Polysaccharides

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Polysaccharides from agro-food industry waste constitute one of the most important renewable resources. The great variety of their chemical composition and structure, and their biodegradability and safety make them ideal for application in diverse fields, such as the food, pharmaceutical, cosmetics, tissue engineering and biofuels industries, among others.

Keywords: agricultural waste ; polysaccharides ; bioactive function ; extraction methods ; nutraceuticals

1. Polysaccharides from Agricultural Waste

Nowadays, 37 million tons of agricultural residues are generated worldwide [1] causing a serious economic and environmental problem [2]. These residues are made up of waste and plant by-products, such as skins and seeds. Plant by-products are rich sources of dietary fiber, soluble polysaccharides, phenolic compounds and fatty acids, making them particularly interesting for use as additives and functional ingredients.

Polysaccharides from agro-food industry waste constitute one of the most important renewable resources. The great variety of their chemical composition and structure, and their biodegradability and safety make them ideal for application in diverse fields, such as the food, pharmaceutical, cosmetics, tissue engineering and biofuels industries, among others [3][4].

Natural polysaccharides from agricultural residues form part of the plant cell walls that are highly variable in terms of structure and composition [5]. In general, cell walls are composed of high molecular weight polysaccharides, which are mainly lignin, cellulose, hemicelluloses, pectins and other non-starch polysaccharides, such as inulin and oligosaccharides [6]. Polysaccharides that form cell walls are classified as insoluble and soluble based on their ability to be soluble in water. Insoluble polysaccharides are lignin, cellulose, hemicelluloses and pectins (insoluble); the soluble polysaccharide group consists of other pectins and hemicelluloses [7].

Composed exclusively of β-glucose molecules linked by β-1,4-glycosidic bonds, cellulose is characterized by its capacity for chemical modification and hydrophilicity [8]. Cellulose is found in different agricultural residues, such as garlic skin [9], corn [10], grape pomace [11] and carrot [12]. Cellulose has multiple applications in the food industry; among others, its application as a fat substitute [13] has been proven to improve food texture [14], and it has also been widely used as a film to protect food. [15] manufactured cellulose-based coatings from Chinese chive root extract, and the results showed that the coatings possessed antioxidant and antimicrobial properties.

Hemicellulose is a heteropolysaccharide, composed of a heterogeneous set of polysaccharides, itself composed of two types of monosaccharides linked by β-bonds, which form a branched linear chain. It is the second most abundant component of agricultural residues, representing approximately 20–40% [16][17]. Hemicelluloses include glucans, xyloglucans, mannans, xylans and β-(1→3,1→4)-glucans [18]. However, the polysaccharides that constitute the majority of hemicelluloses are mannan and xylan [19].

Xylan is the most abundant hemicellulose found in nature [20]. Xylan from agricultural residues can be hydrolyzed and converted into xylose; furthermore, it can be turned into xylooligosaccharides (XOS) with different degrees of polymerization [21]. Aachary and Prapulla [22] reported that XOS with a degree of polymerization of 2–3 have maximum prebiotic potential. XOS are extracted industrially from corn and sugarcane [23][24], although they can also be obtained from agricultural by-products such as pineapple rind [25], straw rice [26] and quinoa stems [27].

Another polysaccharide that is part of the hemicelluloses is mannan, which is significantly present in agricultural residues. By enzymatic hydrolysis, mannan can be turned into mannooligosaccharides (MOS) [28]. MOS are considered as emerging prebiotics and can be obtained from different agricultural residues, such as copra flour [29].

Pectins, mostly considered soluble fiber, are part of the cell wall of plants and are heteropolysaccharide polymers rich in polygalacturonic acid that can be composed of up to 17 different monosaccharides [30], which is why it is characterized as one of the most structurally complex natural plant polysaccharides [31]. It is composed of three structurally distinct domains: homogalacturonan (HG), rhamnogalacturonan (RG-I) and rhamnogalacturonan (RG-II).

Pectins are traditionally obtained from agricultural by-products, such as citrus peels and apple pomace [32][33]. The increasing global demand for this heteropolysaccharide due to the numerous health benefits attributed to it [34] means that alternative sources of pectin are being sought in other vegetables and by-products, such as eggplant [35], tomato peel [36][37], broccoli stem [38] and pomegranate peel [39], among others.

