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# Functional Oligosaccharides Derived from Fruit-and-Vegetable By-Products and Wastes

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**Abstract:** Industrial-scale food manufacturing generates high quantity of fruit-and-vegetable solid by-product and waste streams (FVSW) which have become a challenge to the environment post-production. Due to this, proposals for a better use of resources to reduce the environmental burden and to promote a circular economy have been introduced. Reintroducing discarded materials back into the production through the recovery of valuable components or through the conversion into value-added ingredients is one approach attracting strong interest in research. FVSW is rich in lignocellulosic materials which can be reused to produce bioactive ingredients. This review highlights the potential use of FVSW as low-cost raw materials and describes the valorisation of FVSW for the production of functional oligosaccharides. The focus is on the production technologies of the main functional oligosaccharides, namely pectic-oligosaccharides, inulin and fructooligosaccharides, xylooligosaccharides, and isomaltooligosaccharides.

**Keywords:** fruit-and-vegetable wastes; fruit-and-vegetable by-products; oligosaccharides; prebiotics; lignocellulosic biomass

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## 1. Introduction

The estimated global production of fruits and vegetables in 2020 was 2035 Mt [1]. When those horticultural crops are used in the supply chains of agriculture and food industries, large quantities of organic residues are derived as organic by-products or waste [2]. It was estimated that approximately 12% of food production is lost in manufacturing stage, 90% of which is by-products and production-line waste [3]. Waste derived from the production of fruits and vegetables (drinks and other preservation processes) accounts for 40% of the total waste from the food industry [4]. Table 1 shows the approximate percentages and nature of solid waste streams from certain fruits and vegetables processing.

The chemical composition of these horticultural residues depends on many factors, such as plant sources, varieties, production methods, process parameters. These residues generally are highly perishable as they are high in organic load, water content, and microbial activities. Disposal of large quantities of these residues has become a big challenge for waste management as doing so improperly or through traditional methods, i.e., landfill or burning, causes a significant negative impact on the environment [2]. Alternatively, from the bespoke disposal approaches, they can be converted into fertilisers or animal feeds as these organic residues are rich in nutrients, such as proteins, carbohydrates, flavour compounds, and phytochemicals.

**Table 1.** Approximate percentages and nature of solid waste generated from fruit and vegetable manufacturing lines.

| Sources of Solid Wastes | Approximate Percentage of Waste from Raw Material (*w*/*w*) | References |
|---|---|---|
| Banana peel | 30 | [5] |
| Apple (pomace, skin, seeds, stem) | 25–30 | [6,7] |
| Citrus (orange, lemon, grapefruit-pomace, peel, seeds) | 50 | [8,9] |
| Exotic fruits (pineapple, mango, mangosteen-skin, core, peel, stone) | 35–60 | [10–12] |
| Artichoke (bracts, stem, leaves) | 60 | [13,14] |
| Asparagus spear | 40–50 | [15] |
| Potato peel | 15–40 | [5] |
| Corn cob | 20–30 | [16] |

Nevertheless, considering the market value of these components, fertilisers and animal feeds are not necessarily making the most of these resources. Realising the values of the compounds that can be derived from horticultural by-products, shifting in residue handling, has arisen during the last few decades [17]. The focus of this approach is to reuse/recycle food industry residues by recovering valuable compounds utilising biochemical, chemical and/or thermal/physical processes; the overarching aim is to optimise the use of resources and reduce the amount of waste [18].

Among the high-value components that can be derived from food and agricultural by-products and waste stream, oligosaccharides have been extensively investigated [2,19,20]. Being rich in lignocellulosic materials, fruits-and-vegetable biomass (pomaces, skins, peels, pulps, seeds) serve as inexpensive sources for functional oligosaccharide production [2]. This review focuses on the production of main functional oligosaccharides derived specifically from fruit-and-vegetable solid by-product and waste streams (FVSW) from food manufacturing.

## 2. Lignocellulosic Residues from FVSW

Lignocellulose is produced by photosynthesis, where the constituent polysaccharides are bound together in a strong meshed network by both covalent cross-linked and non-covalent bonds [21]. Lignocellulose forms the structure of the cell wall of fruits and vegetables. Therefore, lignocellulosic residues become the most abundant waste from the manufacturing of plant food products. In general, lignocellulosic materials in plant cell wall composes of water-insoluble components (cellulose, hemicellulose, and lignin) and water-soluble components (e.g., pectin, gum, exudates).

In fruits and vegetables of importance in food manufacturing, the main chemical constituent of lignocellulose includes cellulose, hemicellulose, lignin, and pectin. The proportion of these constituents is influenced by many pre- and post-harvest factors. Cellulose is the main components of lignocellulosic materials, followed by hemicellulose and lignin [2]. Pectin is not always considered one of the main components of cellulosic residues and usually excluded in many reviews on lignocellulosic materials from forestry and agricultural residues [2,19,21,22]. This is because pectin is only present in considerable amounts in certain crops as illustrated in Table 2.

