctive structure affecting its physical and biological attributes (Table 6). Its production predominantly occurs through transglucosylation, a process effectively detailed by Prapula et al. [169], which involves transferring a glucose residue from one molecule to another, using specific microorganisms such as *Aureobasidium pullulans* strain *KFCC10245*. This strain catalyzes the conversion of substrates to panose under controlled conditions, including a specialized growth medium and optimal temperature and maltose concentration adjustments, showcasing a method conducive to industrial-scale production [169]. Further innovation in panose production involves genetic engineering techniques to enhance efficiency. Casa-Villegas et al. [170] made significant advancements by genetically modifying *Saccharomyces cerevisiae* cells to act as catalytic agents for panose synthesis. This was accomplished by integrating the aglA gene, encoding for glucosidase, with glycophosphatidylinositol (GPI) anchor sequences from the SED1 gene, resulting in a hybrid protein anchored to the yeast cell membrane. This modification improves the stability and efficiency of the enzyme, offering a promising approach for industrial IMO production [170]. These developments underscore the integration of microbiology, genetic engineering, and process optimization in the production of valuable oligosaccharides such as panose. Such strides in biotechnological and industrial biochemistry fields highlight the potential for scalable, efficient production of prebiotic compounds, contributing to advancements in food and health industries.

Table 6. Overview of IMOS production from various materials, detailing yields as efficiency indicators for different processes and their impact on gut health.

SNo	Substrate	Yield	Reference
1	Nonglutinous rice flour	169 g/L	[171]
2	Potato processing waste	93 g/L	[172]
3	Tapioca starch	68 g/L	[173]
4	Banana flour	77 g/L	[174]

4. Conclusions

Transforming lignocellulosic biomass into valuable prebiotics such as galactooligosaccharides, fructooligosaccharides, xylooligosaccharides, isomaltooligosaccharides, and mannooligosaccharides is a crucial step towards creating a sustainable bio-economy and enhancing environmental protection. The emphasis on biological methods, particularly enzymatic treatments, presents an eco-friendly alternative to chemical synthesis. Despite the potential, the synthesis of these oligosaccharides confronts challenges such as the lower rate of transglycosylation compared to hydrolysis, where enzymes tend to break down the oligosaccharide products more rapidly than they can synthesize them.

To improve the efficiency of these enzymatic reactions, strategies such as using activated glycosyl donors to enhance the transglycosylation process have been considered. However, overcoming obstacles such as the high costs associated with the initial pretreatment of lignocellulosic biomass remains a significant hurdle for large-scale production. Further research is needed to improve product selectivity, ensuring high yield and purity of desired oligosaccharides, and to refine purification and separation techniques to boost the production process's cost-effectiveness and environmental sustainability.

Addressing these challenges is crucial for leveraging lignocellulosic biomass as a source of valuable prebiotic oligosaccharides, contributing to a more sustainable and eco-friendly bio-economy. This endeavor not only opens up new avenues for the production of health-promoting prebiotics, but also aligns with global efforts to utilize renewable resources efficiently and reduce reliance on nonrenewable inputs.

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References

1. Chourasia, R.; Phukon, L.C.; Singh, S.P.; Rai, A.K.; Sahoo, D. Role of enzymatic bioprocesses for the production of functional food and nutraceuticals. In *Biomass, Biofuels, Biochemical*; Elsevier: Amsterdam, The Netherlands, 2020; pp. 309–334.
2. Khaneghah, A.M.; Fakhri, Y. Probiotics and prebiotics as functional foods: State of the art. *Curr. Nutr. Food Sci.* **2019**, *15*, 20–30. [CrossRef]
3. Chen, Z.; Zhu, S.; Gao, S.; Sun, C.; Tian, Z.; Wen, X.A. Hyperthermophilic anaerobic fermentation platform for highly efficient short chain fatty acids production from thermal hydrolyzed sludge. *Water Res.* **2023**, *243*, 120434. [CrossRef] [PubMed]
4. Chelliah, R.; Park, S.J.; Oh, S.; Lee, E.; Daliri, E.B.M.; Elahi, F.; Park, C.R.; Sultan, G.; Madar, I.H.; Oh, D.H. Unveiling the potentials of bioactive oligosaccharide1-kestose (GF2) from Musa paradisiaca Linn peel with an anxiolytic effect based on gut microbiota modulation in stressed mice model. *Food Biosci.* **2022**, *49*, 101881. [CrossRef]
5. Wang, Y.; Liu, Y.; Polic, I.I.; Matheyambath, A.C.; LaPointe, G. Modulation of human gut microbiota composition and metabolites by arabinogalactan and *Bifidobacterium longum* subsp. longum BB536 in the Simulator of the Human Intestinal Microbial Ecosystem (SHIME®). *J. Funct. Foods* **2021**, *87*, 104820. [CrossRef]
6. Patra, R.; Mitra, S.; Das, N.C.; Mukherjee, S. Prebiotics as promising therapeutics for treating gut-related disorders: Biochemical and molecular perspectives. In *Prebiotics, Probiotics and Nutraceuticals*; Springer Nature: Singapore, 2022; pp. 133–154.
7. Bamigbade, G.B.; Subhash, A.J.; Kamal-Eldin, A.; Nyström, L.; Ayyash, M. An updated review on prebiotics: Insights on potentials of food seeds waste as source of potential prebiotics. *Molecules* **2022**, *27*, 5947. [CrossRef]
8. Marco, M.L.; Sanders, M.E.; Gänzle, M.; Arrieta, M.C.; Cotter, P.D.; De Vuyst, L.; Hutkins, R. The International Scientific Association for Probiotics and Prebiotics (ISAPP) consensus statement on fermented foods. *Nat. Rev. Gastroenterol. Hepatol.* **2021**, *18*, 196–208. [CrossRef] [PubMed]
9. Yi, T.; Zhao, H.; Mo, Q.; Pan, D.; Liu, Y.; Huang, L.; Xu, H.; Hu, B.; Song, H. From cellulose to cellulose nanofibrils—A comprehensive review of the preparation and modification of cellulose nanofibrils. *Materials* **2020**, *13*, 5062. [CrossRef]
10. Hough, C. *Regional Review on Status and Trends in Aquaculture Development in Europe–2020*; Food & Agriculture Org: Rome, Italy, 2022.
11. Panwar, D.; Panesar, P.S.; Saini, A. Prebiotics and their Role in Functional Food Product Development. In *Probiotics, Prebiotics and Synbiotics: Technological Advancements Towards Safety and Industrial Applications*; John Wiley & Sons Ltd.: Hoboken, NJ, USA, 2022; pp. 233–271.
12. Barclay, A.W.; Augustin, L.S.; Brighenti, F.; Delport, E.; Henry, C.J.; Sievenpiper, J.L.; Usic, K.; Yuexin, Y.; Zurbau, A.; Wolever, T.M.; et al. Dietary glycaemic index labelling: A global perspective. *Nutrients* **2021**, *13*, 3244. [CrossRef] [PubMed]