Methoxy esters, located in the C6 carboxyl groups of D-galacturonic acid, are substitutions generally found in the HG region and play an important role in the known functional properties and health benefits of these pectic polysaccharides [40]. It is composed of a repeating backbone of galacturonic acid and rhamnose disaccharide, usually with neutral side chains [41]. Although less common in the pectic fraction, RG-IIs are polysaccharides with abundant bioactive properties and many human health benefits. Their structure comprises a main chain linked to galacturonic acid and side chains of highly complex oligosaccharides and other unusual monosaccharides [42].

In addition, depolymerization of pectin releases pectic oligosaccharides (POS) [43]. POS are currently described as emerging prebiotics with numerous health benefits [44].

2. Extraction of Polysaccharides: Methods and Influence on the Bioactive Function

The different extraction methods, the extraction solvent, the pH, the ratio of raw material to solvent, the temperature and the time have a significant influence on the yield, technological properties and functions of bioactive polysaccharides [45][46]. Each extraction method has its advantages and disadvantages; therefore, the extraction method chosen should be adapted to the final purpose, the nature of the by-product and the cost of the procedure.

Table 1 shows an overview of the optimized extraction methods and their influence on the bioactive function of polysaccharides obtained from plant by-products.

Table 1. Optimized extraction methods and their influence on the bioactive function of polysaccharides obtained from plant by-products.

Optimized Extraction Method	Compounds	By-Products	Influence on Bioactive Function	Reference
Hot water extraction	Polysaccharides	White mulberry	Anti-diabetic, immunomodulatory, anti-inflammatory, antioxidant, hepatoprotective, renoprotective and anti-obesity activity; effect on gut microbiota	[47]
Hot water extraction	Polysaccharides	Watermelon rind	Antihypertensive and antioxidant activity	[48]
Hot water extraction	Polysaccharides	Pomegranate fruit	Prebiotic activity	[49]
Hot water extraction	Polysaccharides	Oleaster fruit	N.d. *	[50]
Ultrasound-assisted extraction	Polysaccharides/starch, pectin	Yam tubers, fruit peel, tomato processing, potato…	Antioxidant, anticoagulant, antitumor, anti-inflammatory and prebiotic activity	[51]
Ultrasound-assisted extraction	Polysaccharides/pectin	Fruit and vegetable peel: eggplant	Antioxidant activity	[35]
Ultrasound-assisted extraction	Polysaccharides/pectin	Pomegranate peel	N.d. *	[52]
Ultrasound-assisted microwave extraction	Polysaccharides/pectin	Tomato peel	N.d. *	[53]
Ultrasound-assisted extraction	Polysaccharides/pectin	Custard apple peel	N.d.*	[54]

Optimized Extraction Method	Compounds	By-Products	Influence on Bioactive Function	Reference
Ultrasound-assisted extraction	**Polysaccharides/pectin**	**Mango peel**	**N.d. ***	[55]
High hydrostatic pressure and ultrasound-assisted extraction	**Polysaccharides/pectin**	**Tomato peel**	**N.d. ***	[36]
Ultrasound-assisted extraction	**Hemicellulose polysaccharides/xyloglycans**	**Grape pomace**	**N.d. ***	[56]
Ultrasound-assisted extraction	**Fructooligosaccharides**	**Artichoke industrial waste**	**Prebiotic activity**	[57]
Microwave-assisted extraction	**Polysaccharides**	**Marshmallow roots**	**Antioxidant, antimicrobial and antitumor activity**	[58]
Microwave-assisted extraction	**Polysaccharides**	***Chuanminshen violaceum* root**	**Increased antioxidant activity**	[59]
Microwave-assisted extraction	**Polysaccharides/pectin**	***Carica papaya* L. peel**	**N.d. ***	[60]
Microwave-assisted extraction	**Polysaccharides**	**Kiwifruit**	**Antioxidant activity**	[61]
Microwave-assisted extraction	**Polysaccharides**	***Camptotheca acuminata* fruits**	**Antioxidant and immunomodulatory activity**	[62]
Surfactant and microwave-assisted extraction	**Polysaccharides/pectin**	**Orange peel**	**N.d. ***	[63]
Microwave-assisted extraction	**Polysaccharides**	**Waste jamun fruit seeds**	**N.d. ***	[64]
Microwave-assisted extraction	**Polysaccharides**	***Sargassum pallidum***	**Hypoglycemic activity**	[65]
Microwave-assisted extraction	**Polysaccharides/pectin**	**Banana peel**	**N.d. ***	[66]
Hot-solvent microwave extraction	**Polysaccharides/pectin**	**Pomelo peel**	**N.d. ***	[67]
Microwave hydrodiffusion and gravity	**Polysaccharides**	**Broccoli**	**N.d. ***	[68]
Microwave-assisted extraction	**Polysaccharides**	**Cocoa bean shell**	**Antioxidant activity**	[69]
Microwave-assisted extraction	**Polysaccharides/pectin**	**Fruit peels**	**N.d. ***	[70]
Microwave-assisted extraction	**Hemicellulose polysaccharides/xyloglycans**	**Tobacco plant residues**	**N.d. ***	[71]
Enzyme-assisted extraction	**Polysaccharides**	***Fritillaria pallidiflora* Schrenk**	**Antioxidant, antimicrobial, anti-inflammatory, antitumor and antihypertensive activity**	[72]
Enzyme-assisted extraction	**Polysaccharides**	***Malva sylvestris* plant**	**Increased antioxidant, antitumor and antimicrobial activity**	[73]
Enzyme-assisted extraction	**Polysaccharides**	**Cup plant (*Silphium perfoliatum* L.)**	**Antioxidant and hypoglycemic activity**	[74]
Enzyme-assisted extraction	**Polysaccharides/pectin**	**Kiwi pomace**	**N.d. ***	[75]
Enzyme-assisted extraction	**Polysaccharides/pectin**	**Apple pomace**	**Antioxidant and anticancer activity**	[76]

