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Review

Potential of Deep Eutectic Solvents in the Extraction of Organic Compounds from Food Industry By-Products and Agro-Industrial Waste

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Abstract: Global food waste has a huge impact on the environment, as it is a source of greenhouse gas emissions and wasted natural resources. Across the world, over 30% of food is lost or wasted each year. Aside from this, the food industry, as well, is one of the biggest sources of agro-industrial waste and by-products, which can be valorized and used for different purposes. Such waste is a good source of bioactive organic compounds that can be extracted without altering their properties, where deep eutectic solvents can serve as green solvents and as an excellent replacement for volatile organic solvents. Isolated compounds can be used in innovative food production, chemical production, cosmetics and other industries. Deep eutectic solvents have attracted extraordinary attention due to their advantages such as environmental friendliness, availability and easy preparation, easy handling and utilization of non-toxic components for their formation. Due to these properties, they are a greener alternative to classic organic solvents for many processes, including extractions. In this paper, we review the utilization of deep eutectic solvents as potential green media for the extraction of organic compounds such as polyphenols, carbohydrates, proteins and alkaloids from by-products of the food industry and from agro-industrial waste.

Keywords: deep eutectic solvents; bioactive organic compounds; food industry by-products; greener solvents; agro-industrial waste

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1. Introduction

According to research conducted within the FUSIONS4 project [1] of the European Commission, about 88 million tons of food are thrown away in the EU member states annually. In EU member states, the amount of discarded food (edible and inedible) amounts to 173 kg per inhabitant. The total amount of food produced in the EU in 2011 was approximately 865 kg/inhabitant, which would mean that 20% of the total produced food was wasted. Furthermore, the food industry generates huge amounts of waste and by-products, which are usually discarded, creating a huge load in the environment. Those by-products are usually rich in different beneficial compounds that can be isolated and used for different purposes [2,3].

Waste management is often a complex and expensive process, and the conversion of by-products into valuable bioactive compounds should be addressed as often as possible. Valorization can reduce or minimize the negative environmental impact and increase sustainable growth by reusing value-added compounds from natural resources. The aim of this paper is to provide an overview of new trends in the problem-solving skills used to address food waste manufacturing and agro-waste obtained from the processing of mostly plant raw materials. This is an area of research of exceptional importance since by-products such as bark, seeds and pulp are good sources of various bioactive organic compounds that can be used as functional ingredients or nutraceuticals in food production, cosmetics or in the pharmaceutical industry. Bioactive substances that are most often isolated from by-products are polyphenols, proteins, organic acids, alkaloids, terpenoids such as carotenoids, sugars, lipids, etc. Many of these compounds possess multiple health benefits such as antitumor [4,5], antioxidant [6–8], antihypertensive [9,10], antimicrobial [11], hypoglycemic [12], antiviral [13,14] and other effects.

Conventional organic solvents such as methanol, acetone, ethyl acetate, benzene, chloroform, dichloromethane, toluene, petroleum ether and hexane are commonly used in the extraction of the above-mentioned compounds from different byproducts. These solvents often show high vapor pressure and are usually harmful and flammable, as well as not environmentally friendly. Capello et al. [15] proposed a substitution of such solvents with green solvents, describing the characteristics that green solvents should possess. The first is to replace hazardous organic solvents with less hazardous ones that have less impact on the environment, human health and safety and possess greater biodegradability or a reduced ozone depletion potential. Another is to use biosolvents like ethanol, produced from renewable sources. The third is the use of supercritical fluids, which are harmless to the environment, and the fourth is the use of ionic liquids with a low vapor pressure, and thus reduce emissions to the air. Ionic liquids, solvents formed from organic cations and anions, do possess some advantages over conventional VOSs (volatile organic solvents), such as high thermal stability and non-volatility, but many of them have shown toxic effects toward different organisms [16]. Very similar, but yet structurally different solvents have arisen in the last decade, showing some advantages over ILs, along with great potential for different processes, called deep eutectic solvents (DESs). DESs have attracted extraordinary attention due to their advantages such as environmental friendliness, good electrical conductivity, stability, low cost and easy preparation. They were first mentioned by Abbott et al. in 2003 [17], and numerous scientific reports have been published to date [18–22]. Many DESs have been used for the extraction of various value-added bioactive compounds because of their efficient potential in green technologies and biodegradable properties [23].

2. Deep Eutectic Solvents

Since their first introduction in the literature in 2003 by Abbot et al. [17], deep eutectic solvents have become the subject of many studies. Abbot et al. (2003) [17] were investigating choline chloride and urea mixtures when they noticed a large melting point depression of the mixtures compared to the melting points of the pure components. Due to the large melting point depression, such solvents were designated as "deep" eutectic solvents. Later, some authors suggested that a different definition of deep eutectic solvents is needed, and that their melting point should not be compared to those of the pure components, but to one of their ideal liquid mixture [24]. Therefore, Martins et al. designated them as: "*a mixture of two or more pure compounds for which the eutectic point temperature is below that of an ideal liquid mixture, presenting significant negative deviations from ideality*" [24]. Due to their interesting properties, DESs have found applications in many scientific areas. They have been extensively used in the extraction of bioactive components from different materials [3,25–30], many synthetic procedures [31–39], the stabilization of bioactive compounds [40–44], the enhancement of drug delivery [45–47] and the pretreatment of food industry waste [48–50].

Hansen et al. [18] classified DESs into 5 types: type 1 is formed by the combination of quaternary ammonium salt (QAS) and metal chloride; type 2, of QAS and metal chloride hydrate; type 3, of QAS and HBD (organic acids, amides or polyoles); type 4, of metal salt and HBDs; and type 5 consists of a new class of nonionic HBAs and HBDs. Furthermore, some authors classify them according to the nature of their components as NADES, which are formed of purely natural components, THEDES, the ones formed of pharmaceutically active components, TDES, the ones formed from three components, etc. Either way, DESs can be prepared by heating the mixture of desired components until the formation of clear liquid, evaporation of water from the solution of desired components, by freeze-drying or simply by grinding the components [18]. The physical and chemical properties of DESs are tunable and can be adjusted for specific uses, since different components can be mixed and combined in different ratios [19]. The net of hydrogen bonding, van der Waals interactions or ionic bonding between components causes a charge delocalization, thus lowering the lattice energy of the system and consequently decreasing the melting point [18,19,51].