**Table 2.** Composition of some lignocellulosic materials discarded from processing lines.

| Source of Lignocellulosic Residues | Cellulose (%) | Hemi-Cellulose (%) | Lignin (%) | Pectin (%) | References |
|---|---|---|---|---|---|
| Pear pomace | 34 | 19–21 | 20–33 | 2–13 | [23,24] |
| Apple pomace | 7–44 | 4–24 | 5–24 | 4–14 | [23,24] |
| Banana peel | 6–10 | 2–26 | 6–18 | 9–22 | [25–27] |
| Orange bagasse and peel | 9–37 | 11–31 | 7–22 | 16–42 | [26,28–30] |
| Lemon peel and pulp | 23–36 | 8–11 | 8 | 13–23 | [30] |
| Carrot pomace | 30–52 | 7–12 | 18–32 | 2–4 | [23,24] |
| Tomato pomace | 6–40 | 8–12 | 5–36 | 6–8 | [23,24] |
| Potato peel | 55 | 12 | 14 | 19–21 | [31] |
| Pomegranate peel | 26 | 11 | 6 | 28 | [31] |
| Sugar beet pulp | 22–30 | 24–32 | 3–4 | 24–32 | [32] |

The chemical structure of cellulose is similar to that of amylose with a difference in their anomeric configuration. Cellulose is a linear chain of D-glucose monomers linked by β-(1→4) glycosidic bonds with degree of polymerisation (DP) ranging from 250 to 10,000 [33]. Glucose chains form long, flat ribbons of cellulose microfibrils which are orderly stacked via hydrogen bonds, excluding water between the chains, thus forming compact crystalline regions [34]. Where the cellulose chain is irregularly arranged, an amorphous region is also present [21].

Unlike cellulose, hemicelluloses are complex branched heteropolysaccharides with short lateral chains linked by β-(1→4)-glycosidic bonds, and occasionally by β-(1→3)-glycosidic bonds [21]. The backbone of hemicellulose is either a homopolymer or a heteropolymer with a DP approximately 200 [35]. Subunits of hemicelluloses include of pentoses, hexoses and sugar acids. Hemicelluloses strengthen and stabilise the cellulose network by cross-linking cellulose microfibrils both in the interfibrillar and on the surface domains [36]. The hemicelluloses comprise xyloglucan, glucuronoxylan, arabinoxylan, arabinoglucuronoxylan, glucomannan, galactomannan and galactoglucomannan. Xylan is among the most found hemicelluloses, and mainly composed of β-(1→4)-xylose units. The xylan backbone is usually highly substituted with arabinose, glucuronic acid, acetyl groups or xylose [37].

Lignin is a water-insoluble, amorphous, three-dimensional phenolic heteropolymer of phenylpropane units (p-coumaryl, coniferyl and sinapyl alcohol). Lignin is responsible for structural support, impermeability, and resistance against microorganisms and oxidative stress [21]. Various types of linkages and functional groups such as methoxyl, phenolic hydroxyl, aliphatic hydroxyl, carboxyl and carbonyl groups, are found in the lignin structure, making it highly reactive [38].

Pectin is a group of complex heteropolysaccharides in the cell wall of higher plants [39]. All the pectic polysaccharides contain galacturonic acid linked at the O-1 and the O-4 position with alternating 'smooth' and 'hairy' regions [40]. The major type of pectin is homogalacturonan (HG), a linear (smooth) region composed of α (1→4)-linked galacturonic acids. In citrus, sugar beet, and apple, the homogalacturonan backbone contains an estimated of 72–100 galacturonic acid units (approximately, 60% of the total pectin; [41]. Rhamnogalacturonan-I (RG-I), is also located in the highly branched area and contains a large number of neutral sugars (e.g., arabinose, galactose, mannose) as side chains. HG-I represents up to 20–35% of pectin and contains a backbone of alternating units of α-(1→4)-galacturonosyl and α-(1→2)-rhamnosyl units [40]. Rhamnogalacturonan II (RG-II) is the most structurally complex pectin accounting for approximately 10% of total pectin. The building blocks of RG-II are galacturonic acid, rhamnose, galactose and unusual neutral sugars [42]. In general, pectin is made up of HG and RGs. Apart from the abovementioned sugars found in sidechains, several others are also present include xylose, glucose, fructose, and galactose [19]. Pectic polysaccharides are associated with cellulose and hemicelluloses in the cell wall structure and have a defensive role against physical injuries and pathogen attack [41].

## 3. Production of Functional Oligosaccharides

Functional oligosaccharides are nondigestible carbohydrates that potentially exert health benefits to the host. Many functional oligosaccharides available in the market are considered 'prebiotic' candidates. A prebiotic was first defined in 1995 as "a nondigestible food ingredient that beneficially affects the host by selectively stimulating the growth and/or activity of one or a limited number of bacteria in the colon, and thus improves host health" [43]. However, in 2010, the definition was revised and a new definition of "dietary prebiotics" is established as "a selectively fermented ingredient that results in specific changes in the composition and/or activity of the gastrointestinal microbiota, thus conferring benefit(s) upon host health" [44]. The latest update was in 2016, from which the prebiotic term refers to "a substrate that is selectively utilised by host microorganisms conferring a health benefit". This definition expands the prebiotic concept to include non-carbohydrate compounds, applications to body sites other than the gastrointestinal tract, and diverse categories other than food [45]. Prebiotics have long

been reported for their effects and crucial role in health maintenance and protection against illnesses and diseases [44,46]. The most studied functional oligosaccharides that fulfil the above definition and have obtained 'prebiotic' status include lactulose, inulin-type fructans, and trans-galactooligosaccharides [44,47]. Fructans and galactans are selectively metabolised by the genus *Bifidobacterium* because they mainly produce β-fructanosidase and β-galactosidase enzymes which are necessary to degrade the polymers [45]. Apart from these established prebiotics, many potential prebiotic candidates have been investigated, for example, xylooligosaccharides (XOS), isomaltooligosaccharides (IMO), pectic oligosaccharides (POS) [44].