13. Augustin, L.S.; Aas, A.M.; Astrup, A.; Atkinson, F.S.; Baer-Sinnott, S.; Barclay, A.W.; Jenkins, D.J. Dietary fibre consensus from the international carbohydrate quality consortium (ICQC). *Nutrients* **2020**, *12*, 2553. [CrossRef]
14. Aguilar-Pérez, K.M.; Ruiz-Pulido, G.; Medina, D.I.; Parra-Saldivar, R.; Iqbal, H.M. Insight of nanotechnological processing for nano-fortified functional foods and nutraceutical—Opportunities, challenges, and future scope in food for better health. *Crit. Rev. Food Sci. Nutr.* **2023**, *63*, 4618–4635. [CrossRef]
15. Sarker, T.R.; Pattnaik, F.; Nanda, S.; Dalai, A.K.; Meda, V.; Naik, S. Hydrothermal pretreatment technologies for lignocellulosic biomass: A review of steam explosion and subcritical water hydrolysis. *Chemosphere* **2021**, *284*, 131372. [CrossRef] [PubMed]
16. Cecchi, T.; De Carolis, C.; Cecchi, T.; de Carolis, C. Biocascading: General Strategy for the Recovery of Valuable Substances from Food Waste. In *Biobased Products from Food Sector Waste: Bioplastics, Biocomposites, and Biocascading*; Springer: Cham, Switzerland, 2021; pp. 109–167.
17. Chatterjee, B.; Mazumder, D. Enzyme mediated hydrolysis of organic fraction of municipal solid waste (OFMSW): A short review. *J. Indian Chem. Soc.* **2018**, *95*, 295–312.
18. Bekhit, A.E.A.; Cheng, V.J.; Harrison, R.; Ye, Z.J.; Bekhit, A.A.; Ng, T.B.; Kong, L.M. Technological aspects of by-product utilization. In *Valorization of Wine Making By-Products*; CRC Press: Boca Raton, FL, USA, 2016; Volume 4, p. 117.
19. Shahidi, F.; Varatharajan, V.; Peng, H.; Senadheera, R. Utilization of marine by-products for the recovery of value-added products. *J. Food Bioact.* **2019**, *6*. [CrossRef]
20. Cantero, D.; Jara, R.; Navarrete, A.; Pelaz, L.; Queiroz, J.; Rodríguez-Rojo, S.; Cocero, M.J. Pretreatment processes of biomass for biorefineries: Current status and prospects. *Annu. Rev. Chem. Biomol. Eng.* **2019**, *10*, 289–310. [CrossRef] [PubMed]
21. Sing, R.D.; Nadar, C.G.; Muir, J.; Arora, A. Green and clean process to obtain low degree of polymerization xylooligosaccharides from almond shell. *J. Clean. Prod.* **2019**, *241*, 118237. [CrossRef]
22. Dávila, I.; Gordobil, O.; Labidi, J.; Gullón, P. Assessment of suitability of vine shoots for hemicellulosic oligosaccharides production through aqueous processing. *Bioresour. Technol.* **2016**, *211*, 636–644. [CrossRef]
23. Surek, E.; Buyukkileci, A.O. Production of xylooligosaccharides by autohydrolysis of hazelnut (*Corylus avellana* L.) shell. *Carbohydr. Polym.* **2017**, *174*, 565–571. [CrossRef] [PubMed]
24. Garrote, G.; Domínguez, H.; Parajó, J.C. Production of substituted oligosaccharides by hydrolytic processing of barley husks. *Ind. Eng. Chem. Res.* **2004**, *43*, 1608–1614. [CrossRef]
25. Vegas, R.; Alonso, J.L.; Domínguez, H.; Parajó, J.C. Processing of rice husk autohydrolysis liquors for obtaining food ingredients. *J. Agric. Food Chem.* **2004**, *52*, 7311–7317. [CrossRef]
26. Gullón, B.; Eibes, G.; Dávila, I.; Moreira, M.T.; Labidi, J.; Gullón, P. Hydrothermal treatment of chestnut shells (*Castanea sativa*) to produce oligosaccharides and antioxidant compounds. *Carbohydr. Polym.* **2018**, *192*, 75–83. [CrossRef]
27. Devi, A.; Bajar, S.; Kour, H.; Kothari, R.; Pant, D.; Singh, A. Lignocellulosic biomass valorization for bioethanol production: A circular bioeconomy approach. *Bioenergy Res.* **2022**, *15*, 1820–1841. [CrossRef]
28. Šekuljica, N.; Jakovetić Tanasković, S.; Mijalković, J.; Simović, M.; Pavlović, N.; Đorđević, N.; Knežević-Jugović, Z. Xylanase Production by Solid-State Fermentation for the Extraction of Xylooligosaccharides from Soybean Hulls §. *Food Technol. Biotechnol.* **2023**, *61*, 439–450. [CrossRef]
29. Dai, X.J.; Liu, M.Q.; Jin, H.X.; Jing, M.Y. Optimisation of solid-state fermentation of *Aspergillus niger* JL-15 for xylanase production and xylooligosaccharides preparation. *Czech J. Food Sci.* **2011**, *29*, 557–567. [CrossRef]
30. Cano, M.E.; García-Martin, A.; Comendador Morales, P.; Wojtusik, M.; Santos, V.E.; Kovensky, J.; Ladero, M. Production of oligosaccharides from agrofood wastes. *Fermentation* **2020**, *6*, 31. [CrossRef]
31. Guo, J.; Cao, R.; Huang, K.; Xu, Y. Comparison of selective acidolysis of xylan and enzymatic hydrolysability of cellulose in various lignocellulosic materials by a novel xylonic acid catalysis method. *Bioresour. Technol.* **2020**, *304*, 122943. [CrossRef]
32. Iqbal, M.W.; Riaz, T.; Mahmood, S.; Liaqat, H.; Mushtaq, A.; Khan, S.; Qi, X. Recent advances in the production, analysis, and application of galacto-oligosaccharides. *Food Rev. Int.* **2023**, *39*, 5814–5843. [CrossRef]
33. Cao, R.; Liu, X.; Guo, J.; Xu, Y. Comparison of various organic acids for xylo-oligosaccharide productions in terms of pKa values and combined severity. *Biotechnol. Biofuels* **2021**, *14*, 69. [CrossRef]
34. Gullón, P.; Gullón, B.; Muñiz-Mouro, A.; Lú-Chau, T.A.; Eibes, G. Valorization of horse chestnut burs to produce simultaneously valuable compounds under a green integrated biorefinery approach. *Sci. Total Environ.* **2020**, *730*, 139143. [CrossRef]
35. Sulaiman, S.H.A.S. Determination of Oxidative Stress Levels and Some Antioxidant Activities in Acute and Chronic Renal Failure Patients. Master's Thesis, Fen Bilimleri Enstitüsü, Mersin, Turkey, 2021.
36. Pinales-Márquez, C.D.; Rodríguez-Jasso, R.M.; Araújo, R.G.; Loredo-Trevino, A.; Nabarlatz, D.; Gullón, B.; Ruiz, H.A. Circular bioeconomy and integrated biorefinery in the production of xylooligosaccharides from lignocellulosic biomass: A review. *Ind. Crops Prod.* **2021**, *162*, 113274. [CrossRef]
37. Santibáñez, L.; Henríquez, C.; Corro-Tejeda, R.; Bernal, S.; Armijo, B.; Salazar, O. Xylooligosaccharides from lignocellulosic biomass: A comprehensive review. *Carbohydr. Polym.* **2021**, *251*, 117118. [CrossRef] [PubMed]
38. Dikkala, P.K.; Mummaleti, G.; Rajput, R.; Modi, R.; Sarma, C.; Himabindu, G.; Srinivas, Y.; Sharma, M.; Gupta, V.K.; Usmani, Z.; et al. Microbial Fructo-Oligosaccharides Derived from Agri-Food Waste. In *Microbial Bioprocessing of Agri-food Wastes*; CRC Press: Boca Raton, FL, USA, 2023.