2.1. Influence of the Nature of the Hydrogen Bond Donor and Hydrogen Bond Acceptor and Their Molar Ratios on Physicochemical Properties

Both the nature of the HBD and HBA and their molar ratio show a huge impact on the physicochemical properties of the DESs. This was shown by Mero et al. (2023), who investigated choline chloride and betaine-based DESs. They found that DESs' densities and viscosities were influenced by both the HBD and HBA, where glycerol as the HBD caused high viscosity and density in the DES, while ethylene glycol-based DESs showed the lowest values. Considering HBAs, when betaine was used instead of choline chloride, a higher viscosity and density were achieved. The influence of the ratio was evident when the HBD content was increased, causing a lower viscosity. When choline chlorine was used as the HBA, the refractive index was higher [52]. The strength of hydrogen bonding has a great influence on the thermal stability of the DESs, so thermal degradation at higher temperatures happens through the weakening and breaking of the hydrogen bonds between the HBAs and HBDs [53].

Although DESs show many promising properties, their high viscosity and density are one of their major drawbacks, especially if the process is scaled up. These are governed by the strength of intermolecular bonding between DES components [54,55] and can be reduced to some extent by a temperature increase or the addition of water. On the other hand, the addition of high amounts of water can weaken the hydrogen bonding between HBAs and HBDs, causing the loss of eutectic properties [56]. Many authors designate DESs as desirable, compared to volatile organic solvents, as they show low vapor pressure, can be made of natural, non-toxic and biodegradable compounds and are easy to handle [40,51,57]. They have found a wide range of applications since, are considered to adhere to the green chemistry principles and are thus designated as green solvents.

2.2. Toxicity Consideration

Many studies so far have dealt with the applications of DESs in different areas and the investigation of their physicochemical properties, but not so many with the assessment of their toxicity [58–61].

Many authors point out their non-toxic nature, but still, it is necessary to further investigate their impact on our ecosystem. As DESs can be formed of many different components, they can be tailored to suit specific applications [19], but if they are to be used in the food, cosmetic or pharmaceutical industries, it is very important to assess their toxicity profile. DESs are mostly presented as green and safe solvents because they are often synthesized from starting materials that are mostly non-toxic and non-carcinogenic, but of course, there are many exceptions. Furthermore, the synergistic effect of the components can change the toxicological profile of the DES compared to the pure components [62,63]. It can also vary according to their concentration. Certain quaternary ammonium compounds, often used as HBAs, have been shown to exhibit a larger toxicity compared to other components [63].

The toxicity of DESs is a complex topic. Some studies were conducted to determine the impact of DESs on the environment and living organisms. However, it is definitely necessary to investigate the ecotoxicological profile, which would provide more information about the biocompatibility, toxicity and degradability of DESs. This is why scientific papers that have provided toxicity profiles of deep eutectic solvents are extremely important [64]. Since it is often accentuated that DESs could be recycled and reused, their toxicity after recycling should also be investigated, since elevated temperature and pressure and microwave and ultrasonic radiation can change their physicochemical properties as well as generate harmful by-products. Since many scientist have rethought the safety of DESs, research that is more recent is focused on ecotoxicological assessments. Viera Sanchez et al.

(2022) published one such study [65] in which the ecotoxicological profiles of 15 NADESs were examined. The results show that the tested NADESs were non-toxic to *D. magna*, *F. enigmaticus* and *A. fischeri*. However, the authors state that in order to classify these solvents as "green", "clean" or ecologically benign", it is necessary to perform additional ecotoxicological biological tests.

2.3. Solubilization Ability of DESs

The physicochemical properties of DESs are the main factor influencing the application of DESs in the extraction of different components. As described above, the chemical nature and ratio of the HBD and HBA determine the final properties of the DES. Therefore, it is important to understand the solubility mechanism of substances in the presence of eutectic solvents or some multicomponent mixtures. Only a few papers clarify the mechanism of solute solubility in DES extracts. One such study [66] was conducted describing the specific interactions responsible for curcumin solubility. They investigated the solvation mechanism in ethanol/NADES binary solutions, using COSMO-RS and NOESY NMR. The research results showed that NADESs could increase the solubility of curcumin in ethanol solutions. Increasing the QA content increases the solubility of curcumin, while the addition of organic acids revealed that levulinic acid NADESs was the best in curcumin extraction [66]. Many studies have shown that DESs are more effective in many extraction procedures compared to classic organic solvents [67–72]. It was also shown that plant extracts obtained using DESs showed higher antioxidant activity in contrast to organic extracts [73]. In addition to a higher extraction efficiency, DESs have also shown a higher selectivity than that of organic solvents. Among other properties, the polarity of DESs is also very dependent on the nature of the HBA and HBD [74], thus influencing the type of extracted compounds.

2.4. Recovery of Extracts from DESs

Recent studies have presented solutions to the significant problem of the recovery of dissolved substances after the extraction and recycling via DESs [41,75]. Due to the low vapor pressure of DESs, their separation via distillation is very limited. According to the literature, several methods for recovery are offered. The most common method is the utilization of macroporous resins for extract recovery, but other methods, such as solid phase extraction (SPE), the antisolvent method and back extraction, can also be used. SPE was shown to be very effective with analyte recovery, above 90% [76].

3. Extractions of Organic Compounds from Fruit By-Products

3.1. Bioactive Compounds in Fruit Waste

During the production process as well as after food consumption, a large amount of waste biomass is created. This can be mainly attributed to fruit waste, which comprises most of the solid municipal waste (SMW). Rapid economic growth causes greater consumption of different varieties of fruit produced throughout the world. Due to the large-scale industrial processing of fruit, fruit waste such as peel, seeds and other inedible parts of fruit accumulates. The main causes of fruit waste generation are fruit injuries obtained during food transport and storage. Fruits can be damaged at all stages of handling, during collection, transport, receiving, packaging, storage, as well as during preparation for consumption [77]. For that reason, it should be protected from external factors such as shocks, which cause mechanical damage, as well as internal ones, which can cause water loss. Two fruit waste disposal techniques are landfills and incineration. Inappropriate procedures with disposed waste can result in the emission of methane and CO_2, while burning can cause the formation of carcinogenic and toxic organic compounds such as dioxins, furans and acid gases [78]. Many strategies have been developed in recent years for food waste management. Fruit waste is mainly used as animal feed or fertilizer [79].