Functional oligosaccharides can be derived from hemicellulose of FVSW (Table 3). As described in the previous section, the plant cell wall components are strongly intermeshed with each other in the form of a network. Various treatments have been introduced for the conversion of the parent components in the hemicellulose fraction into its respective products. They can be produced from FVSW via different approaches such as physico-chemical (hydrothermal process), chemical, (acid hydrolysis, and enzymatic hydrolysis [19,22,48]. Physico-chemical and enzymatic processes provide a greener and more economical approach than the chemical counterpart as it minimises the formation of undesirable by-products/toxic waste, hence the post-process cost of decontamination/disposal. Combination of methods has been shown to increase the effectiveness of the process [2].

**Table 3.** Main functional oligosaccharides derived from fruit-and-vegetable solid by-product and waste streams: sources, processes and yields.

| Functional Oligosaccharides | Source of Lignocellulosic Residues | Treatment | Optimal Conditions | Yields (Dry Weight Basis) | References |
|---|---|---|---|---|---|
| POS | orange peel | Enzymatic hydrolysis (commercial cellulases and pectinases) | 37 °C and pH 5 up to 20 h | 31 g/100 g peel (model predicted) | [49] |
| | | Pectin extraction and acid hydrolysis | HCl at100 °C, 18 h and Trifluoroacetic acid at 85 °C for 2.5 h | 20 g/100 g pectin (DP 6–18) | [50] |
| | Apple pomace | Pectin extraction and acid hydrolysis | HCl, 100 °C, 18 h and Trifluoroacetic acid at 85 °C for 2.5 h | 20 g/100 g pectin (DP 6–18) | [50] |
| | Sugar beet pulp | Hydrothermal treatment | 160 °C; Severity factor ($R_0$) = 326 min | 44 g/100 g solids (80% arabinooligosaccharides, 20% galactooligosacchrides) | [51] |
| | Lemon peel | Hydrothermal treatment | 160 °C; Severity factor ($R_0$) = 326 min | 24 g/100 g solids (60% arabinooligosaccharides, 40% galactooligosacchrides) | [51] |
| | Avocado peel | Hydrothermal treatment | 150 °C, severity ($S_0$) = 1.90 | 14 g/100 g peel Oligogalacturonides | [52] |
| | Passion fruit peel | Subcritical water treatment | 150 °C within 4.5 min, or 175 °C within 5.5 min.; Severity ($S_0$) = 3.4–5.1 | 21 g/100 g peel | [53] |
| | Mango peel | Pectin extraction (acid) and enzymatic hydrolysis (commercial polygalacturonase) | Acidhydrolysis: HCl, 80 °C. 3 h. Enzymatic: 50 °C; pH 4.5 up to 60 min | n.a. | [54] |
| | Melon peel | hydrothermal | 140 °C; Severity ($S_0$) = 2.03 | 15 g/100 g solids | [55] |

**Table 3.** *Cont.*

| Functional Oligosaccharides | Source of Lignocellulosic Residues | Treatment | Optimal Conditions | Yields (Dry Weight Basis) | References |
|---|---|---|---|---|---|
| XOS | Banana peel | Xylan extraction (water, acid or alkali) and enzymatic hydrolysis (*Aspergillus versicolor* endoxylanase) | 55 °C upto 48 h, pH 6 | 11 g/100 g hemicellulose (DP = 3–4) | [26] |
| | Orange bagasse | Xylan extraction (water, acid or alkali) and enzymatic hydrolysis (*Aspergillus versicolor* endoxylanase) | 55 °C upto 48 h, pH 6 | 10 g/100 g hemicellulose (DP = 3–4) | [26] |
| | Guava bagasse | Xylan extraction (water, acid or alkali) and enzymatic hydrolysis (*Aspergillus versicolor* endoxylanase) | 55 °C upto 48 h, pH 6 | 12 g/100 g hemicellulose (DP = 3–4) | [26] |
| | Pineapple peel | Xylan extraction (alkali and hydrothermal-assisted alkali) and enzymatic hydrolysis (*Trichoderma viridea* endoxylanase) | Hydrothermal-assisted: Enzymatic: 50 °C, 24 h, pH 5 | 26 g/100 g of xylan (84% xylobiose and 16% xylotriose) | [56] |
| | Corn cob | Xylan extraction (alkali) and ultrasound-assisted enzymatic hydrolysis (*Trichoderma viridea* endoxylanase) | 50 °C, 10 min, 200 W | 6 g/100 g corncob (Average DP = 2) | [57] |
| | | Xylan extraction (steam explosion) and enzymatic hydrolysis (*Paecilomyces themophila* J18 xylanase) | Steam: 188–204 °C, 2.5–7.5 min, S0 2.99–3.94 Enzymatic: 70 °C, 2.5 h, pH 7.0 | 29 g/100 g xylan (DP = 2–3) | [58] |
| | | Autohydrolysis | 145–200 °C in 40 min | 25 g/100 g xylan (DP = 2–5) | [59] |
| | | Autohydrolysis | 180–225 °C; Severity ($S_0$) = 3.75 | 25 g/100 g xylan (DP = 2–6) | [60] |
| Inulin | Garlic waste (damaged bulb, husk, paste) | Water extraction | 80 °C, 45 min | 8 g/100 g waste | [61] |
| | Artichoke root (Five cultivars) | Water and ultrasound-assisted extraction | Water: 80 °C, 2 h, pH 6.8 Ultrasound: 80 °C, pH 6.8, 70% sonication amplitude, 5 min | 7–21 g/100 g root | [62] |
| | Artichoke waste (bract, stem, leaves) | Ultrasound-assisted extraction | 70 °C, 2 h, 40 Hz | 5–28 g/100 g waste (DP = 32–42) | [63] |
| | Artichoke bract | Ultrasound-assisted extraction | 70 °C, 30 min, 37 Hz | 7 g/100 g bract | [64] |
| | *Stevia rebaudiana* Berton (stem) | Water extraction | 80 °C, 4 h | 4 g/100 g stem | [65] |
| FOS | *Stevia rebaudiana* Berton (stem) | Water extraction | 80 °C, 4 h | 11 g/100 g stem | [65] |
| | Artichoke waste | Ultrasound-assisted extraction | 360 W, 10 min, 60 °C | 1 g/100 g waste (DP = 2–4) | [66] |
| | Banana peel | Solid state fermentation by *Aspergillus flavus* NFCCI 2364 strain | 28 °C, 96 h | 7 g/100 g peel | [67] |
| | Apple pomace | Solid state fermentation by *Aspergillus flavus* NFCCI 2364 strain | 28 °C, 96 h | 5 g/100 g pomace | [67] |
| | Corn cob | Solid state fermentation by *Aspergilus oryzae* CFR 202 | 30 °C up to 120 h | 15 g/100 g waste | [68] |