39. Antia, U.E.; Stephen, N.U.; Onilude, A.A.; Udo, I.O.M.; Amande, T.J. Bioconvertibility of mannan-containing polysaccharides to bioethanol: A comparative study of palm kernel cake and copra meal feedstocks. *Biomass Convers. Biorefin.* **2023**, *13*, 5175–5186. [CrossRef]
40. Dwivedi, S.; Tanveer, A.; Yadav, S.; Anand, G.; Yadav, D. Agro-Wastes for Cost Effective Production of Industrially Important Microbial Enzymes: An Overview. In *Microbial Biotechnology: Role in Ecological Sustainability and Research*; John Wiley & Sons Ltd.: Hoboken, NJ, USA, 2022; pp. 435–460.
41. Banu, R.; Kumar, G.; Gunasekaran, M.; Kavitha, S. (Eds.) *Food Waste to Valuable Resources: Applications and Management*; Academic Press: Cambridge, MA, USA, 2020.
42. Zhang, W.; You, Y.; Lei, F.; Li, P.; Jiang, J. Acetyl-assisted autohydrolysis of sugarcane bagasse for the production of xylo-oligosaccharides without additional chemicals. *Bioresour. Technol.* **2018**, *265*, 387–393. [CrossRef]
43. Khuwijitjaru, P.; Pokpong, A.; Klinchongkon, K.; Adachi, S. Production of oligosaccharides from coconut meal by subcritical water treatment. *Food Sci. Technol.* **2014**, *49*, 1946–1952. [CrossRef]
44. Klinchongkon, K.; Khuwijitjaru, P.; Wiboonsirikul, J.; Adachi, S. Extraction of oligosaccharides from passion fruit peel by subcritical water treatment. *J. Food Process Eng.* **2017**, *40*, e12269. [CrossRef]
45. Gómez, B.; Míguez, B.; Veiga, A.; Parajó, J.C.; Alonso, J.L. Production, purification, and in vitro evaluation of the prebiotic potential of arabinoxylooligosaccharides from brewer's spent grain. *J. Agric. Food Chem.* **2015**, *63*, 8429–8438. [CrossRef] [PubMed]
46. Sato, N.; Takano, Y.; Mizuno, M.; Nozaki, K.; Umemura, S.; Matsuzawa, T.; Amano, Y.; Makishima, S. Production of feruloylated arabino-oligosaccharides (FA-AOs) from beet fiber by hydrothermal treatment. *J. Supercrit. Fluids* **2013**, *79*, 84–91. [CrossRef]
47. Lama, A.; Rodríguez, G.; Rubio, F.; Fernández, J. Production, characterization and isolation of neutral and pectic oligosaccharides with low molecular weights from olive by-products thermally treated. *Food Hydrocoll.* **2012**, *28*, 92–104. [CrossRef]
48. Wang, X.; Chen, Q.; Lü, X. Pectin extracted from apple pomace and citrus peel by subcritical water. *Food Hydrocoll.* **2014**, *38*, 129–137. [CrossRef]
49. Martínez, M.; Yáñez, R.; Alonso, J.L.; Parajó, J.C. Chemical production of pectic oligosaccharides from orange peel wastes. *Ind. Eng. Chem. Res.* **2010**, *49*, 8470–8476. [CrossRef]
50. Gómez, B.; Gullón, B.; Yáñez, R.; Parajó, J.C.; Alonso, J.L. Pectic oligosacharides from lemon peel wastes: Production, Purification, and Chemical Characterization. *J. Agric. Food Chem.* **2013**, *61*, 10043–10053. [CrossRef]
51. Meyer, T.S.M.; Miguel, Â.S.M.; Fernández, D.E.R.; Ortiz, G.M.D. Biotechnological production of oligosaccharides—Applications in the food industry. *Food Prod. Ind.* **2015**, *2*, 25–78.
52. Martins, G.N.; Ureta, M.M.; Tymczyszyn, E.E.; Castilho, P.C.; Gomez-Zavaglia, A. Technological aspects of the production of fructo and galacto-oligosaccharides. Enzymatic synthesis and hydrolysis. *Front. Nutr.* **2019**, *6*, 78. [CrossRef] [PubMed]
53. Crawford, C.; Brown, L.L.; Costello, R.B.; Deuster, P.A. Select dietary supplement ingredients for preserving and protecting the immune system in healthy individuals: A systematic review. *Nutrients* **2022**, *14*, 4604. [CrossRef]
54. Balzano, T. Active Clinical Trials in Hepatic Encephalopathy: Something Old, Something New and Something Borrowed. *Neurochem. Res.* **2023**, *48*, 2309–2319. [CrossRef]
55. Vignesh, A.; Amal, T.C.; Selvakumar, S.; Vasanth, K. Unraveling the role of medicinal plants and Gut microbiota in colon cancer: Towards microbiota-based strategies for prevention and treatment. *Health Sci. Rev.* **2023**, *9*, 100115. [CrossRef]
56. Song, H.; Guo, R.; Sun, X.; Kou, Y.; Ma, X.; Chen, Y.; Wu, Y. Xylooligosaccharides from corn cobs alleviate loperamide-induced constipation in mice via modulation of gut microbiota and SCFA metabolism. *Food Funct.* **2023**, *14*, 8734–8746. [CrossRef] [PubMed]
57. Ashaolu, T.J. Immune boosting functional foods and their mechanisms: A critical evaluation of probiotics and prebiotics. *Biomed. Pharmacother.* **2020**, *130*, 110625. [CrossRef]
58. Mojikon, F.D.; Kasimin, M.E.; Molujin, A.M.; Gansau, J.A.; Jawan, R. Probiotication of Nutritious Fruit and Vegetable Juices: An Alternative to Dairy-Based Probiotic Functional Products. *Nutrients* **2022**, *14*, 3457. [CrossRef]
59. Ajibola, O.O.; Thomas, R.; Bakare, B.F. Selected fermented indigenous vegetables and fruits from Malaysia as potential sources of natural probiotics for improving gut health. *Food Sci. Hum. Wellness* **2023**, *12*, 1493–1509. [CrossRef]
60. Kherade, M.; Solanke, S.; Tawar, M.; Wankhede, S. Fructooligosaccharides: A comprehensive review. *J. Ayurvedic Herb. Med.* **2021**, *7*, 193–200. [CrossRef]
61. Steinbach, E. Investigation of the Duodenojejunal Microbiome in Human Obesity. Ph.D. Dissertation, Sorbonne Université, Paris, France, 2023.
62. Basavaiah, R.; Gurudutt, P.S. Prebiotic Carbohydrates for Therapeutics. *Endocr. Metab. Immune Disord.-Drug Targets (Former. Curr. Drug Targets-Immune Endocr. Metab. Disord.)* **2021**, *21*, 230–245. [CrossRef] [PubMed]
63. Cardoso, B.B.; Amorim, C.; Silvério, S.C.; Rodrigues, L.R. Novel and emerging prebiotics: Advances and opportunities. In *Advances in Food and Nutrition Research*; Academic Press: Cambridge, MA, USA, 2021; Volume 95, pp. 41–95.
64. Salinas, E.; Reyes-Pavón, D.; Cortes-Perez, N.G.; Torres-Maravilla, E.; Bitzer-Quintero, O.K.; Langella, P.; Bermúdez-Humarán, L.G. Bioactive compounds in food as a current therapeutic approach to maintain a healthy intestinal epithelium. *Microorganisms* **2021**, *9*, 1634. [CrossRef]
65. Lordan, C.; Thapa, D.; Ross, R.P.; Cotter, P.D. Potential for enriching next-generation health-promoting gut bacteria through prebiotics and other dietary components. *Gut Microbes* **2020**, *11*, 1–20. [CrossRef]