Fruit waste is a rich source of extremely useful compounds, such as various secondary metabolites, which could be used in the preparation of potential food additives, preservatives, nutraceuticals and other functional foods [80]. It contains bioactive phytonutrients that have potential pharmacological properties and show positive effects on human health. Antioxidant activity stands out as one of the most researched features in the science community. Antioxidants reduce or prevent the formation of reactive oxygen species (ROS) and reactive nitrogen species (RNS) in numerous human, animal and in vitro studies. Antioxidants prevent adverse alterations of lipids, proteins and DNA. Therefore, the application of exogenous antioxidants can help in dealing with oxidative stress [81–84].

The production of citrus fruits, such as orange, mandarin, lemon, lime, grapefruit, pomelo and citron, all belonging to the Rutaceae family, yields up to 15 million tons of by-products per year [85]. The by-products of fruit processing are usually seeds, peel, pulp and leaves. It has been proven that they are an extremely useful source of bioactive organic compounds such as polyphenols, enzymes, monosaccharides (glucose and fructose), disaccharides (sucrose, polysaccharides (cellulose, starch, pectin, dietary fibers)), dicarboxylic acids (citric, malic, oxalic acids), essential oils (limonene), pigments (carotene, lutein), lipids (linolenic, oleic, palmitic, stearic acids), vitamins (Vitamin B complexes, Vitamin C) and others [86]. Therefore, one of the main goals of the food industry is to reuse and recycle fruit by-products [87].

Apple is the basis of fruit production. Because of the natural apple juice production, usually performed by pressing and squeezing the fruit, apple pomace is generated as the major by-product. Pomace makes up 25–30% of the apple, depending on the variety and the efficiency of processing. Apple pomace itself consists of seeds, seedpods and skin. It is a high-quality source of carbohydrates, pectin, fiber and minerals. It contains polyphenols: phlorizin, catechin, epicatechin, procyanidin B2, hyperin, quercetin and others. The most important apple minerals are Ca, Na, K, P and Mg, which play key roles in processes important for human health. This makes it even more important to develop new methods for the extraction of bioactive compounds from apple by-products and convert them into value-added products with different functionalities. It was proven that apple pomace extracts possess anticancer, cardioprotective and antimicrobial biological activities [88–91].

Grapevine (*Vitis vinifera*) is another important crop cultivated throughout the world. It is estimated that 80% of the annual production of grapes is processed into wine, with 20–30% of the mass of used grapes (pulp, skin, stems, seeds) remaining as solid biowaste. Grape pomace, one of the main by-products of the grape wine industry, is used as fertilizer in vineyards, and is burned or dumped unplanned in fields, which can be a major environmental problem. It is a rich source of flavanols, phenolic acids and flavonols, and its extracts usually show an enhanced antioxidant capacity [92]. In addition to the antioxidant capacity, they provide various other health benefits such as anticarcinogenic, anti-inflammatory, antimicrobial, cardioprotective and neuroprotective effects [93].

Berries include chokeberry, raspberries, blackberries, blueberries, currants, cranberries and other berries. Some of them are used to manufacture juice and wine and their processing often results in the generation of pomace by-product. This by-product is often a valuable nutritionally rich source of bioactive organic compounds and dietary fiber that can contribute to the technological and nutritional properties of food [94].

3.2. Phenolic Compounds

Polyphenols, a large group of secondary metabolites with antioxidant properties [95–97], belong to the most important natural antioxidants with numerous health benefits related to cardiovascular disease and various cancer types [98–100]. Newer techniques, including the application of DESs, can improve the existing extraction of polyphenols and shorten the extraction time, reduce solvent consumption and reduce process costs. Therefore, techniques such as microwave [101], ultrasound [102,103], pulsed electric field (PEF) [104], and high-voltage electric discharge (HVED) [105] techniques can improve the extraction efficiency of polyphenols, especially if combined with DESs.

Mango peels, a by-product of mango processing, are rich in antioxidants. Microwave-assisted extraction (MAE) in combination with a DES (lactic acid:sodium acetate:water) was shown to be very efficient in the extraction of phenolic compounds from ripe mango *Mangifera indica* (L.) peels. Phenolic compounds were extracted in 56.17 mg GAE/g dry weight yield [106].

El Kantar et al. showed that DESs and glycerol possess a high extraction efficiency for polyphenols from grapefruit peel. These solvents were a good green alternative to conventional solvents obtaining a higher extraction yield of the flavonoid naringin from grapefruit peels [107].

Natural deep eutectic solvents were very efficient in the extraction of D-limonene, polyphenols and proteins from orange peels [108]. The results of one study showed that DESs combined with ultrasound technology are excellent tools for the extraction of anthocynanins and polyphenols from blueberry pomace. Better yields were obtained with ChCl:butane-1,4-diol (molar ratio 1:3) DES at 63 °C compared to those with 70% ethanol [94].

3.3. Carbohydrates

NADESs could be promising co-solvents in the extraction of various biomaterials, such as polysaccharides. Polysaccharides derived from agro-industrial waste include cellulose, hemicellulose, pectin, starch, carrageenan, fucoidan, chitosan and alginate. Monomeric units in the mentioned polymers are bonded by α-1,4-glycosidic bonds, β-1,4-glycosidic bonds and α-1,6-glycosidic bonds, and intermolecular hydrogen bonds and van der Waals forces [109]. Pectins are water-soluble carbohydrates that are found in cell walls and are built from the majority monomer D-galacturonic acid, whose units are connected by an α-(1,4)-glycosidic bond. Pectin-rich extracts are traditionally used in the food industry as an additive for gelling, thickening, swelling and hydration capacity [79]. Lemon peel is very rich in pectins [110,111], as well as apple pomace [112], mango peel [113], pomegranate peel [114], kiwifruit pomace [115], papaya peel [116], passion fruit peel [117], jackfruit peel [118], sour orange peel [119] and gape pomace [120]. Cellulose is a linear polysaccharide polymer consisting of D-glucose monosaccharide units linked by β-1,4-glycosidic bonds. Chemical reactions on the hydroxyl groups of cellulose are difficult, and the reason for this is intramolecular and intermolecular hydrogen bonds, which make it impossible to dissolve cellulose in water and most organic solvents [79]. High cellulose content is present in corn husks and grape stalks [121], pomegranate peels, strawberry pomace [122], chestnut peels [123], bananas [124] and orange peels [125]. Cellulose as a versatile biopolymer can be used for a variety of food and non-food applications, as well as in pharmaceutical, paper, textile, wood, and industrial chemistry. It is also useful for the generation of micro- and nanocellulose, which are important for polymer composite materials [126].