**Table 3.** *Cont.*

| Functional Oligosaccharides | Source of Lignocellulosic Residues | Treatment | Optimal Conditions | Yields (Dry Weight Basis) | References |
|---|---|---|---|---|---|
| IMO | Potato processing waste | Acid hydrolysis and enzymatic liquefaction, saccharification and transglucosylation (enzyme cocktail: a commercial bacterial α-amylase, a commercial fungal α-amylase, and *Aspergillus niger* PFS08 α-amylase) | Acid hydrolysis: 0.7 M HCl, 3 h reflux Transglucosylation: 55 °C, 12 h, pH 5.0 | 92 g/L reaction mixture (DP = 2–5) | [69] |
| | Rejected unripen plantain fruits | Enzymatic liquefaction, saccharification and transglucosylation of flour made from rejected fruits (commercial α-amylase, β-amylase, pullulanase, α-glucosidase) | Transglucosylation: 50 °C, 30 h, pH 4.5, | 51 g IMO/100 g maltose; (DP = 2, 4–5) | [70] |

### *3.1. Pectic Oligosaccharides*

Pectic oligosaccharides are prebiotic candidates exerting physiological effects beneficial to the human health [71]. The most common and well-known POS are arabinogalacto-oligosaccharides, arabinoxylo-oligosaccharides, arabino-oligosaccharides, galacto-oligosaccharides, oligo-galactouronides and rhamnogalacturonan-oligosaccharides [42]. Pectin from different plants differ in terms of the HG chain length, proportion of RG of total pectin, and types of neutral and acid sugars present [48]. Physico-chemical properties and physiological effects of the resulting POS products therefore are significantly influenced by sources of pectin as well as depolymerisation parameters [72].

POS can be produced by a partial breakdown of pectin fractions, therefore pectin-rich food residues are considered ideal candidates for this purpose. Many food by-products, such as peels (citrus, banana, melon, avocado), sugar beet pulp, apple pomace, have been used as raw material for POS production (Table 3). Reviews on production technologies, covering hydrolysis (acid or enzyme), hydrothermal treatment, dynamic high-pressure microfluidisation, microwave-assisted, ultrasound-assisted extraction on POS production from FVSW have been published [48,71].

#### 3.1.1. Acid hydrolysis

Pectin in FVSW can be partially hydrolysed under acidic pH and high temperature. The method is simple; however, it is not considered ideal due to its high environmental impact [19]. Studies on POS production by acid hydrolysis using commercial pectin as starter material have been previously described [73,74]. A number of studies attempting to use those methods on food residues have also been conducted [50,75,76].

The general process is to extract pectin from food residues, firstly by acid hydrolysis, followed by filtration, alcohol precipitation and lastly drying. The extracted pectin is then further partially hydrolysed using acid (hydrochloric, sulphuric, or nitric acid; pH 1.5–2.0) to yield the desired sugar composition before neutralisation by an alkali. The hydrolysates may be further concentrated and dried to get the final product [76]. Cano et al. [50] described a procedure of two consecutive acid treatments (hydrochloric acid followed by trifluoroacetic acid), which allowed the recovery of a fraction containing medium size pectic oligosaccharides (mixture of oligogalacturonic acid, DP between 6 and 18) from apple pomace and orange peel. A study on pectin and POS production from orange albedo from orange juice manufacturing reported that POS was a notable component of the supernatant remaining after pectin precipitation (mixture of rhamnogalacturonan and xylogalacturonan

pectic oligosaccharides, 8% yield) and could successfully be recovered by nanofiltration [75]. Conversion of pectin extracted from pomelo peel into POS (DP 2–5) with hydrogen peroxide followed by microwave heating and alcohol precipitation was also feasible [77].