66. Peredo-Lovillo, A.; Romero-Luna, H.E.; Jiménez-Fernández, M. Health promoting microbial metabolites produced by gut microbiota after prebiotics metabolism. *Food Res. Int.* **2020**, *136*, 109473. [CrossRef]
67. Zhang, Z.; Lin, T.; Meng, Y.; Hu, M.; Shu, L.; Jiang, H.; Zhou, X. FOS/GOS attenuates high-fat diet induced bone loss via reversing microbiota dysbiosis, high intestinal permeability and systemic inflammation in mice. *Metabolism* **2021**, *119*, 154767. [CrossRef]
68. Barbalho, R.L.D.C.; Castaneda, C.; Araújo, L.F.; Kiess, A.S.; Carvalho, R.S.; Barbalho, C.B.; Bonato, M.A. B-glucans and MOS, essential oil, and probiotics in diets of broilers challenged with *Eimeria* spp. and *Clostridium perfringens*. *Poult. Sci.* **2023**, *102*, 102541. [CrossRef]
69. Hazrati, S.; Rezaeipour, V.; Asadzadeh, S. Effects of phytogenic feed additives, probiotic and mannan-oligosaccharides on performance, blood metabolites, meat quality, intestinal morphology, and microbial population of Japanese quail. *Br. Poult. Sci.* **2020**, *61*, 132–139. [CrossRef] [PubMed]
70. El Hage, R. Ability of a Propionate-Producing Synthetic Microbial Consortium to Restore Functionality in a Dysbiosed Human gut Microbiome. Ph.D. Dissertation, Ghent University, Ghent, Belgium, 2019.
71. Ojwach, J.; Adetunji, A.I.; Mutanda, T.; Mukaratirwa, S. Oligosaccharides production from coprophilous fungi: An emerging functional food with potential health-promoting properties. *Biotechnol. Rep.* **2022**, *33*, e00702. [CrossRef] [PubMed]
72. Zeng, M.; Mohapatra, S.; Oh, J.H.; Astmann, T.; van Pijkeren, J.P.; Pan, X. Novel galacto-oligosaccharides from lactose: Chemical synthesis, structural characterization, and in vitro assessment of prebiotic activity. *ACS Sustain. Chem. Eng.* **2023**, *11*, 14031–14045. [CrossRef]
73. Vera, C.; Córdova, A.; Aburto, C.; Guerrero, C.; Suárez, S.; Illanes, A. Synthesis and purification of galacto-oligosaccharides: State of the art. *World J. Microbiol. Biotechnol.* **2016**, *32*, 197. [CrossRef]
74. Rico-Rodríguez, F.; Villamiel, M.; Ruiz-Aceituno, L.; Serrato, J.C.; Montilla, A. Effect of the lactose source on the ultrasound-assisted enzymatic production of galactooligosaccharides and gluconic acid. *Ultrason. Sonochem.* **2020**, *67*, 104945. [CrossRef] [PubMed]
75. Perotti, M.C.; Vénica, C.I.; Wolf, I.V.; Vélez, M.A.; Peralta, G.H.; Quiberoni, A.; Bergamini, C.V. Biomolecules Derived from Whey: Strategies for Production and Biological Properties. In *Biomolecules from Natural Sources: Advances and Applications*; John Wiley & Sons Ltd.: Hoboken, NJ, USA, 2022; pp. 390–432.
76. Ureta, M.M.; Martins, G.N.; Figueira, O.; Pires, P.F.; Castilho, P.C.; Gomez-Zavaglia, A. Recent advances in β-galactosidase and fructosyltransferase immobilization technology. *Crit. Rev. Food Sci. Nutr.* **2021**, *61*, 2659–2690. [CrossRef] [PubMed]
77. Todea, A.; Boeriu, C.G.; Peter, F.; Biró, E. Immobilized β-d-galactosidases for improved synthesis of short-chain galacto-oligosaccharides. In *Biotechnological Progress and Beverage Consumption*; Academic Press: Cambridge, MA, USA, 2020; pp. 71–110.
78. Yang, X.D.; Wang, L.K.; Wu, H.Y.; Jiao, L. Effects of prebiotic galacto-oligosaccharide on postoperative cognitive dysfunction and neuroinflammation through targeting of the gut-brain axis. *BMC Anesthesiol.* **2018**, *18*, 177. [CrossRef]
79. Hackenhaar, C.R.; Spolidoro, L.S.; Flores, E.E.E.; Klein, M.P.; Hertz, P.F. Batch synthesis of galactooligosaccharides from co-products of milk processing using immobilized β-galactosidase from *Bacillus circulans*. *Biocatal. Agric. Biotechnol.* **2021**, *36*, 102136. [CrossRef]
80. Kittibunchakul, S.; Pham, M.L.; Tran, A.M.; Nguyen, T.H. β-Galactosidase from *Lactobacillus helveticus* DSM 20075: Biochemical characterization and recombinant expression for applications in dairy industry. *Int. J. Mol. Sci.* **2019**, *20*, 947. [CrossRef] [PubMed]
81. Arsov, A.; Ivanov, I.; Tsigoriyna, L.; Petrov, K.; Petrova, P. In Vitro Production of Galactooligosaccharides by a Novel β-Galactosidase of *Lactobacillus bulgaricus*. *Int. J. Mol. Sci.* **2022**, *23*, 14308. [CrossRef]
82. Delgado-Fernandez, P.; Plaza-Vinuesa, L.; Hernandez-Hernandez, O.; de Las Rivas, B.; Corzo, N.; Muñoz, R.; Moreno, F.J. Unravelling the carbohydrate specificity of MelA from *Lactobacillus plantarum* WCFS1: An α-galactosidase displaying regioselective transgalactosylation. *Int. J. Biol. Macromol.* **2020**, *153*, 1070–1079. [CrossRef] [PubMed]
83. Bellikci-Koyu, E.; Sarer-Yurekli, B.P.; Akyon, Y.; Aydin-Kose, F.; Karagozlu, C.; Ozgen, A.G.; Buyuktuncer, Z. Effects of regular kefir consumption on gut microbiota in patients with metabolic syndrome: A parallel-group, randomized, controlled study. *Nutrients* **2019**, *11*, 2089. [CrossRef]
84. Fai, A.E.C.; da Silva, J.B.; de Andrade, C.J.; Bution, M.L.; Pastore, G.M. Production of prebiotic galactooligosaccharides from lactose by *Pseudozyma tsukubaensis* and *Pichia kluyveri*. *Biocatal. Agric. Biotechnol.* **2014**, *3*, 343–350. [CrossRef]
85. Usvalampi, A.; Maaheimo, H.; Tossavainen, O.; Frey, A.D. Enzymatic synthesis of fucose-containing galacto-oligosaccharides using β-galactosidase and identification of novel disaccharide structures. *Glycoconj. J.* **2018**, *35*, 31–40. [CrossRef]
86. Eskandarloo, H.; Abbaspourrad, A. Production of galacto-oligosaccharides from whey permeate using β-galactosidase immobilized on functionalized glass beads. *Food Chem.* **2018**, *251*, 115–124. [CrossRef]
87. Carević, M.; Vukašinović-Sekulić, M.; Ćorović, M.; Rogniaux, H.; Ropartz, D.; Veličković, D.; Bezbradica, D. Evaluation of β-galactosidase from *Lactobacillus acidophilus* as biocatalyst for galacto-oligosaccharides synthesis: Product structural characterization and enzyme immobilization. *J. Biosci. Bioeng.* **2018**, *126*, 697–704. [CrossRef]
88. Yang, J.; Gao, R.; Zhou, Y.; Anankanbil, S.; Li, J.; Xie, G.; Guo, Z. β-Glucosidase from *Thermotoga naphthophila* RKU-10 for exclusive synthesis of galactotrisaccharides: Kinetics and thermodynamics insight into reaction mechanism. *Food Chem.* **2018**, *240*, 422–429. [CrossRef]
89. Botelho-Cunha, V.A.; Mateus, M.; Petrus, J.C.C.; de Pinho, M.N. Tailoring the enzymatic synthesis and nanofiltration fractionation of galacto-oligosaccharides. *Biochem. Eng. J.* **2010**, *50*, 29–36. [CrossRef]