Different carbohydrate polymers have shown anti-cancer, antioxidant and hypoglycemic effects, which allow them to be used in medicine, chemical production and the growth of substances [127–130].

In various studies, ChCl-based eutectic solvents were used for the extraction of pectin from food industry by-products. In one study, pectin was extracted from pomelo (*Citrus grandis* (L.) peels using NADESs in combination with sonication, where choline chloride:malonic acid and choline chloride:glucose:water were the most efficient. Pectin was obtained in both NADEs in 94% yield after 60 min of sonication, at 80 °C, pH < 3.0, and a NADES:water ratio of 1:4.5 (v/v) [131]. Another study showed that treatment of apple pomace with eutectic solvents, such as ChCl:lactic acid and ChCl:urea improved the efficiency of pectin extraction. However, some solvents, like ChCl:oxalic acid, were not suitable for pectin extraction; since carbohydrate degradation occurred, a lower yield was obtained [132].

3.4. Proteins

The data on the presence and extraction of amino acids and proteins from food industry by-products are very limited. The protein content in pomegranate peel was reported to be about 3% [133]. A higher content was found in pomegranate seeds (14%) [134]. Some authors report different amino acid content in food by-products: citrus peel is a rich source of alanine, asparagine, aspartate, alanine, arginine, proline and serine [135]. Authors [105] used high-voltage electrical discharge (HVED) in combination with a DES, which was proven to be very effective for the extraction of proteins and polyphenols from pomegranate seeds. The method includes pre-treatment of pomegranate seeds with HVED, followed by solid-liquid extraction (SLE) in eutectic solvents. Protein extraction was further improved, especially in the eutectic solvent ChCl:Glc, in which a high protein content of 94% was achieved, so the authors concluded that DESs improve the diffusivity of proteins and polyphenols [105].

3.5. Other Compounds

Fruit peel is a rich source of terpenes. Terpenes can be classified as hemiterpenes (1 isoprene unit), monoterpenes (2 units), sesquiterpenes (3 units), diterpenes (4 units), sesterterpenes (5 units), triterpenes (6 units), and tetraterpenes (8 units). In orange peel D-limonen, linalool and myrcene were identified; in tomato peel, lycopene; and in melon peel, carotenoids such as β-carotene, zeaxantin, violaxantin and lutein.

The distillation of aromatic plants yielded by-products in which monoterpenes like carvacrol, thymol and *p*-cymene were found. The mentioned compounds are the active ingredients of aromas and perfumes and are often used in aromatherapy [77].

Due to their hydrophobic nature and harmless and renewable character, terpenes and terpenoids have been used in the design of eutectic solvents. Due to their environmentally friendly properties, they are proposed as possible replacements for conventional organic solvents in various extraction procedures [136].

Eutectic solvents were proven very effective in the extraction of Vitamin E from red palm biodiesel. Manurung et al. (2018) achieved a high extraction efficiency of α-, γ- and δ-tocotrienol from biodiesel using a DES of potassium carbonate:glycerol in different molar ratios, where the most effective molar ratio of potassium carbonate:glycerol was 1:6. [137].

Deep eutectic solvents in combination with the ultrasonic method were used for the delignification of watermelon rind, whereby a maximum lignin removal of 44% was achieved in a sonication time of 40 min at 120 °C [138] Some examples of recent applications of DESs and assisted techniques for extracting bioactive compounds from fruit by-products is given in the Table 1.

Table 1. Extraction of bioactive compounds from fruit waste using various DESs.

Fruits	Waste	Extraction Technique/Conditions	DESs	Bioactive Compounds	Reference
Polyphenols					
Orange	peel	Solid-liquid extraction (SLE)	LA:glucose (Glc) (5:1), L-proline:malic acid (1:1)	Polyphenols, flavonoids	[139]
		SLE, 50 °C, 30 min	ChCl:citric acid (CA) (1:1)	Hesperidin	[140]
		SLE, 45 ± 5 °C, 20 min	ChCl:malic acid ChCl:glycerol	Flavonoids	[139]
Lemon	peel	RP-LC–QTOF-MS/MS	ChCl:glycerol (1:3)	Quercetin, *p*-coumaric acid	[141]
Tangerine	peel	USE (20 W, 35 kHz)	ChCl:levulinic acid:*N*-methyl urea (1:1.2:0.8)	Polymethoxylated flavonoids and their glycosides	[142]
Grapefruit	peel	HVED SLE, 50 °C, 60 min solid/liquid ratio (1:10)	LA:Glc (5:1)	Polyphenols, naringin	[107]
Mango	peel	MAE	Sodium acetate:LA (1:3)	Mangiferin	[106]
Apple	pomace	USE, 83.2 W	ChCl:glycerol (1:2)	Quercetin, chlorogenic acid, gallic acid, phloretin, phloridazin, rutin	[89]
		SLE, 60 °C, 6 h	ChCl:ethylene glycol (1:4)	Procyanidin, chlorogenic acid, epicatehin hydrate, vanillin, phloridzin	[88]
Grape	pomace	MWE, 300W + USE, 50 W	ChCl:CA (1:2)	Anthocyanins, gallic acid, catehin and quercetin 3-*O*-glucoside	[143]
				Anthocyanins (malvidin-3-*O*-monoglucosid)	[144]
		SLE, rt, 24 h	Betain:CA (1:1)	Malvidin	[145]
		USE, 100 W	Betain:Glc (1:1)	Flavan-3-ols	[146]
		SLE, HPLE	ChCl:ethylene glycol:water, ChCl:glycerol:levulinic acid:water, ethylene glycol:water, glycerol:water	Phenolic acids, flavanols, flavonols	[92]
	skin	USE, 59 kHz	ChCl:CA (1:2)	Flavan-3-ols, catechin, epicatechin, protocatechuic acid	[147]