#### 3.1.2. Enzymatic Process

POS can be obtained from depolymerisation of pectin by the action of enzymes. Some pectin-degrading enzymes commonly used to produce POS are polygalacturonase (EC 3.2.1.15), pectin lyase (EC 4.2.2.10), and pectin esterase (EC 3.1.1.11) [48]. The hydrolysis of the smooth (galacturonan) and hairy (rhamnogalacturonan) regions requires different enzymes. Depolymerisation of the galacturonan backbone (smooth region) needs the actions of pectin esterases and polygalacturonases, whereas that of the rhamogalacturonan chains (hairy region) needs rhamnogalacturonan hydrolase, rhamnogalacturonan lyase, and rhamnogalacturonan acetyl esterase. The removal of side chains from RG-I required many others, depending on the sidechains themselves, such as arabinofuranosidase B, arabinases, endogalactanase, β-galactosidase [42].

Two main approaches are reported in the literature; (1) direct hydrolysis of FVSW to POS, or (2) pectin extraction from FVSW prior to conversion. The enzymes used can be commercial or from microbial fermentation, such as *Aspergillus* species (polygalacturonases and lyases), *Rhizopus* species (polygalacturonases), and *Bacillus* species (lyases) [78]. Both production approaches were studied on a range of FVSM with high-pectin content, such as sugar beet pulp [79–82], orange peel [83–85], bergamot peel [86], mandarin peel [87], mango peel [54,88]. Tailored POS production from onion skins using a commercial endo-polygalacturonase was also investigated [89]. The use of membrane technology in combination with the enzymatic conversion was investigated. It was reported that using a cross-flow continuous membrane enzyme reactor (10 kDa membrane cut-off) improved volumetric productivity (3–5 times) of onion-skin-derived POS [90]. The use of ultrafiltration (1 kDa membrane cut-off) successfully fractionated and recovered POS post enzymatic conversion of artichoke pectin [91].

#### 3.1.3. Hydrothermal Treatment

Among the reports on POS production from FVSW, hydrothermal treatment has attracted considerable attention. The hydrothermal process is a thermochemical process. The process can be categorised into two reaction conditions: subcritical (usually 250–374 °C at 4–22 MPa) and supercritical water conditions (above 374 °C and 22.1 MPa) [92]. The conditions applied for the conversion of FVSW into oligosaccharides is under the subcritical range, and in this context, sometimes the method is referred to as 'autohydrolysis' and 'subcritical water treatment' (Table 3).

Hydrolysis of plant cell wall under hydrothermal treatment is based on the ionisation of water at elevated temperatures. Above 120 °C, water molecules get ionised, thereby increasing the concentration of $H_3O^+$ and enabling the dissociation of intramolecular β-(1–4) glycosidic bonds and intermolecular hydrogen bonds of carbohydrates [93]. Hydrolysis of plant cell wall components takes place above 150 °C for a short period of time. Resistance to decomposition under subcritical water conditions of plant cell wall components is predominantly influenced by the level of crystal structure present. Due to this, cellulose is the most resistant in comparison to hemicellulose, lignin, and pectin [92]. Apart from POS, glucooligosaccharides, galactooligosacchatides, and xylooligosaccharids are also derived from the raw materials to a certain extent [51,52,55]. A severity factor ($R_0$; temperature profiles during the heating and the cooling of the reactor according to the respective residence times), and severity ($S_0$; logarithmic of $R_0$) can be calculated and used for evaluation and comparison of the effect of different process conditions [94]. The yield and profile of oligosaccharides obtained depend on the source of biomass, and process parameters [49,51]. The yield of the targeted oligosaccharides is controlled by severity. Severity promotes the hydrolysis of hemicellulose to the targeted products, up to a certain level, beyond which the formed oligosaccharides become hydrolysed to smaller oligomers [52,56].

POS production from sugar beet pulp [51,95], lemon peel [51], orange peel [96], avocado peel [52] indicated temperatures between 150 and 160 °C as optimal, whereas the $S_{0,}$ between 1.9 and 5.1, is high enough to solubilise the galacturonan fraction from the pectin residues (Table 3). Gonzalez-Garcia et al. [97] assessed the environment impact of hydrothermal and enzymatic processes of POS production from sugar beet pulp. The hydrothermal process is energy intensive as high-running temperature is needed, nevertheless, the process to obtain the enzymes needed also requires a large amount of energy as well as chemicals for the formulation of the culture medium. Between the two approaches, the authors concluded that the hydrothermal process imposed less burden to the environment than the enzymatic process because it provided a higher yield (approximately 20%) and lower monosaccharide contamination (approximately 300%), which required less energy input in the downstream process [97].