90. Vera, C.; Guerrero, C.; Conejeros, R.; Illanes, A. Synthesis of galactooligosaccharides by β-galactosidase from *Aspergillus oryzae* using partially dissolved and supersaturated solution of lactose. *Enzym. Microb. Technol.* **2012**, *50*, 188–194. [CrossRef]
91. Jovanovic-Malinovska, R.; Fernandes, P.; Winkelhausen, E.; Fonseca, L. Galacto-oligosaccharides synthesis from lactose and whey by β-galactosidase immobilized in PVA. *Appl. Biochem. Biotechnol.* **2012**, *168*, 1197–1211. [CrossRef]
92. Oh, S.Y.; Park, M.S.; Lee, Y.G.; Thi, N.N.; Baek, N.I.; Ji, G.E. Enzymatic synthesis of β-galactosyl fucose using recombinant bifidobacterial β-galactosidase and its prebiotic effect. *Glycoconj. J.* **2019**, *36*, 199–209. [CrossRef]
93. Kumar, N.V.; Sawargaonkar, G.L.; Rani, C.S.; Singh, A.; Prakash, T.R.; Triveni, S.; Venkatesh, B. Comparative Analysis of Pigeonpea Stalk Biochar Characteristics and Energy Use under Different Biochar Production Methods. *Sustainability* **2023**, *15*, 14394. [CrossRef]
94. Dahiya, S.; Rapoport, A.; Singh, B. Biotechnological Potential of Lignocellulosic Biomass as Substrates for Fungal Xylanases and Its Bioconversion into Useful Products: A Review. *Fermentation* **2024**, *10*, 82. [CrossRef]
95. Peng, P.; Peng, F.; Bian, J.; Xu, F.; Sun, R. Studies on the starch and hemicelluloses fractionated by graded ethanol precipitation from bamboo *Phyllostachys bambusoides* f. shouzhu Yi. *J. Agric. Food Chem.* **2011**, *59*, 2680–2688. [CrossRef]
96. Samanta, A.K.; Kolte, A.P.; Elangovan, A.V.; Dhali, A.; Senani, S.; Sridhar, M.; Roy, S. Value addition of corn husks through enzymatic production of xylooligosaccharides. *Braz. Arch. Biol. Technol.* **2016**, *59*, e16160078. [CrossRef]
97. Jnawali, P.; Kumar, V.; Tanwar, B.; Hirdyani, H.; Gupta, P. Enzymatic production of xylooligosaccharides from brown coconut husk treated with sodium hydroxide. *Waste Biomass Valorization* **2018**, *9*, 1757–1766. [CrossRef]
98. Miao, Y.; Li, P.; Li, G.; Liu, D.; Druzhinina, I.S.; Kubicek, C.P.; Zhang, R. Two degradation strategies for overcoming the recalcitrance of natural lignocellulosic xylan by polysaccharides-binding GH 10 and GH 11 xylanases of filamentous fungi. *Environ. Microbiol.* **2017**, *19*, 1054–1064. [CrossRef]
99. Nasirpour, N.; Mousavi, S.M. RSM based optimization of PEG assisted ionic liquid pretreatment of sugarcane bagasse for enhanced bioethanol production: Effect of process parameters. *Biomass Bioenergy* **2018**, *116*, 89–98. [CrossRef]
100. Pu, J.; Zhao, X.; Wang, Q.; Wang, Y.; Zhou, H. Development and validation of a HPLC method for determination of degree of polymerization of xylo-oligosaccharides. *Food Chem.* **2016**, *213*, 654–659. [CrossRef]
101. Chapla, D.; Pandit, P.; Shah, A. Production of xylooligosaccharides from corncob xylan by fungal xylanase and their utilization by probiotics. *Bioresour. Technol.* **2012**, *115*, 215–221. [CrossRef]
102. Wang, C.; Lu, X.; Gao, J.; Li, X.; Zhao, J. Xylo-oligosaccharides inhibit enzymatic hydrolysis by influencing enzymatic activity of cellulase from *Penicillium oxalicum*. *Energy Fuels* **2018**, *32*, 9427–9437. [CrossRef]
103. Dhiman, S.; Mukherjee, G. Recent advances and industrial applications of microbial xylanases: A review. In *Fungi and Their Role in Sustainable Development: Current Perspectives*; Springer: Singapore, 2018; pp. 329–348.
104. da Silva Menezes, B.; Rossi, D.M.; Squina, F.; Ayub, M.A.Z. Comparative production of xylanase and the liberation of xylooligosaccharides from lignocellulosic biomass by *Aspergillus brasiliensis* BLf1 and recombinant *Aspergillus nidulans* XynC A773. *Int. J. Food Sci. Technol.* **2018**, *53*, 2110–2118. [CrossRef]
105. Adsul, M.G.; Bastawde, K.B.; Gokhale, D.V. Biochemical characterization of two xylanases from yeast *Pseudozyma hubeiensis* producing only xylooligosaccharides. *Bioresour. Technol.* **2009**, *100*, 6488–6495. [CrossRef]
106. Yue, P.; Hu, Y.; Tian, R.; Bian, J.; Peng, F. Hydrothermal pretreatment for the production of oligosaccharides: A review. *Bioresour. Technol.* **2022**, *343*, 126075. [CrossRef]
107. Alawiye, T.T.; Babalola, O.O. Metagenomic insight into the community structure and functional genes in the sunflower rhizosphere microbiome. *Agriculture* **2021**, *11*, 167. [CrossRef]
108. Reddy, S.S.; Krishnan, C. Production of xylooligosaccharides in SSF by *Bacillus subtilis* KCX006 producing β-xylosidase-free endo-xylanase and multiple xylan debranching enzymes. *Prep. Biochem. Biotechnol.* **2016**, *46*, 49–55. [CrossRef]
109. Boonchuay, P.; Takenaka, S.; Kuntiya, A.; Techapun, C.; Leksawasdi, N.; Seesuriyachan, P.; Chaiyaso, T. Purification, characterization, and molecular cloning of the xylanase from *Streptomyces thermovulgaris* TISTR1948 and its application to xylooligosaccharide production. *J. Mol. Catal. B Enzym.* **2016**, *129*, 61–68. [CrossRef]
110. Sun, M.-Z.; Zheng, H.-C.; Meng, L.-C.; Sun, J.-S.; Song, H.; Bao, Y.-J.; Pei, H.-S.; Yan, Z.; Zhang, X.-Q.; Zhang, J.-S.; et al. Direct cloning, expression of a thermostable xylanase gene from the metagenomic DNA of cow dung compost and enzymatic production of xylooligosaccharides from corncob. *Biotechnol. Lett.* **2015**, *37*, 1877–1886. [CrossRef]
111. Boonchuay, P.; Techapun, C.; Seesuriyachan, P.; Chaiyaso, T. Production of xylooligosaccharides from corncob using a crude thermostable endo-xylanase from *Streptomyces thermovulgaris* TISTR1948 and prebiotic properties. *Food Sci. Biotechnol.* **2014**, *23*, 1515–1523. [CrossRef]
112. Bian, J.; Peng, F.; Peng, X.-P.; Peng, P.; Xu, F.; Sun, R.-C. Structural features and antioxidant activity of xylooligosaccharides enzymatically produced from sugarcane bagasse. *Bioresour. Technol.* **2013**, *127*, 236–241. [CrossRef]
113. Wang, T.H.; Lu, S. Production of xylooligosaccharide from wheat bran by microwave assisted enzymatic hydrolysis. *Food Chem.* **2013**, *138*, 1531–1535. [CrossRef]
114. Samanta, A.K.; Jayapal, N.; Kolte, A.P.; Senani, S.; Sridhar, M.; Mishra, S.; Prasad, C.S.; Suresh, K.P. Application of pigeon pea (*Cajanus cajan*) stalks as raw material for xylooligosaccharides production. *Appl. Biochem. Biotechnol.* **2013**, *169*, 2392–2404. [CrossRef]
115. Jayapal, N.; Samanta, A.K.; Kolte, A.P.; Senani, S.; Sridhar, M.; Suresh, K.P.; Sampath, K.T. Value addition to sugarcane bagasse: Xylan extraction and its process optimization for xylooligosaccharides production. *Ind. Crops Prod.* **2013**, *42*, 14–24. [CrossRef]