Optimized Extraction Method	Compounds	By-Products	Influence on Bioactive Function	Reference
Enzyme-assisted extraction	**Polysaccharides/pectin**	**Pomegranate peel**	**Antioxidant activity**	[77]
Enzyme-assisted extraction	**Polysaccharides**	***Dendrobium chrysotoxum***	**Immunological activity**	[78]
Enzyme-assisted supercritical fluid extraction	**Polysaccharides**	**Pomegranate peel**	**Antioxidant activity**	[79]
Supercritical fluid extraction	**Polysaccharides**	***Artemisia sphaerocephala* Krasch. seeds**	**N.d.***	[80][81]
Supercritical fluid extraction	**Polysaccharides**	**Pomegranate peel**	**Antioxidant activity**	[81]
Deep extraction with eutectic solvent/microwave-assisted extraction	**Polysaccharides**	**Bladder-wrack (*Fucus vesiculosus*)**	**Antioxidant activity, cell growth inhibition**	[82]
Accelerated solvent extraction	**Polysaccharides**	**Bamboo shoots**	**Antioxidant activity**	[83]
Dynamic high-pressure microfluidization	**Polysaccharides**	***Nelumbo nucifera* leaves**	**Antioxidant activity**	[84]
Ultrasonic-cellulase synergistic extraction	**Polysaccharides**	**Pineapple pomace**	**Hypoglycemic and anticancer activity**	[85]
Deep extraction with eutectic solvent	**Polysaccharides/pectin**	**Pomelo peel**	**N.d. ***	[86]

* Not determined.

HWE is one of the most widely used methods to extract polysaccharides, being conventional, simple and cheap. However, the use of HWE is limited due to its low yield; only extracellular polysaccharides can be obtained since the cell wall is not degraded [87]. High temperatures and long extraction times are needed to achieve high yields [47], which results in degradation of the structure and a decrease in quality and bioactivity [88][59]. Therefore, there is a need to explore new methods of polysaccharide extraction that ensure a good yield besides maintaining the bioactive and functional characteristics of the polysaccharides.

UAE is based on a phenomenon called acoustic cavitation, which involves the generation and formation of gas vapor-filled bubbles in a liquid that expand and finally collapse. Cavitation generates circulating liquid currents and turbulence as well as an increase in temperature and pressure [51]. This leads to an increase in the overall extraction yield [89]. Among the advantages of UAE are that it is considered one of the most cost-effective techniques for the extraction of polysaccharides [51], apart from being efficient, fast and environmentally friendly.