### *3.2. Inulin and Fructooligosaccharides*

Inulin and fructooligosaccharides (FOS) are fructan-type carbohydrates. Inulin is a mixture of fructose oligomers and polymers that have β-(2→1) fructosyl-fructose linkages. A glucose molecule typically resides at the end of each fructose chain and is linked by an α-(1→2) bond. In vegetable origin inulin, the fructose chain lengths of these fructans range from 2 to 60 units, with an average DP of 10 [98]. Many vegetables, such as artichoke, garlic, onion, and chicory, are high in inulin content. Inulin can be retrieved from plant materials by a conventional extraction process. On an industrial scale, inulin is extracted from plants i.e., chicory root, Jerusalem artichoke, and dahlia, with higher than 10% inulin by weight, at high temperature (70–80 °C) [99]. Alternative methods such as enzyme, supercritical carbon dioxide, simultaneous ultrasonic, microwave, and pulsed electric field have been investigated to improve the extraction performance [100].

Recovery of inulin from FVSW was performed from artichoke and garlic wastes [99]. It was reported that in globe artichoke (*Cynara scolymus*), the stem gave the highest inulin yield (28 g/100 g waste) in comparison to those obtained from the bract (16 g/100 g waste) and leaves (5 g/100 g waste) after ultrasonic-assisted extraction (70 °C, 2 h, 40 Hz) [63]. Another study (ultrasonic-assisted extraction at 70 °C, 37 Hz for 30 min) reported the bracts gave 7.5 g inulin/100 g waste whereas the stem gave insignificant amounts and demonstrated that the yield was affected by seasonality [64]. Garcia-Castello et al. [13] compared the effectiveness of water and ethanol-water extraction in recovering inulin from artichoke waste. These authors demonstrated that, at optimal conditions, higher inulin yield (approximate 40%) was obtained from water extraction (89 °C, 139 min) than from ethanol-water extraction (22.4% ethanol, 80 °C, 217 min) [13]. Castellino et al. [62] studied the inulin extraction from five cultivars of artichoke roots and compared the yields obtained from conventional water extraction and ultrasound-assisted extraction. The results demonstrated the ultrasound-assisted approach did not always improve the yields as it worked only on certain cultivars. The authors concluded that it is not only the extraction method that affects extraction yields, but also genetic and pedo-climatic variables [62].

Apart from artichoke, other FVSW sources were also studied for inulin extraction [61,65]. Water extraction of garlic manufacturing waste at 80 °C gave a yield of 8 g/100 g waste [61]. In a recent study, Lopes et al. [65] simultaneously extracted inulin and fructooligosaccharides (FOS) from the stem of *Stevia rebaudiana* Bertoni using hot water extraction (80 °C reflux for 4 h). A higher yield of FOS (11 g/100 g stem) was obtained compared to that of inulin (4 g/100 g stem) [65].

Fructooligosaccharides (α-D-glucopyranosyl-[β-D-fructofuranosyl]n−1-D-fructofuranosides) belong to the inulin-type fructan family and they are one class of the established prebiotics [101]. Fructooligosaccharides are derivatives of inulin, with a terminal glucose unit linked by β-(2→1) glycosidic bond (DP = 2–10, average DP = 5) [102]. Native inulin can be converted into FOS by the enzyme inulinase [103]. Physiological effects and modulation of the gut microflora by FOS has been extensively studied in the last few decades and substantial evidence was acquired, granting FOS status as one of a few established

prebiotics [44,104–106]. On an industrial scale, FOS can be produced by either hydrolysis of inulin (enzymatic or chemical) or enzymatic synthesis from sucrose via transfructosylation by β-fructofuranosidase (3.2.1.26) and fructosyltransferases (2.4.1.9) [107]. For the enzymatic synthesis approach, a high initial sucrose concentration is required to induce transfructosylation [108].

Reports on the use of by-products and wastes in the production of FOS are mostly on agricultural residues because high yields can be obtained [109]. Examples include solid state fermentation using sugar cane bagasse (36% FOS yield) [67], rice and wheat brans (approximately 50% FOS yield) [68]. With an emphasis on FVSW, the studies on production of FOS were mainly on their potential as substrates to produce β-fructofuranosidases and fructosyltransferases or biofuels with FOS as co-products [67,68].

Santiago et.al. [110] modelled industrial scale FOS extraction as targeted co-products in the biorefinery process using onion solid waste as main raw material. The process can be achieved by pretreatment (drying and size reduction), conventional solvent extraction (ethanol), and product recovery (centrifugation, membrane filtration, and evaporation). Nevertheless, the authors stated that the bespoke process posed high environmental loads and alternative methods (e.g., microwave and ultrasound-assisted extractions) should be explored [110]. A study by Machado et al. [66] concluded that direct ultrasound increased the speed of extraction, and a sonication treatment 360 W at 60 °C for 10 min offered maximum yield (22% dry weight basis) and great economical energy saving.

Studies on the enzymatic process of FOS on FVSW were mostly performed using the residues as a solid support or nutrients for solid state fermentation by fungi, especially *Aspergilus* species, and, to a lesser extent, *Aureobasidium*, and *Rhizupus* species [109,111]. Fructosyltransferases obtained from microorganisms are single enzymes with both transferase and hydrolase activities [68]. Apart from the process parameters, the origin of the residues and enzymes affect the yield and structures of the FOS obtained. The results from a number of studies indicated that agricultural residues (i.e., sugarcane bagasse, spent coffee, spent tea, rice bran, wheat bran, corn cob; 12–60% yields) give significantly higher yields of FOS than those obtained from FVSW (e.g., pineapple peel, banana peel, apple pomace; 2–7% yields) [67,68,109,111,112]. A comparative study using various low-cost agricultural wastes as substrates for solid state fermentation to produce fructosyltransferase from *Aspergillus flavus* NFCCI 2364 indicated that among FVSW tested (apple pomace and peels of banana, beet root, orange, guava, pineapple, papaya, mango, passion fruit), banana peel was the most promising raw material for FOS and enzyme production, yielding 7 g FOS/100 g waste [67].