116. Samanta, A.K.; Jayapal, N.; Kolte, A.P.; Senani, S.; Sridhar, M.; Dhali, A.; Suresh, K.P.; Jayaram, C.; Prasad, C.S. Process for enzymatic production of xylooligosaccharides from the xylan of corn cobs. *J. Food Process. Preserv.* **2015**, *39*, 729–736. [CrossRef]
117. Khat-Udomkiri, N.; Sivamaruthi, B.S.; Sirilun, S.; Lailerd, N.; Peerajan, S.; Chaiyasut, C. Optimization of alkaline pretreatment and enzymatic hydrolysis for the extraction of xylooligosaccharide from rice husk. *AMB Express* **2018**, *8*, 115. [CrossRef]
118. Singh, R.S.; Singh, R.P.; Kennedy, J.F. Recent insights in enzymatic synthesis of fructooligosaccharides from inulin. *Int. J. Biol. Macromol.* **2016**, *85*, 565–572. [CrossRef] [PubMed]
119. Narisetty, V.; Parhi, P.; Mohan, B.; Hazeena, S.H.; Kumar, A.N.; Gullón, B.; Binod, P. Valorization of renewable resources to functional oligosaccharides: Recent trends and future prospective. *Bioresour. Technol.* **2022**, *346*, 126590. [CrossRef] [PubMed]
120. Singh, R.S.; Singh, T.; Kennedy, J.F. Enzymatic synthesis of fructooligosaccharides from inulin in a batch system. *Carbohydr. Polym. Technol. Appl.* **2020**, *1*, 100009. [CrossRef]
121. Mohan, C.O.; Carvajal-Millan, E.; Ravishankar, C.N. Current Trends in the Biotechnological Production of Fructooligosaccharides. In *Research Methodology in Food Sciences*; Apple Academic Press: Palm Bay, FL, USA, 2018; pp. 199–220.
122. Smaali, I.; Jazzar, S.; Soussi, A.; Muzard, M.; Aubry, N.; Marzouki, M.N. Enzymatic synthesis of fructooligosaccharides from date by-products using an immobilized crude enzyme preparation of β-D-fructofuranosidase from *Aspergillus awamori* NBRC 4033. *Biotechnol. Bioprocess Eng.* **2012**, *17*, 385–392. [CrossRef]
123. Bahlawan, R.; Karboune, S.; Liu, L.; Sahyoun, A.M. Investigation of Biocatalytic Production of Lactosucrose and Fructooligosaccharides Using Levansucrases and Dairy By-products as Starting Materials. *Enzym. Microb. Technol.* **2023**, *169*, 110279. [CrossRef]
124. Díez-Municio, M.; de las Rivas, B.; Jimeno, M.L.; Muñoz, R.; Moreno, F.J.; Herrero, M. Enzymatic synthesis and characterization of fructooligosaccharides and novel maltosylfructosides by inulosucrase from *Lactobacillus gasseri* DSM 20604. *Appl. Environ. Microbiol.* **2013**, *79*, 4129–4140. [CrossRef] [PubMed]
125. Tian, F.; Karboune, S.; Hill, A. Synthesis of fructooligosaccharides and oligolevans by the combined use of levansucrase and endo-inulinase in one-step bi-enzymatic system. *Innov. Food Sci. Emerg. Technol.* **2014**, *22*, 230–238. [CrossRef]
126. Soliman, A.B.; Hassan, M.H.; Huan, T.N.; Abugable, A.A.; Elmehalmey, W.A.; Karakalos, S.G.; Alkordi, M.H. Pt Immobilization within a Tailored Porous-Organic Polymer–Graphene Composite: Opportunities in the Hydrogen Evolving Reaction. *ACS Catal.* **2017**, *7*, 7847–7854. [CrossRef]
127. de Siqueira, E.C.; Öner, E.T. Co-production of levan with other high-value bioproducts: A review. *Int. J. Biol. Macromol.* **2023**, *235*, 123800. [CrossRef]
128. Huang, M.P.; Wu, M.; Xu, Q.S.; Mo, D.J.; Feng, J.X. Highly efficient synthesis of fructooligosaccharides by extracellular fructooligosaccharide-producing enzymes and immobilized cells of *Aspergillus aculeatus* M105 and purification and biochemical characterization of a fructosyltransferase from the fungus. *J. Agric. Food Chem.* **2016**, *64*, 6425–6432. [CrossRef] [PubMed]
129. Peña-Cardeña, A.; Rodríguez-Alegría, M.E.; Olvera, C.; Munguía, A.L. Synthesis of Fructooligosaccharides by IslA4, a truncated inulosucrase from *Leuconostoc citreum*. *BMC Biotechnol.* **2015**, *15*, 2. [CrossRef]
130. Kralj, S.; Leeflang, C.; Sierra, E.I.; Kempiński, B.; Alkan, V.; Kolkman, M. Synthesis of fructooligosaccharides (FosA) and inulin (InuO) by GH68 fructosyltransferases from *Bacillus agaradhaerens* strain WDG185. *Carbohydr. Polym.* **2018**, *179*, 350–359. [CrossRef] [PubMed]
131. Chen, W.C. Medium improvement for β-fructofuranosidase production by *Aspergillus japonicus*. *Process Biochem.* **1998**, *33*, 267–271. [CrossRef]
132. Hirayama, M.; Sumi, N.; Hidaka, H. Purification and properties of a fructooligosaccharide-producing β-fructofuranosidase from *Aspergillus niger* ATCC 20611. *Agric. Biol. Chem.* **1989**, *53*, 667–673. [CrossRef]
133. Patil, M.; Ashwin, B. Fructosyltransferase production by indigenously isolated *Syncephalastrum racemosum* Cohn. *J. Glob. Biosci.* **2014**, *3*, 597–603.
134. Bhatia, L.; Sharma, A.; Bachheti, R.K.; Chandel, A.K. Lignocellulose derived functional oligosaccharides: Production, properties, and health benefits. *Prep. Biochem. Biotechnol.* **2019**, *49*, 744–758. [CrossRef]
135. Usami, S.; Ishii, T.; Kirimura, K.; Uehara, K.I.; Chen, J. Production of β-fructofuranosidase showing fructose-transferring activity by *Penicillium frequentans* (*P. glabrum*). *J. Ferment. Bioeng.* **1991**, *72*, 303–305. [CrossRef]
136. Barthomeuf, C.; Pourrat, H. Production of high-content fructo-oligosaccharides by an enzymatic system from *Penicillium rugulosum*. *Biotechnol. Lett.* **1995**, *17*, 911–916. [CrossRef]
137. Yun, J.W.; Kim, D.H.; Moon, H.Y.; Song, C.H.; Song, S.K. Simultaneous formation of fructosyltransferase and glucosyltransferase in *Aureobasidium pullulans*. *J. Microbiol. Biotechnol.* **1997**, *7*, 204–208.
138. Hayashi, S.; Ito, K.; Nonoguchi, M.; Takasaki, Y.; Imada, K. Immobilization of a fructosyl-transferring enzyme from *Aureobasidium* sp. on shirasu porous glass. *J. Ferment. Bioeng.* **1991**, *72*, 68–70. [CrossRef]
139. Fujita, K.; Hara, K.; Hashimoto, H.; Kitahata, S. Transfructosylation catalyzed by β-fructofuranosidase I from *Arthrobacter* sp. K-1. *Agric. Biol. Chem.* **1990**, *54*, 2655–2661. [CrossRef]
140. de Menezes, C.R.; Silva, Í.S.; Pavarina, É.C.; Guímaro Dias, E.F.; Guímaro Dias, F.; Grossman, M.J.; Durrant, L.R. Production of xylooligosaccharides from enzymatic hydrolysis of xylan by the white-rot fungi Pleurotus. *Int. Biodeterior. Biodegrad.* **2009**, *63*, 673–678. [CrossRef]
141. Bersaneti, G.T.; Pan, N.C.; Baldo, C.; Celligoi, M.A.P.C. Co-production of Fructooligosaccharides and Levan by Levansucrase from *Bacillus subtilis* natto with Potential Application in the Food Industry. *Appl. Biochem. Biotechnol.* **2018**, *184*, 838–851. [CrossRef] [PubMed]

