Review

Bioactive Edible Films and Coatings Based in Gums and Starch: Phenolic Enrichment and Foods Application

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Abstract: Edible films and coatings allow preserving fresh and processed food, maintaining quality, preventing microbial contamination and/or oxidation reactions and increasing the shelf life of food products. The structural matrix of edible films and coatings is mainly constituted by proteins, lipids or polysaccharides. However, it is possible to increase the bioactive potential of these polymeric matrices by adding phenolic compounds obtained from plant extracts. Phenolic compounds are known to possess several biological properties such as antioxidant and antimicrobial properties. Incorporating phenolic compounds enriched plant extracts in edible films and coatings contribute to preventing food spoilage/deterioration and the extension of shelf life. This review is focused on edible films and coatings based on gums and starch. Special attention is given to bioactive edible films and coatings incorporating plant extracts enriched in phenolic compounds.

**Keywords:** edible coatings and films; phenolic compounds; starches; gums; antioxidant activity

1. Introduction

According to Adeyeye and co-workers [1], "Packaging is all products that are of any nature and used as the containment, handling, protection, presentation, and delivery of goods from the raw materials state to the processed goods and from the producers to the consumers." Therefore, food packaging is responsible for the containment and protection of food from spoilage, contributing to its validity, and should protect against microbial, chemical and environmental contaminants in assuring food quality and safety and increasing the shelf life of food products [2,3]. However, conventional plastics used for food packaging have some production and environmental issues due to the fact they are based on petroleum, are non-renewable or non-degradable, emit greenhouse gases and present a severe waste disposal concern [1,4,5]. Therefore, it is important to develop new packaging materials that are more sustainable and "green" with the same functions as conventional packaging [1,2,6,7].

In an attempt to solve this problem, biopolymers/bioplastics emerged. Biopolymers are organic polymers and according with its origin are classified, mainly, into three types: biomass-derived polymers (e.g., proteins and polysaccharides including chitosan, starch and cellulose); synthetic polymers derived from oil or biomass monomers such as polylactic acid (PLA), polycaprolactones (PCL) and polyvinyl alcohol (PVA); polymers produced by natural microorganisms or genetically modified (e.g., polyhydroxyalcanoates and bacterial cellulose) [1,6,8–10]. In general, bioplastics present some advantages relative to common plastics: namely, they use less energy and are biodegradable, safe and emit less greenhouse gases; provide longer shelf life and are suitable for the production of compost; the chemical structure is more diversified, making possible the customization of the properties of the final package according to the type of food; and possibility of nanoparticles incorporation and adding or improving important properties such as thermic stability, gas impermeability/barrier including

to oxygen, antimicrobial properties and strength [4,9]. Regardless of these advantages, bioplastics have some issues concerning its mechanical and barriers properties, which require the addition of additives or other synthetic polymers (e.g., via hydroxypropylation) improving the referred properties [1,7]. Therefore, biopolymers may not be 100% made of renewable sources as some are blends of natural polymers with synthetic polymers or incorporate additives that improve the functional properties of the final package [1].

Biopolymers are used to prepare edible films and coatings, materials with thickness below 0.3 mm that are produced through a blend of biopolymers with different additives dispersed in an aqueous phase, and are safe to eat [5,11,12]. Edible films and coatings are easily manufactured, economically viable [13] and provide mechanical protection and improvement of food quality through moisture regulation and internal equilibrium between gases and solutes [5,12,13]. In addition to that, these materials can protect against UV radiation, fungi and bacterial contamination [5]. Edible films and coatings have the capacity to carry bioactive compounds, such as phenolic compounds, vitamins, nutraceuticals and probiotics, which improve food quality and, at the same time, provide additional health properties to the consumer after food consumption [5,14]. All these characteristics contribute to shelf-life increase [5,12] and a reduction in the use of additives [15]. In general, edible films and coatings are classified into the following: oil/water emulsions and colloidal dispersions [5]. They can be applied by dipping, spreading, spraying and wrapping (Figure 1) [5,12,13]. They can also be classified according to the type of application: edible coatings applied by dipping, spreading or spraying and edible films produced by compression molding, solvent casting or extrusion and that are used to wrap food [5,12].
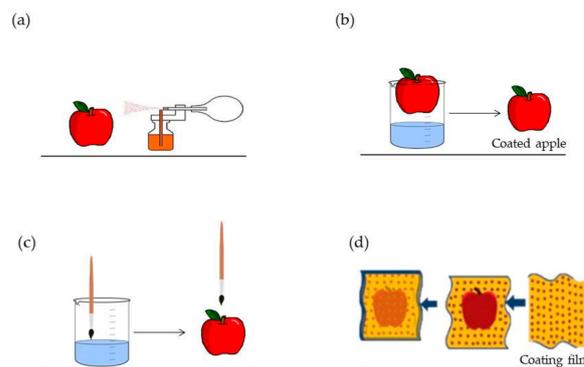
Figure 1. Schematic representation of coating techniques: (**a**) spraying; (**b**) dipping; (**c**) spreading and (**d**) wrapping (adapted from [16]).

In the food industry, food is submitted to several treatments (e.g., heat treatment, pH reduction, salting and drying) in order to prevent fungal and bacterial growth contamination, and synthetic antioxidants, namely butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT), are frequently added to prevent lipid oxidation [14,17]. However, nowadays, consumers tend to prefer food products that are minimally processed and that are healthier [14,18,19]. In this line, the scientific community evaluated the possibility of incorporating bioactive compounds (antioxidants, antimicrobials, flavours and probiotics) in food packaging, mainly in edible films and coatings to prevent microbiological contaminations and/or oxidative processes [14,18]. Natural antioxidants such as phenols, carotenoids and ascorbic acid are extracted from plants, vegetables, fruits, oil seeds and cereal crops [18,20,21]. Many phenolic compounds and essential oils from plants have antimicrobial activity [20] such as lemongrass, oregano, cinnamon, garlic [18] and thymus [21].

This review reports the most recent studies in this area and specifically gathers information about phenolic compounds incorporation in starch and gum edible films. The compilation of information is carried out in a systematic manner and is an asset for those who want to start studies in the scope of edible packaging based on gums and starches.