### *3.3. Xylooligosaccharides*

Xyloooligosaccharides (XOS) are a linear chain of xylose joining together by β-(1→4) glycosidic bonds with a DP range between 2 and 20 [22]. XOS is obtained from the hydrolysis of xylan in hemicellulose [2]. Xylan is a complex of a carbohydrate group including xylan, xyloglucan (heteropolymer of D-xylose and D-glucose), glucomannan (heteropolymer of D-glucose and D-mannose), galactoglucomannan (heteropolymer of D-galactose, D-glucose and D-mannose) and arabinogalactan (heteropolymer of D-galactose and arabinose) [113]. The general procedure for XOS production from biomass is a pretreatment (xylan extraction) followed by XOS conversion by hydrolysis [114]. Pretreatments include hydrothermal and/or chemical process, whereas XOS conversion is enzymatic or acid hydrolysis [22,33]. Xylan is soluble in alkali and its solubility depends on the alkali concentration [16,115]. Other extraction parameters (temperature, time, solute-to-solvent ratio) also affect xylan yield [57]. The use of pressurised steam or alkali solution (121–150 °C) enhances the effectiveness of the extraction [16,116], and may help to reduce the amount of chemicals used in the process without compromising the yield [56]. The use of emerging technologies such as microwave-assisted delignification and ultrasound-assisted xylan extraction helped to increase the rate of extraction with lower energy usage [117]. The extracted xylan is subsequently hydrolysed by acid or xylanases (EC 3.2.1.8). Xylanase is

a glycoside hydrolase cleaving the β-(1→4) bond in the xylan backbone, yielding short xylooligomers [113]. Employing ultrasound during the enzymatic hydrolysis stage is reported to lower the diffusion-limiting barrier between enzymes, therefore, increasing the rate of enzyme-substrate binding [37]. XOS can also be produced directly from biomass by hydrothermal process [59,60]. The products derived from this approach contain variable DP and a rich substitution pattern because functional groups are not completely removed as observed in the alkaline extraction [59]. However, undesirable by-products or excessive amounts of monosaccharides could also be produced and may need further fractionation [60]. For this reason, the 2-stage method is preferable because process control can be achieved more easily [57].

Being the most abundant biomass and rich in xylan, forestry and agricultural residues (such as bagasse, straws, stalks, leaves), have been extensively studied as raw materials for XOS production [2,20,22,50,114]. The yields of XOS from forestry and agricultural residues depends on the raw materials and the process conditions [114]. Cotton stalk and wheat straw pretreated with alkali followed by acid hydrolysis gave a yield range of 8–10%, which is relatively low in comparison to cane bagasse and switchgrass pretreated with acid followed by hydrothermal or hydrolysis by enzyme or acid (40–80%) [2,22]. With regards to FVSW in particular, the yields of XOS obtained are in the 10–30% range, with corn cob and pineapple peel giving more than 20% yields (Table 3).

Pereira et al. [26] demonstrated that different alkali treatments (NaOH, KOH, and $H_2O_2$) affected the solubility of hemicellulose from different FVSW (banana peel, guava bagasse, and orange bagasse), and therefore the final XOS yields post enzymatic treatment (purified *Aspergillus versicolor* endoxylanase). Highest XOS yields (DP = 2–4; dry basis) were obtained from KOH-pretreated banana peel (15%), $H_2O_2$-pretreated guava bagasse (7%) and KOH-pretreated orange bagasse (13%) [26]. However, Samanta et al. [16] reported that NaOH was more effective than KOH in xylan extraction from corn cob.

Reports on XOS production from different parts of pineapple waste (peel, pomace, bagasse, and core) have been published [56,118,119]. The conditions of pretreatments and sources of enzymes used in these studies were different. Nonetheless, significantly higher XOS yield (approximately 10 times) was obtained in the investigation of pineapple peel pretreated with hydrothermal-assisted alkali extraction (121 °C and 15 psi pressure) and enzymatic hydrolysis (a commercial xylanase; 26 XOS g/100 g of xylan; DP = 2–3) [56].

One of the most promising FVSW for XOS production is corn cob as its hemicellulose fraction is rich in xylan (30–40% hemicellulose) [16]. Xylose units accounted for approximately 75% (*w*/*w*) of XOS obtained [59]. Many investigations on XOS production from corn cob by varied process parameters and yields have been reported. Samanta et al. [16,115] reported that the hydrolysis of steam-assisted alkali-pretreated corn cob xylan by a commercial *Trichoderma viride* endoxylanase gave a better yield (1.2 mg XOS/mL reaction mixture) than that by sulphuric acid (0.8 mg XOS/mL reaction mixture). Endo-xylanases from many microbial species are highly effective in converting xylan into XOS with different profiles of chain length. Khangwal et al. [120] reported that alkali-pretreated xylan by *Thermomyces lanuginosus* VAPS-24 endoxylanase gave 3.0 mg XOS/mL reaction mixture. Xylanase obtained from *Paecilomyces themophila* J18 was able to produce XOS (60% DP 2 and 40% DP 3) from hydrothermal-treated corn cob xylan satisfactorily, achieving 29 g XOS/100 g xylan [58]. Endo-xylanase from *Streptomyces thermovulgaris* TISTR1948 produced XOS from alkali-pretreated xylan showing a different profile (52% DP 2, 12% DP 3, 15% DP 4, 21% DP 5) and yield (75 g XOS/100 g xylan) [121]. Non-enzymatic process such as hydrothermal treatment (135 °C) of acid-delignified corn cob hemicellulose also offered a high XOS conversion (68 g/100 g xylan; DP = 2–3) [116].