142. Silva, M.F.; Rigo, D.; Mossi, V.; Golunski, S.; de Oliveira Kuhn, G.; Di Luccio, M.; Dallago, R.; de Oliveira, D.; Oliveira, J.V.; Treichel, H. Enzymatic synthesis of fructooligosaccharides by inulinases from *Aspergillus niger* and *Kluyveromyces marxianus* NRRL Y-7571 in aqueous–organic medium. *Food Chem.* **2013**, *138*, 148–153. [CrossRef] [PubMed]
143. Nascimento, A.K.C.; Nobre, C.; Cavalcanti, M.T.H.; Teixeira, J.A.; Porto, A.L.F. Screening of fungi from the genus *Penicillium* for production of β-fructofuranosidase and enzymatic synthesis of fructooligosaccharides. *J. Mol. Catal. B Enzym.* **2016**, *134*, 70–78. [CrossRef]
144. Karboune, S.; Appanah, N.; Khodaei, N.; Tian, F. Enzymatic synthesis of fructooligosaccharides from sucrose by endo-inulinase-catalyzed transfructosylation reaction in biphasic systems. *Process Biochem.* **2018**, *69*, 82–91. [CrossRef]
145. Kuhn, G.d.O.; Silva, M.F.; Mulinari, J.; Golunski, S.; Dallago, R.M.; Dalla Rosa, C.; Val´erio, A.; Oliveira, D.d.; Oliveira, J.V.; Mossi, A.J.; et al. *Aspergillus niger* inulinase immobilized in polyurethane foam and treated in pressurized LPG: A potential catalyst for enzymatic synthesis of fructooligosaccharides. *Biocatal. Biotransform.* **2016**, *34*, 291–294. [CrossRef]
146. Aung, T.; Jiang, H.; Liu, G.L.; Chi, Z.; Hu, Z.; Chi, Z.M. Overproduction of a β-fructofuranosidase1 with a high FOS synthesis activity for efficient biosynthesis of fructooligosaccharides. *Int. J. Biol. Macromol.* **2019**, *130*, 988–996. [CrossRef]
147. Sathitkowitchai, W.; Sathapondecha, P.; Angthong, P.; Srimarut, Y.; Malila, Y.; Nakkongkam, W.; Rungrassamee, W. Isolation and Characterization of Mannanase-Producing Bacteria for Potential Synbiotic Application in Shrimp Farming. *Animals* **2022**, *12*, 2583. [CrossRef]
148. Ghosh, K.; Ray, M.; Adak, A.; Halder, S.K.; Das, A.; Jana, A.; Mondal, K.C. Role of probiotic *Lactobacillus fermentum* KKL1 in the preparation of a rice based fermented beverage. *Bioresour. Technol.* **2015**, *188*, 161–168. [CrossRef] [PubMed]
149. Jana, U.K.; Suryawanshi, R.K.; Prajapati, B.P.; Soni, H.; Kango, N. Production optimization and characterization of mannooligosaccharide generating β-mannanase from Aspergillus oryzae. *Bioresour. Technol.* **2018**, *268*, 308–314. [CrossRef] [PubMed]
150. Cescutti, P.; Campa, C.; Delben, F.; Rizzo, R. Structure of the oligomers obtained by enzymatic hydrolysis of the glucomannan produced by the plant Amorphophallus konjac. *Carbohydr. Res.* **2002**, *337*, 2505–2511. [CrossRef] [PubMed]
151. Rantanen, H.; Virkki, L.; Tuomainen, P.; Kabel, M.; Schols, H.; Tenkanen, M. Preparation of arabinoxylobiose from rye xylan using family 10 *Aspergillus aculeatus endo*-1, 4-β-D-xylanase. *Carbohydr. Polym.* **2007**, *68*, 350–359. [CrossRef]
152. Cao, Y.; Zhang, Z.; Ling, N.; Yuan, Y.; Zheng, X.; Shen, B.; Shen, Q. *Bacillus subtilis* SQR 9 can control Fusarium wilt in cucumber by colonizing plant roots. *Biol. Fertil. Soils* **2011**, *47*, 495–506. [CrossRef]
153. Zhang, M.; Chen, X.L.; Zhang, Z.H.; Sun, C.Y.; Chen, L.L.; He, H.L.; Zhou, B.C.; Zhang, Y.Z. Purification and functional characterization of endo-β-mannanase MAN5 and its application in oligosaccharide production from konjac flour. *Appl. Microbiol. Biotechnol.* **2009**, *83*, 865–873. [CrossRef] [PubMed]
154. Blibech, M.; Chaari, F.; Bhiri, F.; Dammak, I.; Ghorbel, R.E.; Chaabouni, S.E. Production of manno-oligosaccharides from locust bean gum using immobilized *Penicillium occitanis* mannanase. *J. Mol. Catal. B Enzym.* **2011**, *73*, 111–115. [CrossRef]
155. Oda, Y.; Tonomura, K. Characterization of β-mannanase and β-mannosidase secreted from the yeast Trichosporon cutaneum JCM 2947. *Lett. Appl. Microbiol.* **1996**, *22*, 173–178. [CrossRef]
156. Ghosh, A.; Verma, A.K.; Tingirikari, J.R.; Shukla, R.; Goyal, A. Recovery and purification of oligosaccharides from copra meal by recombinant endo-β-mannanase and deciphering molecular mechanism involved and its role as potent therapeutic agent. *Mol. Biotechnol.* **2015**, *57*, 111–127. [CrossRef]
157. Jian, H.-L.; Zhu, L.-W.; Zhang, W.-M.; Sun, D.-F.; Jiang, J.-X. Enzymatic production and characterization of manno-oligosaccharides from *Gleditsia sinensis* galactomannan gum. *Int. J. Biol. Macromol.* **2013**, *55*, 282–288. [CrossRef]
158. Albrecht, S.; van Muiswinkel, G.C.J.; Xu, J.; Schols, H.A.; Voragen, A.G.J.; Gruppen, H. Enzymatic production and characterization of konjac glucomannan oligosaccharides. *J. Agric. Food Chem.* **2011**, *59*, 12658–12666. [CrossRef]
159. Yang, J.K.; Chen, Q.C.; Zhou, B.; Wang, X.J.; Liu, S.Q. Manno-oligosaccharide preparation by the hydrolysis of konjac flour with a thermostable endo-mannanase from *Talaromyces cellulolyticus*. *J. Appl. Microbiol.* **2019**, *127*, 520–532. [CrossRef] [PubMed]
160. Nopvichai, C.; Charoenwongpaiboon, T.; Luengluepunya, N.; Ito, K.; Muanprasat, C.; Pichyangkura, R. Production and purification of mannan oligosaccharide with epithelial tight junction enhancing activity. *Peer J.* **2019**, *7*, e7206. [CrossRef] [PubMed]
161. Rabelo, M.C.; Honorato, T.L.; Gonçalves, L.R.B.; Pinto, G.A.S.; Rodrigues, S. Optimization of enzymatic synthesis of isomalto-oligosaccharides production. *J. Food Biochem.* **2009**, *33*, 342–354. [CrossRef]
162. Sorndech, W.; Nakorn, K.N.; Tongta, S.; Blennow, A. Isomalto-oligosaccharides: Recent insights in production technology and their use for food and medical applications. *LWT* **2018**, *95*, 135–142. [CrossRef]
163. Saman, P.; Chaiongkarn, A.; Moonmangmee, S.; Artjariyasripong, S. Prebiotic isomalto-oligosaccharide production from economic crops of Thailand. *Asia-Pac. J. Sci. Technol.* **2012**, *17*, 794–799.
164. Basu, A.; Mutturi, S.; Prapulla, S.G. Production of isomaltooligosaccharides (IMO) using simultaneous saccharification and transglucosylation from starch and sustainable sources. *Process Biochem.* **2016**, *51*, 1464–1471. [CrossRef]
165. Basu, A.; Mutturi, S.; Prapulla, S.G. Modeling of enzymatic production of isomaltooligosaccharides: A mechanistic approach. *Catal. Sci. Technol.* **2015**, *5*, 2945–2958. [CrossRef]
166. Shinde, V.K.; Vamkudoth, K.R. Maltooligosaccharide forming amylases and their applications in food and pharma industry. *J. Food Sci. Technol.* **2022**, *59*, 3733–3744. [CrossRef]
167. Plongbunjong, V.; Graidist, P.; Knudsen, K.E.; Wichienchot, S. Isomaltooligosaccharide synthesised from rice starch and its prebiotic properties in vitro. *Int. J. Food Sci. Technol.* **2017**, *52*, 2589–2595. [CrossRef]