2. Gums Used in Food Packaging

Gums are complex polysaccharides of high molecular weight composed of glucose, fructose, mannose and other sugars [22], and they have the ability to form gels or mucilages [23]. They are also called hydrocolloids because of their solubility in water [23,24]. Gums are chemically inert, biocompatible, non-toxic, odourless and widely available in the nature [24]. Due to their chemical structure and properties, gums are used in the industry, namely in cosmetic [25], pharmaceutical [26] and food industry [24–27] as gel, thickener, moisturizer, emulsifier, stabilizer [23], coating agents and packaging films [27]. Natural gums are derived from plants, bacteria and animals [23]. In the vegetable kingdom, gums provide protection against microbial or mechanical injury [24,25] and can be found in plant seed endosperm (e.g., guar gum) [24,25]; plant exudates [24,25], shrubs or trees; and algae extracts (agar) [23]. The gums xanthan, pullulan, curdlan, gellan and cellulose are microbial gums, and according to their physicochemical properties, they are used to form gels (e.g., curdlan and gellan), thickeners agents (xanthan and pullulan) and film solutions (e.g., cellulose, pullulan and gellan) [22]. Examples of tree gums extruded are Arabic gum, tragacanth gum, ghatti gum, Persian gum, karaya gum and cashew gum [27]. Locust bean, guar gum (Figure 2), tara gum, basil gum, flaxseed gum, psyllium gum and Barhang gum are from seeds [27]. Konjac mannan is a tuber gum [27].

Xanthan gum (Figure 2), a Generally Recognized as Safe compound (GRAS) [22], is an exopolysaccharide produced by the bacteria *Xanthomonas campestris* at unfavorable conditions by fermentation [28–31]. Xanthan gum is an anionic polymer constituted by β-(1/4)-D-glucopyranose glucan (similar to cellulose) as a base framework and by the residues -(3/1)-α-linked D-mannopyranose-(2/1)-β-D-glucuronic acid-(4/1)-β-D-mannopyranose [23,30]. At low concentrations, xanthan gum presents high viscosity, and it is soluble in water, stable at a wide range of pH and temperature [10,23] and has resistance to enzymatic degradation. In 1969, xanthan gum was approved by FDA as a food additive [22], and later in 1980, it was recognized by the European Union under designation E415 [30] to be used as a stabilizing and thickening agent [32,33]. Moreover, in addition to its uses in the food industry as a stabilizer and thickener, xanthan gum is also used as gel, emulsifier and as an adhesive to prevent the formation of ice crystal [30]. In addition, the safety of xanthan gum as a food additive was re-assessed by EFSA in 2017, and this additive was considered safe for human consumption [31].
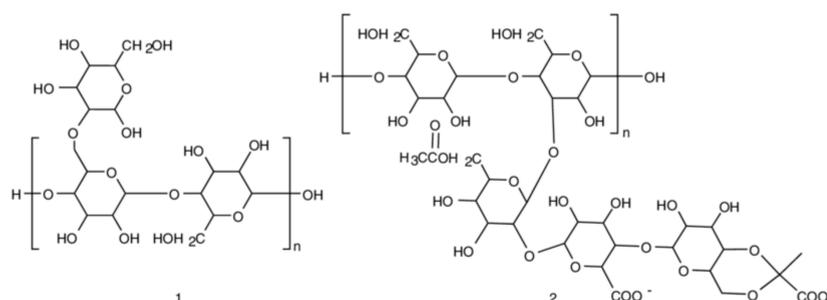
Figure 2. Struturee of guar gum (1) and xanthan gum (2) (retired from [33]).

Table 1 summarizes some of the gums used as food coatings or films and their properties as edible food packaging. In a general manner, most of the edible coatings of gums have antimicrobial properties, inhibiting its growth, preventing weight and moisture loss, providing firmness and preserving the organoleptic properties of the food, which, in this manner, contributes to quality maintenance and shelf-life extension. According with the data compiled in Table 1, xanthan, Arabic and guar gums are the most used gums in producing edible packaging. Moreover, dipping and immersion are the most commonly used food application methods.

Table 1. Gums coatings and films with or without incorporation of bioactive compounds and their properties as food preservatives.

| Polysaccharide	Food Application	Bioactive Compounds Incorporated	Type of Packaging	Application Coating Method	Results	References
Xanthan	Melon	β-carotene nanoparticles	Coating	Immersion	Improvement of coatings properties and increase in shelf time to 21 days at 4 °C	[34,35]
	Refrigerated fish	Chitosan	Film	-	Inhibition of the growth of *Staphylococcus coagulase-positive*, *Salmonella* spp. and coliforms at 45 °C Quality preservation	[36]
	Acerola	-	Coating	-	Reduction of weight loss and the respiration process Increase in shelf life (prolongation of 6 days at 30 °C without deterioration signs	[10]
	Pear	-	Coating	-	Retained the weight during 9 days of storage Prevention of oxidation	[37]
Galactomannan	Baby carrots	α-tocopherol	Coating	Dipping	Edible coatings improve the surface colour without organoleptic properties alterations	[38]
	Ricotta cheese	Nisin	Coating	Dipping	Delay of microbial growth during 28 days Weight loss and moisture content decreasing	[39,40]
	Fruits	Nisin	-	-	Decrease in gas transfer rates	[40–42]
Guar gum	-	Ag/Cu nanoparticles	Film	Spreading	Strong antibacterial activity against Gram-positive *Listeria monocytogenes* bacteria and Gram-negative *Salmonella enterica sv typhimurium* Excellent UV, light and oxygen barrier capability Firmness enhancement	-
	Roma tomato	-	Coating	Immersion	Reduce the weight loss Retarded loss of total acidity Respiration rate decrease	[42]
	Blackberries	-	-	-	Shelf-life extension for 13 days	[43]
	lemon	Spice extracts	Coating	Dipping	Shelf-life extension Maintenance of quality during cold storage Inhibition of bacterial growth	[44]
	Litchi fruits	-	Coating	-	Maintenance of fruit quality Shelf-life extension up to 10 days under low temperature storage	[45]
	Red chilli pepper	-	Coating	Dipping	Maintenance of fruit quality without deterioration during 20 days at storage temperature of 6 °C	[46]