Table 1. *Cont.*

Fruits	Waste	Extraction Technique/Conditions	DESs	Bioactive Compounds	Reference
Mangosteen	peel	USE	ChCl:LA (1:2)	Anthocyanins	[148]
Pomegranate	peel	SLE, 50 °C USE, 50 °C, 50 W	LA:ChCl (3:1), malic acid:sucrose (1:1), glycerol:glycine (3:1), ChCl:fructose (1.9:1), Glc:tartaric acid (1:1), glycerol:urea (1:1), malic acid:Glc:glycerol (1:1:1), LA:glycine (3:1)	Caffeic acid, kaempferol, luteolin, protocatechuic acid, ellagic acid, chlorogenic acid, hydroxybenzoic acid, gallic acid, quercetin	[149]
		SLE	ChCl:glycerol (1:11)	Polyphenols, flavonoids	[150]
		USE TPC (29.30% water; liquid:solid 53.50 mL/g; 238.20 W; 29.50 min), PC (25.65% water; liquid:solid 44.20 mL/g; 120 W; 20 min), and EC (33.13% water; liquid:solid 60 mL/g; 300 W; 20 min)		Total polyphenol content (TPC) punicalagin content (PC), ellagic acid content (EC)	[151]
		pretreatment with HVED or US, SLE	ChCl:CA, ChCl:acetic acid, ChCl:LA, ChCl:glycerol, ChCl:Glc	Polyphenols	[105]
Jabuticaba	pomace	USE	ChCl:propyleneglycol (1:2), ChCl:CA (1:1), ChCl:malic acid (1:1), CA:Glc:water (1:1:3), CA:propylene glycol (1:1), betaine:CA (3:1)	Anthocyanins	[152]
Blueberry	pomace	USE	ChCl:LA (1:1)	Anthocyanins	[153]
		USE	ChCl:oxalic acid (1:1)	Anthocyanins	[154]
		USE	ChCl:butane-1,4-diol	Cyanidin-3-*O*-rutinoside	[155]
Cranberry	pomace	USE	ChCl:betaine hydrochloride:levulinic acid (1:1:2)	Procyanidins, anthocyanins	[156]
Strawberry and raspberry waste	extrudate	SLE	ChCl:glycolic acid:oxalic acid (1:1.7:0.3)	Anthocyanins	[157]

Table 1. *Cont.*

Fruits	Waste	Extraction Technique/Conditions	DESs	Bioactive Compounds	Reference
Black chokeberry	pulp	(UMAE-EtOH) Solid:liquid 1:15, 230 W, 52 °C, 367 s	ChCl:CA (1:1), ChCl: malic acid (1:1), ChCl:LA (1:1), ChCl: Glc (1:1), ChCl: sucrose (1:1), ChCl: glycerol (1:2), ChCl:CA:Glc (1:1:1), ChCl:CA:glycerol (1:1:1)	Cyanidin-3-*O*-galactoside, cyanidin-3-*O*-glucoside, cyanidin-3-*O*-arabinoside, cyanidin-3-*O*-xyloside, cyanidin-3,5-*O*-dihexoside, dimer of cyanidin-hexoside	[158]
Sour cherry	pomace	USE, 40 °C, 30 min MWE, 90 W three successive cycles of 5 s (15 s of total time)	ChCl:malic acid	Cyanidin-3-*O*-sophoroside, cyanidin 3-*O*-glucosylrutinoside, cyanidin-3-*O*-rutinoside, quercetin-3-*O*-glucoside, quercetin-3-*O*-rutinoside, quercetin-*O*-glycoside, isorhamnetin-3-*O*-rutinoside	[159]
Carbohydrates					
Pomelo	peel	USE, 80 °C, 60 min, liquid:solid ratio (40:1)	ChCl:malic acid, ChCl:Glc:water	Pectin	[131]
Apple	pomace		ChCl:LA, ChCl:oxalic acid, ChCl:urea (1:2)	Pectin	[132]
Banana	puree	MWE, 25 °C, 30 min, 30% water	Malic acid:β-alanine:water (1:1:3)	Soluble sugars	[160]
Grape	seed	USE, 30 °C, 10 min,	Dodecanoic acid:octanoic acid (1:1)	Gsps	[161]
Proteins					
Pomegranate	seed	Pressurized liquid extraction (PLE) and DES extraction	ChCl:CA, ChCl:AA, ChCl:LA, ChCl:glycerol, ChCl:Glc	Protein	[105]
Orange	peel	4 °C, 15 min	ChCl-based NADES with ethylene glycol	Protein	[108]
Other compounds					
Watermelon	rind	USE	ChCl:LA	Lignin	[138]

4. Extraction from Vegetable By-Products

Polyphenols

Onion, as one of the most easily cultured vegetables around the world, is a source of huge amounts of by-products, especially in the form of onion peel. It can be used as a source of valuable polypehnols, like quercetin, kaempferol, myricetin as well as other polyphenols [30]. In order to apply a green extraction of such compounds, Pal and Jadeja (2019) used DESs for polyphenol extraction and compared it to conventional methods, such as Soxhlet (with methanol) and microwave extraction (with distilled water). They found that a choline chloride:urea:water DES can be effectively applied for the above-mentioned extraction as a green solvent [30]. The use of NADESs in combination with ultrasound technology also proved the efficacy of these green types of solvents in the extraction of quercetin from onion peels [162]. The same research proved the efficacy of NADESs in the extraction of bioactive polyphenols from broccoli [162], while Cao et al. (2023) also effectively combined the use of NADESs and ultrasound technology to extract neochlorogenic, ferulic, erucic, quinic, chlorogenic and caffeic acid from broccoli leaves. Their extracts also showed greater TPC, antioxidant and antimicrobial activity than the ones obtained using other extraction methods [163].

Curcuminoids, *Curcuma longa* yellow pigments, as biologically active compounds with various pharmacological properties, have a high potential for pharmaceutical, food or cosmetic applications. They can be extracted from *C. longa* rhizomes using NADESs, in higher yields than using conventional solvents [164]. Another group of colorants, betalains, was extracted from beetroot waste, using a NADES as both the extracting and stabilizing solvent as well. The results for NADES extraction were comparable to those of conventional water extraction, while the stabilization of betalaines was much higher than that with water extracts [165]. Carotenoids are an important group of pigments, with significant biological activity. Tomatoes' most significant carotenoid is lycopene, and tomato waste generated during the production of tomato sauce, juice or paste, is rich in this health-promoting compound. Menthol:hexanoic acid (2:1) DES was found to be very effective in lycopene extraction compared to conventional solvents [166].