168. Cui, J.; Li, Y.; Wang, Q.; Li, J.; Ou, Y.; Wang, J.; Wang, W. Production, purification and analysis of the isomalto-oligosaccharides from Chinese chestnut (*Castanea mollissima* Blume) and the prebiotics effects of them on proliferation of *Lactobacillus*. *Food Bioprod. Process.* **2017**, *106*, 75–81. [CrossRef]
169. Prapulla, S.G.; Subhaprada, V.; Karanth, N.G. Microbial production of oligosaccharides: A review. *Adv. Appl. Microbiol.* **2000**, *47*, 299–343. [PubMed]
170. Casa-Villegas, M.; Marín-Navarro, J.; Polaina, J. Synergies in coupled hydrolysis and fermentation of cellulose using a Trichoderma reesei enzyme preparation and a recombinant *Saccharomyces cerevisiae* strain. *World J. Microbiol. Biotechnol.* **2017**, *33*, 140. [CrossRef] [PubMed]
171. Saman, P.; Kuancha, C.; Chaiongkarn, A.; Moonmangmee, S.; Fungsin, B. Isomalto-oligosaccharides production from rice flour and cassava starch. *J. Food Sci. Agric. Technol. (JFAT)* **2019**, *5*, 188–192.
172. Kaulpiboon, J.; Rudeekulthamrong, P.; Watanasatitarpa, S.; Ito, K.; Pongsawasdi, P. Synthesis of long-chain isomaltooligosaccharides from tapioca starch and an in vitro investigation of their prebiotic properties. *J. Mol. Catal. B Enzym.* **2015**, *120*, 127–135. [CrossRef]
173. Sorndech, W. Isomaltooligosaccharides as prebiotics and their health benefits. In *Probiotics, Prebiotics and Synbiotics: Technological Advancements Towards Safety and Industrial Applications*; John Wiley & Sons Ltd.: Hoboken, NJ, USA, 2022; pp. 361–377.
174. Chockchaisawasdee, S.; Poosaran, N. Production of isomaltooligosaccharides from banana flour. *J. Sci. Food Agric.* **2013**, *93*, 180–186. [CrossRef]