Table 1. *Cont.*

Polysaccharide	Food Application	Bioactive Compounds Incorporated	Type of Packaging	Application Coating Method	Results	References
Arabic gum	Tomato	-	Coating	Immersion	Delay of the ripening process Shelf-life extension for 20 days without deterioration and off-flavours, stored at 20 °C	[47]
	Guava	Sodium caseinate and Tulsi extract	Coating	Dipping	Maintenance of suitable internal gas composition delaying ripening Shelf-life of 7 days at 28 ± 2 °C compared to 4 days of control	[48]
	Green chillies	Glycerol Thyme oil Tween 80	Coating	Dipping	Preservation of the quality and organoleptic properties During 12 days	[49]
	Persimmon fruits	-	Coating	Dipping	Lower weight loss, membrane leakage, H_2O_2 and malondialdehyde content relative to the control Suppression of the increase in activities of polygalacturonase, pectin methylesterase and cellulase enzymes Higher superoxide dismutase, peroxidase, ascorbate peroxidase and catalase activities	[50]
	Mangoes	-	Coating	Dipping	Gas and water vapour barrier properties Slower the ripening process Shelf-life extension for 15 days relative to less of 10 days in control	[51]
Gum tragacanth	Button mushroom	Aloe vera leaves extract	Coating	Immersion	Slow the loss of weight and colour changes under cold storage	[52]
	Fresh apricots	Chitosan	Coating	Dipping	Improvement of firmness and stability in terms of weight loss, pH and moisture content during storage	[53]
		-	Coating	Dipping	Good sensorial qualities Antioxidant properties Maintenance of ascorbate peroxidase (APX), catalase (CAT), superoxide dismutase (SOD) and peroxidase (POD) enzymes activities Inhibition of polygalacturonase (PG), pectin methylesterase (PME) and cellulase (CX) enzymes activities	[54]
Locust bean	Fortune mandarins	-	Coating	Immersion	Weight loss delay Improve gloss of the fruits	[55]

Table 1. *Cont.*

Polysaccharide	Food Application	Bioactive Compounds Incorporated	Type of Packaging	Application Coating Method	Results	References
Konjac glucomanann	Fresh-cut cucumber	Saffron petal extract	Film	-	Reduction in the water vapour permeability Antimicrobial properties against *Escherichia coli* (*E. coli*), *Shigella sonnei*, *Salmonella* Typhi, *Staphylococcus aureus* (*S. aureus*) and *Bacillus cereus* Preservation of fruits and vegetables quality Shelf-life extension	[56]
[57]	Guava	-	Coating	Immersion	Maintenance of firmness and colour Reduce weight loss	[57]
	Cantaloupe	Potassium sorbate	Coating	-	Maintenance of weight loss, hardness and firmness Inhibition of microbial growth Preservation of sliced cantaloupe up to 5 days	[58]
Gellan gum	Mango	-	Coating	Dipping	Improvement of sensorial characteristics namely appearance and firmness Stabilization of colour and volatiles composition during storage	[59]
Almond gum/Persian gum	Tomato	-	Coating	Immersion	Delay changes in colour, weight loss, firmness, acidity, ascorbic acid content, soluble solids concentration and decay percentage during a storage period of 20 days.	[60]
	Cherries	Gum Arabic	Coating	Immersion	Delay the ripening process and increase the shelf life of cherries without spoilage or off-flavour	[61,62]

3. Starches Used in Food Packaging

Starch is produced by plants, some strains of fungi and algae and is used for energy storage [63]. It comprises amylose and amylopectin polymers (Figure 3) [7,64,65] made up of α-D-glucose monomers [7,64]. Amylose is mainly a linear polymer where α-D-glucose monomers are linked by α-1,4-glycosidic bonds with some ramifications connected by α-1,6-glycosidic bonds while amylopectin is a branched polymer linked by α-1,4 and α-1,6 glycosidic bonds [7,64]. In general, starch has more amylopectin abundance than amylose although this ratio depends on the starch origin, resulting in a range of granules crystallinities [64,65]. Starch can be found in potato, cassava, wheat, rice, corn, maize, bean and in other sources [2,64].
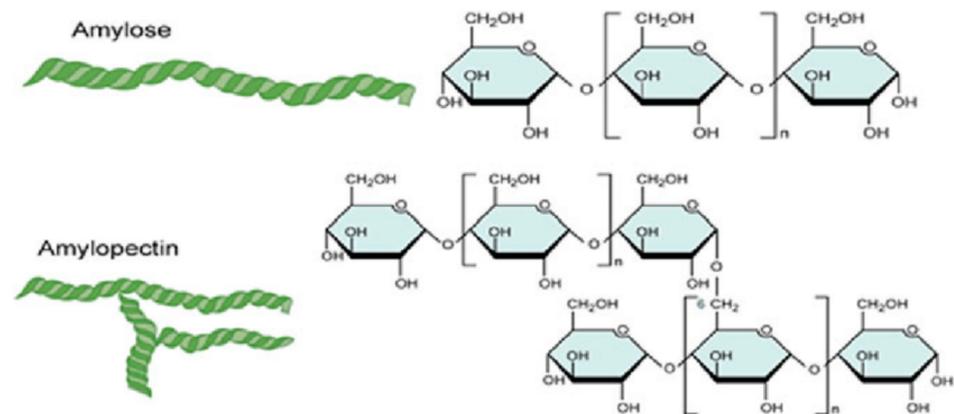
Figure 3. Structure of amylose and amylopectin that constitute starch molecule (retired from [65]).

In the food industry, starch is used in food packaging as coatings or films produced mainly by extrusion and casting [2]. In the manufacture of starch edible coatings, amylose is preferred over amylopectin [66]. Starch based films are low cost, abundant, tasteless, colourless and odourless with very good oxygen barrier properties (due to its compact structure and low solubility) contributing to the improvement of food preservation and, consequently, its shelf-life extension [65–67]. In addition, starch is biodegradable and edible [67]. However, starch presents some disadvantages due to its hydrophilic nature caused by hydroxyl groups and formation of hydrogen bonds among starch molecules [66]. The hydrophilic nature of starch-based films is responsible for moisture absorption, resulting in swelling of the starch matrix, disruption of H bonds and increase in free volume within the starch-based film matrix. These events cause the weakening of moisture and gases barriers and low water vapour barrier capacity [7,68]. In addition, starch films have poor mechanical properties, resulting in the use of additives, including plasticizers (e.g., glycerol and sorbitol) [68] and hydrophobic substances [67] in order to improve these properties [64]. Although starch is relatively cheap, the incorporation of others substances to improve the film matrix makes starch films more expensive [64].

In food packaging, corn-starch is used as thermoplastic starch and is commercially available under brand names such as EcoframTM (National starch), solanylTM (Rodenburg biopolymers), BiocoolTM (Novamont), BioplastTM (Biotec) and PlanticTM from Plantic Tecnhologies [69]. Table 2 summarizes the uses of starch-based edible films and its benefits in food packaging. Among the described starch-based coatings and films compiled in Table 2, cassava starch is the most commonly used. Starch-based coatings and films are mainly applied in highly perishable foods such as fruits and vegetables.

Table 2. Starch based coatings and films with or without incorporation of bioactive compounds and their properties as food preservatives.