Potato peel is another example of the usual waste containing high amounts of bioactive compounds, like quercetin, chlorogenic acid, caffeic acid, α-solanine and α-chaconine [167]. Polyphenol and bioactive component content varies depending on variety and location, but it can go up to 20 mg/g of the peel [168]. An excellent source of polyphenols is black carrot waste as well. This vegetable is very popular in Turkey, where large amounts of waste are generated due to the production of a beverage called Shalgam juice. This waste, in form of fermented black carrot, is rich in polyphenols, which can be effectively extracted using NADESs. According to Toprak and Unlu, ChCl:glycerol was very efficient in TPC extraction, while ChCl:fructose:water and ChCl:sucrose:water were very efficient in total flavonoid and total monomeric anthocyanin extraction [169].

Farajzadeh et al. (2018) found a new application of DESs when they investigated the extraction of pesticides from different fruit juices and vegetables, finding that this approach is effective for the fast and green analysis of these analytes [170].

Many authors have also investigated the removal of lignin from different agro-industrial wastes to facilitate cellulose hydrolysis using green methodologies. Ternary DES (TDES) were successfully utilized in this manner, both for the pretreatment of onion roots and garlic skin, which increased the cellulose content in both cases [171]. Some examples of recent applications of DESs and assisted techniques for extracting bioactive compounds from vegetables by-products is given in the Table 2.

Table 2. Extraction of bioactive compounds from vegetable waste using various DESs.

Vegetable	Waste	Extraction Technique	DESs	Bioactive Compounds	Reference
Polyphenols					
Onion	peel	60 °C, 120 min, 20:1 (liquid:solid)	ChCl:U:H_2O (1:2:4)	TPC, quercetin, kaempferol, myricetin	[30]
Black carrot	waste	Ultrasonic bath, 50 °C, 30 min, 37 kHz, 140 W	ChCl:glycerol (1:2)	Polyphenols	[169]
Onion		20 °C, 35 min, 400 µL of DES	Methanol solution of betaine:D-mannitol	Quercetin, isorhamnetin, kaempferol	[162]
Curcuma longa	rhizomes	50 °C, 0.1:10 g/mL (solid:liquid), 30 min	Citric acid:glucose (1:1), 15% water	Pigments: curcuminoids	[164]
Broccoli		20 °C, 35 min, 400 µL of DES	Methanol solution of betaine:D-mannitol	Quercetin, isorhamnetin, kaempferol	[162]
Broccoli	leaves	Solvent:solid 36.35 mL/g, 49.5 °C, 31.4 min, ultrasonic power 383 W	ChCl:1,2-propylene glycol (1:2)	Neochlorogenic acid, ferulic acid, erucic acid, quinic acid, chlorogenic acid, caffeic acid	[163]
Tomato	by-products	Solvent:solid 25:1, 90 min, 50 °C	Menthol:hexanoic acid (2:1)	Carotenoids	[166]
Other					
Beet, cucumber, potato, and tomato		70 °C, 5 min, 142 µL DES, 1610× *g* 5 min centrifugation	ChCl:*p*-chlorophenol	Pesticides: diazinon, metalaxyl, bromopropylate, oxadiazon, fenazaquin	[170]
Garlic	skin	Ultrasound 20 + 28 + 40 kHz, 30 min, r.t., followed by microwave 20 min, 80 °C	ChCl:glycerin:$AlCl_3 \cdot 6H_2O$ (1:2:0.2)	Removal of lignin	[171]
Beetroot	peel and pulp	1:30 g/mL (solid:liquid), 25 °C, ultrasonic bath, 3h, agitation 900 s	$MgCl_2x6H_2O$:urea (2:1)	Betalains	[165]
Onion	root	Ultrasound 20 + 28 + 40 kHz, 30 min, r.t., followed by microwave 20 min, 80 °C	ChCl:glycerin:$AlCl_3 \cdot 6H_2O$ (1:2:0.2)	Removal of lignin	[171]

5. Extraction from Oilseed Agro-Waste

Oilseeds crops are one of the major high-value agricultural commodities. Oils are organic compounds with a major role in the construction of living beings. According to their chemical composition, they are esters of glycerol and higher fatty acids, so they are classified as triglycerides, and they also belong to a wider group of compounds called lipids. For their production, oilseeds must be properly prepared before the refining process. Processes include seed cleaning, mechanical beating, solvent extraction, degumming, neutralization, bleaching and deodorization. Although the mentioned processes yield high-quality oil, large amounts of by-products are also produced. The disposal of these by-products into the environment leads to its pollution and causes many other problems. Therefore, it is necessary to develop methods that will enable the reusage and exploitation of by-products like husk, hull, cake, pomace or meal, gum, soap stock, spent bleach earth/clay and distillate [172]. By-products obtained from olive wood and olive oil extraction are known as "olive by-products". A large number of by-products and residues obtained from olive (*Olea europaea*) harvesting industries were preserved over the years and had no practical application for a long time. More recently, it was confirmed that residual mass is a favorable source of energy and chemicals. Olive pomace is one of the two byproducts of olive oil production. It consists of olive pits, skins and pulp and contains 35–40% of the total weight of olives. The most important ingredients of olive pomace are oil, water, proteins and polyphenols. The composition of olive pomace, apart from the variety of olive, also depends on the method of the production of virgin olive oil. If oil is obtained by pressing on hydraulic presses, olive pomace has low oil content and vegetable water. Lately, olive processing by-products such as olive pomace have attracted great interest from scientists due to the presence of biologically active compounds, which have a beneficial effect on human health [173,174]. The benefit is mostly contributed by the high content of phenolic compounds such as phenolic acids, flavones, alcohol, lignans and secoiridoid derivatives, which possess various biological effects such as antimicrobial, antiviral, antioxidant, anti-inflammatory and anticancer properties [175,176]. The extraction of bioactive phenolic compounds for their potential applications in the pharmaceutical and food industries using conventional procedures usually requires a long extraction time and a large amount of solvents [177,178]. Today, alternative innovative methods such as microwave (MAE), ultrasonic extraction (UAE), homogenization-assisted extraction (HAE), and high hydrostatic pressure extraction (HHPAE) are replacing the conventional ones. The results of one study showed that ChCl:citric acid and choline chloride:lactic acid DESs were very effective in the extraction of phenolic compounds from olive pomace. The highest total phenol content as well as the highest antioxidant activity was obtained using the HAE and UAE methods combined with the ChCl:citric acid DES at optimal conditions of 60 °C and 12,000 rpm/min. Additionally, it was determined that the optimal conditions for the total content of polyphenols using the MAE method were 60 °C, and the most suitable NADES is ChCl:LA, and for the HHPAE method in the same eutectic solvent, a pressure of 600 MPa and a time of 10 min. Moreover, HAE was the best extraction method compared to the others mentioned above. The extracts' HPLC phenolic profiles revealed that NADESs were more efficient in the extraction of desired compounds compared to aqueous ethanol or water. Through the synergy of new methods (HAE, MAE, UAE, HHPAE) and the combination of suitable NADESs, it is possible to achieve a high extraction efficiency in a significantly shorter time. The results confirmed that the correct combination of innovative extraction techniques and NADESs could be an excellent alternative for the sustainable and green extraction of phenolic compounds from plant sources [179]. The term "olive leaf" describes a mixture of leaves and twigs preserved during olive tree pruning and olive cleaning and picking and contains up to 10% of the total olive weight at olive oil mills. Olive leaf as biowaste is a rich source of natural bioactive compounds such as polyphenols, secoiridoids, phenylethanoids and others with many positive biological effects such as anti-carcinogenic, antimicrobial, antiviral, antihypertensive, antithrombotic, anti-inflammatory, hypoglycemic and hypocholesterolemic properties [180–185]. When combining microwave-assisted extraction with DESs in the extraction of phenolic compounds from olive leaves, choline chloride:ethylene glycol (1:2) proved to be the most effective at optimal extraction conditions of 79.6 °C, 43.3% water and 16.7 min of irradiation time [186].