UAE has been used to extract pectic polysaccharides from the peel of fruit and vegetables, such as eggplant [35], pomegranate [52], tomato [53], custard apple [54] and mango [55]. UAE of tomato peel was able to efficiently extract two valuable bioactive ingredients (pectin and polyphenols) simultaneously, in addition to shortening the extraction time with respect to conventional extraction techniques [36]. It has also been reported that high yields of hemicellulose polysaccharides can be obtained with short extraction times, especially xyloglycans by UAE in grape pomace [56]. UAE turns out to be efficient for extracting prebiotic sugars from industrial artichoke residues: 1-kestose, nystose, fructofuranosylnystose and raffinose were successfully extracted, obtaining an extract of approximately 9.6 mg of prebiotic saccharides/g of dry raw material [57].

MAE involves the penetration of electromagnetic radiation into a solid matrix. The heating generated is due to the molecular friction caused by the ionic conduction of the dissolved ions and the rotation of the dipoles of the polar solvent, which favor the extraction of the bioactive compounds. Both the heating produced and the internal pressure originated cause rupture of the cell; as a consequence, the structure is altered, which facilitates the release into the solvent of the bioactive compounds, improving the transfer coefficient [60][61][62]. MAE is a promising technique for polysaccharide extraction; it has advantages such as high yields, less solvent used, shorter extraction times and being environmentally friendly [63][64][65].

Currently, MAE is widely used in the extraction of polysaccharides from various sources of by-products, such as banana peel [66], grapefruit peel [67], broccoli by-products [68] and cocoa shell [69], among others. [70] reported higher yields (18.73%) and higher purity of pectic polysaccharides extracted from fruit peels, such as dragon fruit and passion fruit, compared to conventional methods. In addition, extraction times were shortened, resulting in a reduction in energy consumption. High yields of hemicellulose polysaccharides such as xylan, glucuronoxylan and xyloxylan extracted by MAE from tobacco plant residues have also been reported [71].

While the UAE and MAE methods break the cell wall, EAE degrades the cell parts by enzymatic hydrolysis, which causes an improvement in the yield and biological activity of polysaccharides [72][90]. The method is selective for the extracted bioactive compounds and environmentally friendly [58]. The extraction yield depends on several factors, such as the liquid–solid ratio, pH, amount of enzyme, temperature and extraction time [74][91].

EAE has been used effectively for extraction of pectin from kiwifruit pomace, demonstrating a higher pectin yield by enzymatic extraction with Celluclast (cellulases, polygalacturonase, pectin lyase and rhamnogalacturonan lyase) than by acid extraction with citric acid [75]. [76] also reported higher extraction yields for pectin (15.3%) from apple pomace by EAE compared to acid extraction with sulfuric acid. EAE achieved higher polysaccharide extraction yields from pomegranate peel than those obtained with HWE and UAE, the extracted polysaccharides having strong antioxidant properties [77]. Extraction of polysaccharides fromDendrobium chrysotoxumby EAE provided 1.25-fold higher yields than with HWE [78].

SFE is an emerging technology in the extraction of bioactive compounds that allows the natural qualities of the compounds to be preserved and ensures food safety [92]. The critical point of CO2(31 °C and 7.38 MPa) allows the recovery of bioactive compounds with a high degree of purity and especially useful clean extracts for functional foods [79]. SFE technology has, to a large extent, been used for apolar substances, although selective extraction of polar compounds is possible by using modifications. [80] reported a yield of 18.59% for SFE of polysaccharides fromArtemisia sphaerocephalaplants with extraction parameters of 45 MPa at 45 °C, with a CO2flow of 20 L/h for 2 h. Rivas et al.

New polysaccharide extraction methods are being developed, such as deep extraction with eutectic solvents [82], accelerated extraction with solvents [83][86] and high-pressure dynamic microfluidization [84]. In addition, the previously mentioned methods are also combined to improve the yields and bioactive functions of polysaccharides.

The polysaccharides, mannose, rhamnose, glucose, galactose, xylose, arabinose, fucose, glucuronic acid and galacturonic acid, were extracted from pineapple pomace by combining UAE and EAE to provide ultrasonic-cellulase synergistic extraction; the extracted polysaccharides demonstrated the ability to inhibit development of HepG2 cells resistant to insulin and therefore can be considered potential ingredients to develop a new beneficial food [85]. In addition, Liew et al. [86] reported a 1.5 to 3.5 times higher yield for the extraction of pectic polysaccharides combining UAE and EAE compared to separate extraction methods.

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