Starch Source	Food Application	Bioactive Compounds	Type of Packaging	Application Coating Method	Results	References
Tropical fruits: banana "Pear", soursop, stenospermocarpic mango	Stenospermocarpic mangoes	-	Coating	Immersion	Mango starch showed better results than the other starches coatings tested: less weight loss, greater firmness, high content of total soluble solid and shelf-life extension up to 15 days (10 days at 10 °C and 5 days at 22 °C)	[70]
Corn	Banana	Gum Arabic Glycerol Sorbitol	Film	Dipping	At a temperature of 26 °C, the coated fruits lose less weight than control (uncoated fruits), retaining firmness and delaying the ripening process	[71]
Cassava	Mango	Chitosan	Coating	Immersion	At 25 °C, the coating showed good sensorial qualities and decreased respiration rate without prejudicing the ripening process Shelf-life extension up to 3 days	[72]
Rice and cassava	Pummelo	Pummelo juice	Film	Immersion	Lower weight loss Physical appearance stabilization	[73]
Potato	"Orri" mandarins	Glycerol	Coating	Immersion	Weight loss reduction	[74]
Tapioca	Cauliflower	Gelatin	Coating	Dipping	Weight loss reduction Total soluble solids increase	[75]
Cassava	Blackberries	Chitosan Glycerol	Coating	Immersion	Good sensorial properties (colour maintenance) Weight loss reduction and firmness increase during 10 days of storage	[76]
Banana "Pear"	Mango	Pectin	Coating	-	High firmness High total soluble solids Post harvested period extended to 21 days Retention of colour mango fruits	[77]
Wheat	Plums	-	Coating	Immersion	No colour changes Improvement of water vapour and oxygen permeability	[78]
Chickpea	Papaya	Glycerol Stearic acid	Film	Dipping	Reduced weight loss, better firmness and colour retention at 10 °C during 10 days of storage	[79]

Table 2. *Cont.*

Starch Source	Food Application	Bioactive Compounds	Type of Packaging	Application Coating Method	Results	References
Cassava	Black mulberry	Chitosan	Coating	Immersion	Minimized weight loss and mold decay during cold storage (5 °C) for 16 days No alterations at firmness, colour and anthocyanin content	[80]
	Tomato	Vegetable oil, glycerol, soy lecithin and cellulose and derivates	Film	Immersion	Delay changes in firmness, weight, titratable acidity, pH, total soluble solids, sugar/acidity ratio and colour development Increased shelf life of tomatoes stored at 20 ± 2 °C up to 1 month	[81]
	Toasted groundnuts	Soy protein concentrate	Coating	Dipping	Excellent sensorial properties Shelf-life extension during 14 days at ambient temperature	[82]
	Pineapple	Alginate Ascorbic acid Glycerol	Coating	Immersion	Lower levels of reducing and total sugars Better appearance and general acceptance during room storage for 18 days Preservation of sweetness, taste and odour and better appearance	[83]
	Guavas	Gelatin Chitosan	Film	Immersion	Decrease weight loss Shelf life increased up to 9 days relative to non-coated guavas Slowed the ripening process (after 27 days of storage, the fruits had not reached senescence)	[84]
Waxy corn	Rice cakes	Gellan gum	Film	Dipping	Preservation of texture of rice cakes during 24 h of storage Reduction in moisture loss and delay of hardening	[85]
Potato	Grape	Rice bran oil	Coating	-	Good water vapour barrier Preservation of the grape's quality Long-term shelf-life extension	[86]
Corn	Apples	Papaya polysaccharides	Film	-	Increase in swelling and tensile strength, reduction in thickness, transparency and solubility Improved sensorial acceptance	[87]

4. Methods of Incorporation Phenolic Compounds in Gums and Starch-Based Coatings and Films

The incorporation of phenolic compounds must consider final packaging characteristics, the bioactive substances used and their application in foods. To produce active films, the incorporation of bioactive compounds can be made by mixing them directly with films production or by encapsulation resulting in nanoparticles formation [88]. The usual methods for starch films production are by casting, pressing and extrusion [88]. The solvent casting method is the most used for the incorporation of phenolic compounds in polysaccharide films or coatings [89]. With respect to the formation of film, the polymeric solution is spread on the intended surface and posteriorly air dried in a ventilated oven. After solvent evaporation, the film is removed from the surface maintaining the surface form. However, the removal of film surface can cause it to wrinkle and tear. The physicochemical properties of films depend on the composition of casting solution, wet casting thickness, temperature and relative humidity conditions [89]. The extrusion method is based on the thermoplastic properties of biopolymers. The biopolymer solution contains a plasticizer that is heated above its glass transition temperature under reduced water conditions. Posteriorly, the films are submitted to compression, casting and extrusion, resulting in the formation of more elastic films [89]. The applications of coatings, thickness and their ability to adhere to food surface are important characteristics for food quality preservation. Therefore, coatings can be applied by spraying, dipping and spreading [89].

Table 3 summarizes some of the used formulations for incorporating polyphenols in active starch/gums films. Active starch and gum-based films and coatings were prepared mainly by casting method. Glycerol was the most used plasticizer in the preparation of active coating and films [90–94]. In general, the percentage of bioactive compound or extract in starch and gum-based coatings is low, ranging between 0.03 and 1.20%, and all the extracts and isolated bioactive compounds tested acted at the level of lipidic oxidation and microbiological contamination, preventing food spoilage and increasing its shelf-life.

Table 3. Formulations and main effects of phenolic compounds incorporated in starch and gums edible films or coatings.

| Active Compound.	% of Bioactive Compound/Extract in the Formulation	Material Base	Methodology	Food Application to Evaluate Effectiveness	Means/Tests/Assays for the Evaluation of Films Effectiveness	Main Effects/Conclusions of the Study	Reference
Green tea	5%	Cassava starch	Casting	Butter	Film characterization (thickness, mechanical properties and water vapour permeability); peroxide index	Improved functional properties mechanical, water vapour barrier and antioxidant properties of the resulting films. The results provide oxidative protection in packaged butter, by decreasing peroxide index, when using these film additives at low concentrations	[90]
Tea polyphenols	0.06%, 0.03% and 0.6%	Hydroxypropyl starch	Casting	N/A	Antioxidant tests performed by using DPPH free radical assay scavenging; Antimicrobial activity against Gram-positive bacteria *S. aureus* and Gram-negative bacteria *E. coli* was evaluated by shake flask culture method	Slight impact on the surface and barrier properties of the films due to a good dispersity of tea polyphenols in the starch matrix through hydrogen bonding improved antioxidant activity and antimicrobial activity, while inducing a decrease in mechanical properties	[91]
Pink pepper phenolic compounds	4%, 6% and 8%, *v/w*	Rice starch/fish protein	Casting	Fresh-cut apples	Evaluation of antioxidant activity by DPPH assay and inhibition of peroxidase	Rice starch/fish protein films with 6% (*v/w*) pink pepper phenolic compounds achieved better conservation during 12 days, especially in terms of inhibition of enzymatic browning.	[94]