6. Extractions from Animal By-products

The largest part of animal waste is produced in the meat industry as by-products after slaughter and represents raw materials for other industries. These are skin, bones, fat, hair, tendons, contents of the gastrointestinal tract, blood and some internal organs. Most animal by-products are bioactive peptides that have been intensively researched recently [187]. Blood and collagen are an abundant source of protein and are present at up to 4% of total animal weight [188]. Collagen is the body's own protein found in skin, cartilage, connective tissue and bones. Its nutritional value is very low because it is not made of essential amino acids. The most favorable way to create bioactive peptides from collagen is through the hydrolysis of microorganisms. Research has shown that peptides exhibit different biological properties such as AChE-inhibition [189], antimicrobial [190,191] or antioxidant effects [192].

NADESs have shown potential for application in industry, use in extraction, biocatalysis and electrochemistry [193]. NADESs were used in a simple and green analytical method for the extraction of free seleno amino acids from lyophilized milk samples. Of all tested NADESs, lactic acid:glucose (5:1); citric acid:glucose (1:1), and fructose:citric acid (1:1), lactic acid:glucose showed the best extraction capacity. After optimization, it was determined that the most important factors affecting the extraction capacity are the percentage of water in the DES, the duration of ultrasound, the amount of sample, and DES volume. The method is applicable on real samples such as cow's milk powder samples, freeze-dried selenium-biofortified sheep's milk and others. Extraction with eutectic solvents is a green method and at the same time a one-step alternative to the traditional solubilization of powdered milk samples [194]. Immunoglobulins (Ig) or antibodies are glycoproteins produced by plasma cells. The highly selective purification of Ig from quail eggs was performed via ultrasound-assisted liquid–liquid microextraction (UA-LLME) in a deep eutectic solvent formed by a combination of QAS and glycerol. The properties of aqueous two-phase systems with different salts of different anionic (K_2HPO_4, Na_2SO_4 and Na_2CO_3) and cationic combinations were investigated. Aqueous biphasic systems were designed with task-specific deep eutectic solvents (TDESs) (70%) combined with various salts (20%). TDESs based on benzyl tributyl ammonium chloride (BTBAC) had higher densities and an improved extraction efficiency [195].

Fish and seafood industry waste are a source of many bioactive compounds, such as omega-3 fatty acids, amino acids, peptides, enzymes, gelatine, collagen, chitin, vitamins, polyphenolic constituents, carotenoids, etc. It can also serve as a favorable material for the production of biodiesel. Depending on the structural and functional characteristics, bioactive compounds from fish waste as well as from seafood waste have potential applications in the food industry, the pharmaceutical industry, medicine and agriculture [196].

New studies have shown that NADESs based on terpenes are an alternative extraction medium for the recovery of astaxanthin (AXT) from waste biomass from shrimp shells, mussels, *Heamatococcus pluvialis* shells and from other biomasses. The results show that the extraction in terpene-based NADESs at 60 °C in a time of 2 h obtains yields similar to those of the Soxhlet extraction in 6 h with acetone as a solvent. With the application of NADESs, AXT yields were increased up to 657 times. Research has shown that terpene-based NADES extracts have the potential to inhibit colon cancer cells and affect the negative growth of Gram-positive and Gram-negative bacteria. Of the tested NADESs, menthol:myristic acid (8:1) proved to be the most effective solvent. The authors state that such extracts could be used in the pharmaceutical, food or cosmetic industries [197].

Fish scales are waste obtained in the fish processing industry. About 1 million tons of FS are produced every year [198]. Fish scales are a rich source of biocompatible hydroxyapatite (HAp) and contain proteins, collagen and keratin, a small proportion of fat and vitamins. HAp is increasingly used in the food industry for the adsorption of heavy metals in food. The research results showed that a choline chloride:glycerol (1:2) DES is the optimal solvent for the extraction of Hap, where under optimal conditions (70 °C, solid:liquid ratio 1:15 *g*/*g* and 2.5 h), a Hap yield of 47.67% ± 1.84% was obtained. The analysis of the chemical composition of Hap determined an irregular morphology and a high Ca/P ratio. However, a new, environmentally friendly method of Hap recovery from fish scales was developed [199].

In one study, a simple, green method based on a choline chloride:malic acid NADES for the extraction of chitin from shrimp shell was investigated. Chitin is linear polysaccharide composed of *β*-1,4-glycosidic-linked *N*-acetyl-D-glucosamine monomer units. The high efficiency of chitin extraction using eutectic solvents is attributed to the formation of hydrogen bonds between the eutectic solvents and shrimp shell components causing the rupture of the intramolecular network of hydrogen bonds and subsequent dissolution of chitin in the NADES and its separation from proteins. The research showed that this extraction method based on NADES is a suitable approach for the extraction of chitin from shrimp shells and can be applied in the extraction of biopolymers from natural sources [200].