### *3.4. Isomaltooligosaccharides*

Isomaltooligosaccharides (IMO) are chains of glucose units joined together mainly with α-D-(1→6) linkages, with a small proportion of α-(1→4), α-(1→3) or α-(1→2) linkages [108]. Different oligomers exists in each class of IMO according to the position of

the linkages, for example, IMO with DP = 2 involve isomaltose, nigerose, kojibiose, and IMO with DP = 3 involve isomaltotriose, panose, isopanose. Long-chain IMO (DP = 4–10) and cyclo-IMO (DP 7–12) also exist [122]. IMO is produced commercially by the hydrolysis of starch to α-D-Glu-(1→4) oligomers using α-amylase (EC 3.2.1.1), pullulanase (EC 3.2.1.41) and β-amylase (EC 3.2.1.2) and the conversion of these oligomers to α-D-Glu-(1→6) oligosaccharides through the action of the α-transglucosidase (EC 2.4.1.24) from *Aspergillus* sp. [108]. Enzymatic IMO production has been investigated using starch-rich raw materials such as rice [123], sweet potato [124], cassava [125], tapioca [126], unripened banana [127]. With regard to IMO production from FVSW, unlike other functional oligosaccharides, only the use of potato processing waste [69] and rejected unripened plantain fruits have been reported [70].

Basu et al. [69] investigated the potential of IMO production from potato processing waste using simultaneous saccharification and transglucosylation in comparison with consecutive saccharification and transglucosylation. The authors indicated that although the yields from both processes at the optimal conditions were not different (approximately 90 g IMO/L), the simultaneous approach led to a lower accumulation of glucose in the reaction mixture (86 g/L in simultaneous and 110 g/L in consecutive saccharification and transglucosylation, respectively). In another study, plantain flour was obtained from rejected unripened plantain fruits, and subsequently subjected to the conventional IMO production steps (i.e., liquefaction, saccharification, and transglycosylation) via the actions of commercial *Bacillus licheniformis* α-amylase (EC 3.2.1.1), barley β-amylase (EC 3.2.1.2), *Bacillus licheniformis* pullulanase (EC 3.2.1.41), and *Aspergillus niger* α-glucosidase (EC 3.2.1.20) [70]. The IMO yield (DP = 4–5) was 51 g/100 g maltose. The authors indicated that it was economically feasible to produce IMO from rejected unripened plantain fruits on a large scale, providing a co-process (for example single cell protein) concurrently run to convert waste steams into another value-added product [70].

## 4. Conclusions

The amount of FVSW generated from the manufacturing lines of the food industry worldwide undeniably imposes a huge post-production environmental load. Many studies have demonstrated that the derived biomass is rich in lignocellulose of varied chemical structures and composition. Instead of using conventional disposal methods to manage the waste, reintroducing those discarded/unwanted parts of fruits and vegetables as low-cost materials for recovery/conversion into functional oligosaccharides to promote a circular economy would be a better approach. Functional oligosaccharides and their beneficial physiological effects to the host, in many ways beyond the gut health, have been studied extensively. They are widely applicable in various product segments reaching consumers of all ages. As the hemicellulose and pectin composition depends on the sources, seasonality, and genetics of FVSW themselves, it is feasible to extract or convert those lignocellulosic fractions containing parent molecules into their respective functional oligosaccharides. This approach would add value to the wastes by turning them into higher-value products.

Many attempts have been made to produce POS, inulin, FOS, XOS, and IMO from variety of fruits and vegetable wastes, using chemical, physico-chemical, and enzymatic processes. The use of emerging technologies, mainly ultrasound and microwave, to optimisethe production process has also been investigated. Nevertheless, there are still some challenges that need to be addressed before scaling up the processes. Firstly, it can be observed from the information available that the yields of these processes varied considerably between the reports published. This is due to the diverse FVSW composition and oligosaccharide structures. Strategies to improve the yield for prospective raw materials need further exploring, especially the use of emerging technologies in order to minimise environmental impact. So far, yield improvement has focused on the use of ultrasound-assisted, microwave-assisted, and subcritical water conversion processes. Investigation on the application of other emerging technologies (such as pulsed electric field) or new sources of enzymes should also be explored. Another challenge requiring further in-depth

studies is to identify suitable strategies for the tailored production of functional oligosaccharides with targeted DPs, which could enhance their physiological performance. Lastly, techno-economic feasibility also needs to be evaluated in order to assess if the process can be scaled up. As demonstrated in the previous sections, only a few studies have explored this area so far. In order to ensure sustainable circular integration and waste management, the aforementioned gaps on identifying suitable sources for each oligosaccharide, improvement of yields of the targeted oligosaccharides, and minimizing the environmental impact of the production processes still need to be filled.

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