Table 3. *Cont.*

Active Compound.	% of Bioactive Compound/Extract in the Formulation	Material Base	Methodology	Food Application to Evaluate Effectiveness	Means/Tests/Assays for the Evaluation of Films Effectiveness	Main Effects/Conclusions of the Study	Reference
Origanum vulgare L. essential oil	0.4%, 0.8% and 1.2% (*w/v*)	Fish gelatin/chitosan	Casting	N/A	Characterization of films Antimicrobial activity evaluation against *S. aureus*, *L. monocytogenes*, *S. enteritidis* and *E. coli*	The films were more effective against Gram-positive bacteria (*S. aureus* and *L. monocytogenes*) decrease) in tensile strength and elastic modulus Increase water vapour permeability in a dose-dependent manner Higher barrier capability relative to UV light	[95]
Quercetin	0, 0.025%, 0.05% and 0.1%	Cassava starch/gelatin	Casting	Pork Lard	Antioxidant activity assessment by DPPH assay; Film mechanical properties determination by analytic techniques; Estimation of peroxide value	The films have antioxidant capacity; Tensile strength, water solubility and water vapour increasing and elongation at the break of composite films decreasing, at 34% of relative humidity Quercetin delayed the oxidation of lard for more than 35 days and redness discolouration of pork	[92]
Feijoa (*Acca sellowiana* (Berg) Burret) pulp and husk extracs	25% (*v/v*)	Starch/citric pectin	Casting	Beef Bread Grapes	Films characterization; Antimicrobial activity against *E. coli* (Gram-negative), *Salmonella* (Gram-negative), *Shigella* (Gram-negative), mold and yeasts Lipid oxidation inhibition studies	Strong antimicrobial activity in beef and bread; Stabilization of lipid oxidation reactions in meat during six months of refrigerated storage; Maintenance of grapes conservation for 30 days	[96]

Table 3. *Cont.*

Active Compound.	% of Bioactive Compound/Extract in the Formulation	Material Base	Methodology	Food Application to Evaluate Effectiveness	Means/Tests/Assays for the Evaluation of Films Effectiveness	Main Effects/Conclusions of the Study	Reference
Persicaria minor leaves ethanolic extracts	0.4%, 1.0% and 2.0% *w/w*	Carrageenan	Casting	Meat patties	Antioxidant activity determination by DPPH and ORAC methods; Lipid degradation study by TBARS assay Lipid oxidation evaluation through malondialdehyde (MDA) assay;	High antioxidant activity; Lower lipid deterioration exhibited by 2% *Persicaria minor* extract active film	[97]
Oregano, clove, and rosemary leaves essential oils	Several compositions	Starch	Extrusion	Beef	Antioxidant activity assessment by DPPH and ORAC methods; Antimicrobial activity against *E. coli, S. aureus, Lactobacillus Plantarum* and *Pseudomonas aeruginosa* (*P. aeruginosa*) during 12 days of refrigerated storage	Effective inhibition of tested bacteria in 3% or more oregano essential oils; Clove oils showed greater antioxidant activity compared to oregano and rosemary essential oils, retarding lipid oxidation more effectively	[98]
Ethanolic extracts from *Capsicum chinense* Jacq. fruits	n.d.	Arrowroot starch	Casting	N/A	Evaluation of antioxidant activity by DPPH, ABTS and FRAP; Characterization of films	Increase in thickness, decrease in solubility and change in colour in a dose-dependent manner; Good antioxidant activities	[93]
Spent black tea extract	0.17% and 0.34%	Cassava starch	Casting	Aqueous and fatty food simulants; Soybean oil	Evaluation of antioxidant activity by DPPH assay and determination of peroxide value	High antioxidant properties due to radical scavenging preventing lipid oxidation in soybean oil	[99]

5. Phenolic Compounds Incorporated in Gums and Starches for Preparing Active Films

Phenolic compounds are secondary metabolites produced by plants (seeds, fruit, peels and leaves) [100] to protect them from environmental aggressions such as UV light, infections and herbivores [101]. Polyphenols have at least one aromatic ring with one or more hydroxyl groups attached and according to the number of aromatic rings and its functional group. Polyphenolic compounds are classified into phenolic acids, flavonoids, anthocyanins, stilbenes, lignans, coumarins and tannins (Figure 4) [101–103]. These compounds exhibit several biological activities such as antioxidant [103,104], anti-inflammatory [105–107], antimicrobial [106] and antidiabetic [104,108] properties.
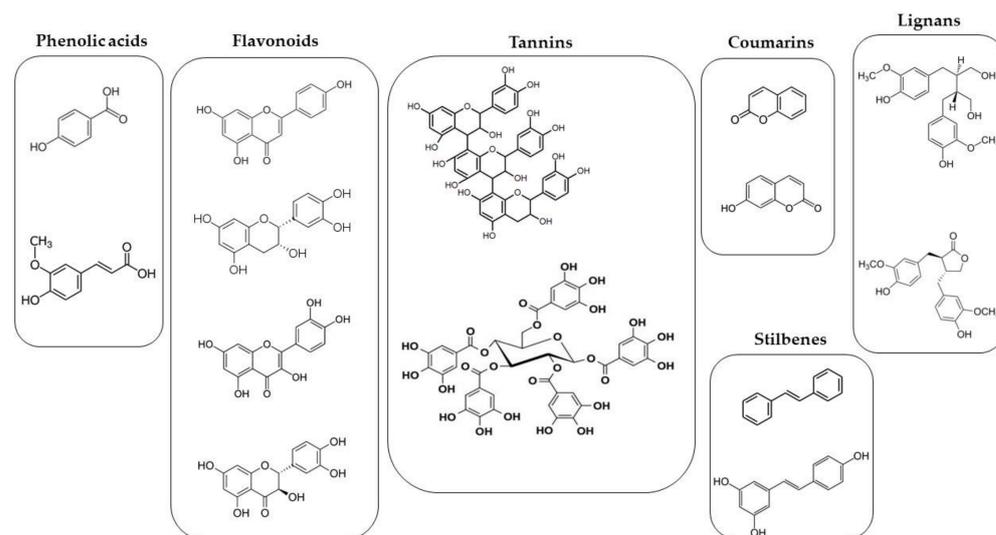
Figure 4. Examples of phenolic compounds structures presents in plant extracts (https://chem.nlm.nih.gov/chemidplus/, accessed on 10 November 2021).