7. Extractions from Other Agri-Food By-products

Eutectic solvents were used in the extraction protein of from bamboo shoots, also known as bamboo sprouts, which are the edible part of bamboo, of which the two most famous species are *Bambusa vulgaris* and *Phyllostachys edulis*. They are used in Asian cuisine in fresh, dried or canned form. The protein yield obtained via extraction using a choline chloride and levulinic acid eutectic solvent under optimal conditions (80 °C, 50 min, HBD:HBA molar ratio of 6, solid:liquid ratio of 30 mg/mL, 40% water *v*/*v*) was 39 mg/g dry weight for the tender tip of the bamboo shoot, 15 mg/g dry weight for the basal bamboo shoot, and 9.54 mg/g dry weight for the sheath. Compared to conventional extraction using sodium hydroxide, significantly higher protein yields were obtained with deep eutectic solvents [201].

Eucalyptus (*Eucalyptus globulus*) plantations provide a large amount of biomass residues such as leaves and branches. The leaves of *E. globulus* are rich in natural monoterpene eucalyptol, sesquiterpenes, flavonoids, tannins, phloroglucinol derivatives and related polyphenols [202]. Extracts and essential oils are used in different industries, like the pharmaceutical, agricultural, cosmetic and food industries, and in medicine for the treatment of tuberculosis, flu, different fungal infections and diabetes. The results showed that the quality of the extract was maintained when microwave and ultrasonic methods were applied with shorter extraction times. The specific energy consumption was 2 and 13 times lower for ultrasound-assisted extraction and microwave-assisted extraction, respectively, when compared to a conventional method. Eucalyptus leaf extracts are rich in quercetin 3-*O*-β-D-glucuronide, rutin, eucaglobulin or globulusin, cypellocarpin, sideroxylonal, ellagic acid, methylellagic acid pentoside, tellimagrandin, and others [203].

Forestry by-products created after felling and wood production are branches, leaves, bark, cones, roots, etc. They are a natural source of bioactive compounds. Ginkgo biloba is one of the oldest known trees on earth, whose fossil remains are around 200 million years old. It is a treasure of useful phytochemicals with multimedicinal applications. Several chemical compounds have been isolated from *G. biloba* with a wide range of biological activities. About 38 different flavonoid compounds were isolated and identified from Ginkgo biloba leaves. [204]. Ginkgo flavonoids are strong antioxidants, which act as free radical scavengers. They protect against capillary fragility, by reducing edema caused by tissue injury, have anti-inflammatory properties, etc. New terpenoids, lignans, alkylphenols and alkylphenolic acids, carboxylic acids, proanthocyanidins, polyprenols, polysaccharides and others were identified in Ginkgo leaves [205]. Plant extracts have exhibited a variety of pharmacological activities, including antibacterial, antioxidant, anti-inflammatory, antiallergic, and cytotoxic anticancer activities [206,207]. A new method for the green extraction of flavonoids based on DESs was developed. Fifty DESs were prepared in order to investigate the extraction of Ginkgo flavonoids. The research proved that three DES, choline chloride:1,3-butanediol, choline chloride:levulinic acid and 1,2-propanediol:levulinic acid, had a higher extraction efficiency than a conventional solvent mixture (70% ethanol in water). After the optimization process and defining the reaction conditions, the ChCl:LA DES containing 40% water was used for the extraction of Ginkgo flavonoids in a solvent:solid ratio of 10:1 (v/w), and after mixing at a temperature of 50 °C for 15 min, Ginkgo flavonoids were obtained from the powder of Ginkgo biloba leaves in a yield of 99.87%. The recovery of Ginkgo flavonoids in the DES extraction solution was achieved in a 93.7% yield using a macroporous resin [204].

8. Conclusions

Concern for the environment and the growing demand for various drugs and biologically active substances have led to a tendency of finding new uses for food and agricultural by-products. Since waste is rich in bioactive compounds, it can be called "raw material" and not "waste". This review paper presents an overview of DES applications for the valorization of mainly the food and agricultural industry waste, in order to obtain products with added value. The use of eutectic solvents as green solvents in the extraction of organic compounds from food industry by-products and agro-waste is in accordance with green chemistry principles. The effectiveness of the eutectic solvent depends on the structure, polarity and other physical and chemical properties of the solvent, as well as the extraction method used. Although the extraction mechanisms are still understudied, DESs are promising green solvents due to their tunable properties and numerous advantages over conventional solvents. They are widely used in different scientific areas, especially in the treatment of agricultural industrial waste, one of the strategic goals of green sustainable development. Choosing the best solvent remains a challenge for further applications in the extraction of bioactive compounds. DESs are very promising solvents that are yet to be investigated in more detail, but their potential has already been shown in various extraction processes and reaction systems. Their utilization could help in the development of methods for the usage of bioproducts in a cost-effective way. Therefore, the continuation of such DES investigations could lead to the development of green sustainable technologies.

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Abbreviations

DES	deep eutectic solvent
QAS	quaternary ammonium salt
QAC	quaternary ammonium cation
SMW	solid municipal waste
HIFU	high-intensity ultrasound extraction
HBD	hydrogen bond donor
HBA	hydrogen bond acceptor
NADES	natural deep eutectic solvent
THEDES	therapeutic deep eutectic solvent

TDES	ternary deep eutectic solvent
SLE	solid-liquid extraction
ROS	reactive oxygen species
RNS	reactive nitrogen species
DNA	deoxyribonucleic acid
PEF	pulsed electric field
GAE	gallic acid equivalent
DW	dry weight
ChCl	choline chloride
Glc	glucose
LA	lactic acid
CA	citric acid
Mal	malonic acid
AA	acetic acid
TPC	total polyphenol content
PC	punicalagin content
EC	ellagic acid content
RP-LC–QTOF-MS/MS	reversed-phase ultra-pressure electrospray liquid chromatographic time-of-flight massspectrometric method
USE	ultrasound extraction
HVED	high-voltage electrical discharge pretreatment
MAE	microwave-assisted extraction
HPLE	hot pressurized liquid extraction
UMAE-EtOH	ultrasonic microwave-assisted ethanol extraction methods solid–liquid
PLE	pressurized liquid extraction
Gsps	grape seed polysaccharides

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