Food storage is limited by enzymatic, non-enzymatic and microbial reactions that alter the quality of the food through flavour, texture and aroma changes and deterioration [108]. Intrinsic chemical reactions that occur in foods are linked to microbial contamination, as changes in pH, temperature, light and exposure to air create favourable conditions for microbial growth. These chemical reactions are oxidation, proteolysis, putrefaction, Maillard reaction, pectin hydrolysis and hydrolytic rancidity [108].

Proteolysis occurs in nitrogen-containing compounds, where proteins are converted into small amino acids. The Maillard reaction is a non-enzymatic reaction that occurs in groups of amino acids in proteins or in amino acids present in food. This reaction is responsible for darkening colour and reducing protein solubility and is responsible for developing undesirable flavours and decreasing the nutritional properties of foods. The Maillard reaction occurs mostly in milk, dry whole eggs and breakfast cereals. Pectin hydrolysis occurs in fruits and vegetables during the ripening process or due to mechanical damages that activate indigenous pectinases, favouring microbial contamination. Moreover, pectin can be de-esterified by methyl esterase responsible for spoilage and colour change in fruit jams or jellies. Hydrolytic rancidity occurs mainly in fats, such as butter [108]. Lipid oxidation is responsible for the development of off-flavours and rancidity, the formation of toxic aldehydes and the loss of nutritional quality due to unsaturated fatty acids degradation, namely polyunsaturated fatty acid (PUFA) [109]. In addition, foods are prone to microbial contamination, also resulting in food spoilage. In order to solve these problems, synthetic preservatives, such as BHT and BHA, are commonly added to foods in order to prevent lipid oxidation and food spoilage [17,94,110–112]. However, due to the apparent toxic effects of these additives and the search for healthier food products [93], natural compounds such as polyphenols appear as an alternative to synthetic additives [17],

serving as a valuable ally in the quality preservation of foods and extending their shelf life [103].

Antimicrobial activity of polyphenolic compounds is generally related with the induction of the leakage of cellular content; blocking DNA/RNA/protein synthesis; and, consequently, their function; modifications on membrane potential; disruption of membrane structure and function and induction of cytoplasmatic acidification; and coagulation of cytoplasmatic constituents [112]. On the other hand, most polyphenols present antioxidant properties as they can act against oxidative species such as Reactive Oxygen Species (ROS) through radical scavenging and also prevent ROS generation by iron chelation, presenting antioxidant properties [113]. In addition to its biological properties, many polyphenols have the capacity to enhance physico-chemical properties of the film or coating packages such as enhancement of its water/vapour barriers, tensile strength, colour, solubility and organoleptic characteristics [100]. Essential oils also have antimicrobial activity against bacteria and fungi. When added to films or coatings, due to its volatile compounds content, essential oils can also modify the organoleptic characteristics of foods (e.g., unpleasant odour and flavour). However, comparatively to plant extracts, the production of essential oil has lower yields and, consequently, less economic value. Thus, essential oils applications in food packaging are limited [112].

There are several studies concerning the incorporation of polyphenols in films containing polysaccharides. Cassava starch films with green tea and aqueous palm oil extracts were developed and applied in butter. The active edible starch films presented antioxidant activity decreasing the peroxide index, but in high concentrations of green tea extract polyphenols can act as pro-oxidants. Moreover, green tea and palm oil extracts reduced tensile strength and water vapour barriers [90].

Mango peel powder, a by-product, was added to starch edible films and applied on apple slices storage at a temperature of 4 °C. The phenolic compounds present in mango peel powder were responsible for higher antioxidant activity. The mango peel extract also increased the resistance of edible films, maintaining the film structure as almost intact [114].

In order to preserve acidic foods and drinks (e.g., wine and juices), chitosan/genipin active edible films were developed. The authors fractionated the wine extract in three fractions and produced three active edible chitosan films: chitosan films with phenolic compounds mixture (PCM), chitosan films with anthocyanins (AN) and chitosan edible films with phenolic acids (PA). The qualitative analysis of phenolic acids fraction showed the presence of gallic ((6), Figure 5), *p*-coumaric ((3), Figure 5), *trans*-caftaric ((7), Figure 5), *p*-hydroxybenzoic ((5), Figure 5), caffeic ((1), Figure 5) and chlorogenic acids ((4), Figure 5), (+)-catechin ((23), Figure 5) and (−)-epicatechin ((24), Figure 5). The anthocyanins fraction was mainly comprised malvidin-3-glucoside ((19), Figure 5). PCM and AN active edible chitosan/genipin films showed lower solubility and, therefore, lower weight loss. Concerning antioxidant activity, PA chitosan films were the most active followed by anthocyanins and phenolic compounds mixture. Thus, this active chitosan film has potential as an acidic food preservative [115].
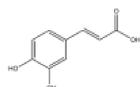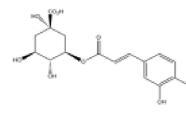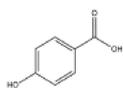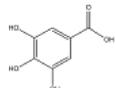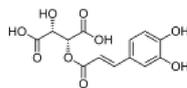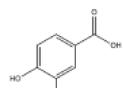
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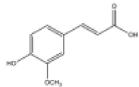
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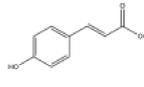
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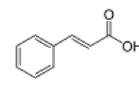
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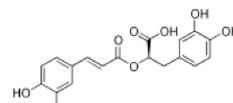
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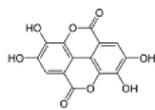
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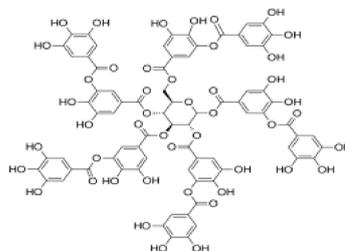
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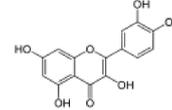
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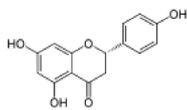
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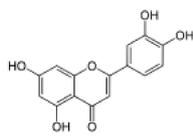
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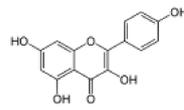
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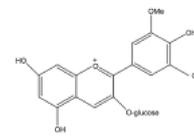
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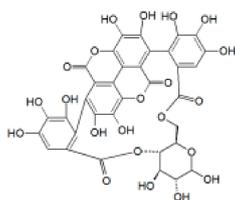
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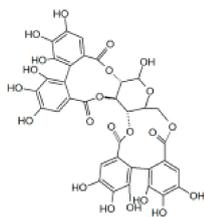
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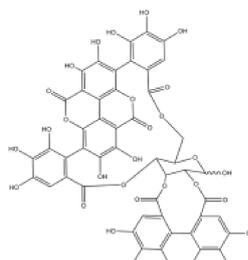
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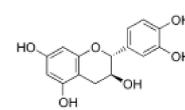
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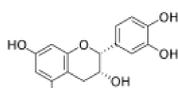
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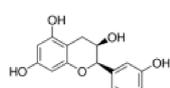
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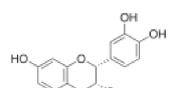
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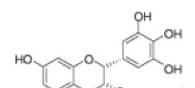
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Figure 5. Some phenolic compounds used in the described studies: (1) caffeic acid; (2) ferulic acid; (3) *p*-coumaric acid; (4) chlorogenic acid; (5) p-hydroxybenzoic acid; (6) gallic acid; (7) *trans*-caftaric acid; (8) *trans*-cinnamic acid; (9) vanillic acid; (10) protocatechuic acid; (11) gentisic acid; (12) rosmarinic acid; (13) ellagic acid; (14) tannic acid; (15) quercetin; (16) naringenin; (17) luteolin; (18) kaempferol; (19) malvidin-3-glucoside; (20) punicalin; (21) pedunculagin; (22) punicalagin; (23) (+)-catechin; (24) (−)-epicatechin; (25) (−)-epigallocatechin; (26) (−)-epicatechin gallate; and (27) (−)-epigallocatechin-3-gallate.

Thyme infusion was incorporated in starch and chitosan films in order to improve antioxidant activity in food preservation. The infusion has an acceptable taste and has in their composition caffeic acids and its oligomers such as flavonoid glycosides, hydroquinone derivatives and terpenoids. Rosmarinic acid is the main phenolic acid. In addition to its antioxidant activity, thyme extract changed, in a good way, the physico-chemical characteristics of films matrices. The chitosan film has higher tensile properties presenting better resistance at break and higher degrees of stiffness. However, the films become more opaque and less stretchable [116].

Gelatin/chitosan active edible films with gallic and *trans*-cinnamic acids were developed and tested regarding their antioxidant and antimicrobial properties. The results showed that gallic acid chitosan films were more active than *trans*-cinnamic acid ((8), Figure 5) in both tested activities. However, the addition of food simulants (e.g., water and ethanol) decreased phenolic acids activities. The authors concluded that gelatin/chitosan active films with gallic acid are suitable for fatty and dry foods [117]. Chitosan active edible films with pomegranate peel ethanolic extract were developed, and its antioxidant properties were evaluated. The ethanolic extract rich in flavonoids, anthocyanins, catechins, tannins (punicalin ((20), Figure 5), pedunculagin ((21), Figure 5), punicalagin ((22), Figure 5)), gallic and ellagic ((13), Figure 5) acids showed good antioxidant properties compared to BHA, which is slight lower when compared to ascorbic acids. Pomegranate extract also improved the mechanical properties of chitosan films and reduced the oxygen permeability [118], preventing lipidic oxidation reactions.

The addition of tea polyphenols (mixture of catechins) in chitosan, gelatin and starch edible films improved antioxidant and antimicrobial properties of the films. However, tea polyphenols decreased the mechanical properties of the films [91]. In another study, active edible starch-based films with potato peel phenolic extracts applied to smoked seabream revealed good antioxidant activity, good organoleptic properties, such as pleasant smell and flavour, and improved the fillets colour when compared to conventional plastic package [119].

Kaczmarek-Saczepanska and co-workers [120] have studied the incorporation of tannic, caffeic and ferulic acids in chitosan edible films. These films were irradiated with UVC light in order to sterilize them. Phenolic acids improved the physicochemical properties of films and ferulic acid showed good antimicrobial activity against *S. aureus* (Gram positive) and *E. coli* (Gram negative) [120].

Active chitosan coatings with clove (*Eugenia caryophyllata*) essential oil were tested in fresh apples. The clove essential oil composition in the weak acids' eugenol (68.95%) and acetyl eugenol (4.88%) enhanced chitosan solubility in aqueous solution and significantly reduced water vapour permeability. Moreover, the treated apples with active chitosan coatings delayed the spoilage process (firmness and colour) and showed antimicrobial activity against *P. aeruginosa*, *S. aureus* and *E. coli* [121]. The physicochemical and antioxidant properties of active *Capsicum chinense* Jacq. ethanolic extract/starch films were evaluated. Liquid chromatography coupled to mass spectrometry (LC-MS) analysis of ethanolic extract showed the presence of phenolic acids (protocatechuic ((10), Figure 5), gentisic ((11), Figure 5), caffeic, vanillic ((9), Figure 5), *p*-coumaric and ferulic ((2), Figure 5) acids), flavonoids (quercetin ((15), Figure 5), naringenin ((16), Figure 5), luteolin ((17), Figure 5) and kaempferol ((18), Figure 5)) and alkaloids (capsaicin and dihydrocapsaicin). Active starch films showed good antioxidant capacity attributed to phenolic compounds and increased films thickness and decreased solubility [93].

An extract obtained from *Araucaria angustifolia* (Bertol.) Kuntze cooking process, containing (+)-catechin ((23), Figure 5) and (−)-epicatechin ((24), Figure 5), was added to TPS (thermoplastic starch)/PBAT (poly (butylene-co-terephthalate) aromatic-aliphatic polyester) films. Moreover, phenolic compounds caused hydrophilic/hydrophobic alterations in matrix films and lowered its solubility, crystallinity degree and starch glass transition temperature [122], improving the flexibility of biopolymers [123].

Falcó et al. [124] studied the antiviral potential of green tea incorporated in alginate/oleic acid edible coating in strawberries and raspberries. Green tea is rich in polyphenols, mainly (−)-epicatechin, (−)-epigallocatechin ((25), Figure 5), (−)-epicatechin gallate ((26), Figure 5) and (−)-epigallocatechin-3-gallate ((27), Figure 5). Catechins are unstable at some values of pH, and its antiviral efficacy depends on these compounds. Thus, for pH above 5.5 the green tea extract inhibited completely murine norovirus and hepatite A virus after storage at 25 °C. The green tea extract also revealed antioxidant activity, which was not pH dependent [124].

The described studies show that the incorporation of phenolic compounds in active gums or starch edible films improves, in general, the physicochemical properties of the films and coatings in addition to its antioxidant and antimicrobial activities.

6. Intelligent and Active Starch/Gums Films with Phenolic Compounds

While active polysaccharides films have an active function for enhancing food quality and preventing food spoilage, intelligent polysaccharides films monitor food conditions inside packaging, providing information about food safety and quality [125]. Unlike active packaging, intelligent packaging does not release its compounds into food [126]. Bio-based smart packaging present environmental and economic advantages and allow real-time monitorization, assuring food safety and quality [125]. Intelligent packaging can be classified as indicators, sensors and data carrier. Indicators provide visual, qualitative or quantitative information about food to colour change or dye diffusion. Changes in pH and temperature are responsible for food packaging colour change and can be identified by indicators such as freshness, microbial spoilage, gases, integrity and time-temperature indicators (TTI). The most common indicators are based in pH changes due to metabolite production such as n-butyrate, L-lactic acid, D-lactate, acetic acid and volatile amines [126]. The second type of intelligent packaging is sensors, which detect and register information about biological reactions. The most common sensors are biosensors that use biological or organic material that recognize the respective analyte. Some biosensors are currently commercialized to detect food pathogens. Food Sentinel System (SIRA Technologies Inc.) is a biosensor used to detect the presence of pathogens through the formation of a dark colouration band. ToxinGuard® (Toxin Alert, Canada) is a biosensor that detects specific microorganisms such as *Salmonela* spp., *E. coli*, *Listeria* spp. and *Campylobacter* spp. The third type of smart packaging includes data carriers. Data carriers do not provide quantitative or qualitative information about food products. They are responsible for identification, automation, anti-theft prevention and traceability of food products. Foods can be identified through radiofrequency identification labels (RFID), bar and QR codes [126].

Polyphenolic compounds and others present in plant extracts (e.g., chlorophyll and carotenoids) have a proper colour that, due to pH variations, change to another colour [125]. However, the type of phenolic compounds chosen is important for intelligent film packaging development due to their physico-chemical properties and pH-sensitive capacity. Among phenolic compounds, anthocyanins and curcumin are more suitable for smart packaging development [127].

Fernández-Marín et al. [128] developed pH sensitive intelligent films by adding curcuma oil and anthocyanins extracts into a chitosan matrix reinforced with α-chitin nanocrystals. The essential oil of curcuma and anthocyanins extracts improved the mechanical properties of chitosan films, decreased water solubility and moisture content and blocked partially the UV/Vis light at wavelengths below 550 nm. Moreover, these intelligent films proved to be sensible to ammonia gas and different pH, changing the

colour of packaging. Due to these results, intelligent active chitosan films have potential to be applied as food quality sensors [128].

Yun et al. (2019) prepared a cassava starch film with anthocyanin-rich bayberry (*Myrica rubra* Sieb. et Zucc.) extract (BBE), with antioxidant and pH-sensitive properties. Phytochemical analysis of BBE was performed by HPLC-MS, identifying cyanidin-3-O-glucoside as the main anthocyanin. The addition of BBE into the film matrix improved water vapour permeability, tensile strength, UV-Vis light barrier and antioxidant properties. Relative to pH changes, cassava starch BBE film showed colour changes when hydrogen chloride and ammonia gases were present. The developed film was tested in pork where freshness and antioxidant capacity were evaluated. The results showed good antioxidant properties and pH-sensitive capability [129].

Other authors developed intelligent and active κ-carrageenan films incorporated with pomegranate flesh (PFE) and peel (PPE) ethanolic extracts. PFE presented higher total anthocyanins content; therefore, it was used for intelligent packaging film. Pomegranate peel extract was rich in total phenolic compounds responsible for antioxidant and antimicrobial properties. PPE incorporation in κ-carrageenan films increased tensile strength and decreased moisture content and water vapour permeability compared with PFE films. The pH sensibility of films was assessed. κ-carrageenan with PFE change its colour to pH variation (red at pH = 3 and yellow to pH = 11). Both PEE and PFE κ-carrageenan films showed good antioxidant and antibacterial activities when compared to control [130].

Several concentrations of Propolis extract and anthocyanin were added to polyvinyl alcohol (PVA)-starch films in order to prepare intelligent and active films. The prepared films were tested against *E. coli* and Methicillin-resistant *S. aureus* (MRSA) in pasteurized milk. The authors of this study verified that the film containing 20% of propolis extract exhibited better mechanical strength and highest antibacterial activity. Moreover, when immersed in different solutions of pH from 1 to 14, the films were responsive to these variations, changing its colour [131].

Roselle anthocyanins were used to prepare an intelligent active starch/PVA and chitosan films to monitor pork freshness. The physico-chemical properties and antioxidant activity were evaluated. The researchers observed that anthocyanins had a significant effect in the microstructure of the films, improving the compatibility of films and tensile strength. Anthocyanins Roselle extract showed good antioxidant activity and presented visible colour changes at pH variation (at 25 °C) of pork meat [132].

Anthocyanins from black soybean seed were extracted and incorporated in chitosan films. Phytochemical characterization was assessed by HPLC-PDA, and the antioxidant potential by DPPH assay was evaluated. The main identified anthocyanins were cyanidin-3-O-galactoside, cyanidin-3-O-glucoside and peonidin-3-O-glucoside. Anthocyanins from black soybean extract improved water vapour and UV-vis light barrier as well the mechanical strength of the films. on the other hand, the anthocyanins extract decreased moisture content and transparency. Regarding antioxidant activity, the extract exhibited antioxidant capacity in a dose-dependent manner. By changing pH, the films were able to colour change [133].

Intelligent and active packaging is an innovative, safe, healthy and eco-friendly method for enhancing food safety and quality, increasing food shelf life. Active and intelligent packaging possesses different functions, thus, choosing the better type of polyphenols to achieve the intended function of coating is necessary. Anthocyanins are suitable for smart packaging development while, for example, phenolic acids and flavonoids are more suitable for active packaging (due to its antioxidant and antimicrobial properties) [130].

Phenolic compounds are known to be unstable at pH and temperature variations and UV-vis light exposure [134]. In order to provide structural stability and to enhance biological properties, phenolic compounds are encapsulated and posteriorly added to starch/gums edible films [134]. Zhang et al. [134] studied β-chitosan nanoparticles of several sizes in order to encapsulate catechins by ionic gelation. The authors evaluated the antimicrobial properties of the developed catechins nanoparticles against *E. coli* and

Listeria innocua (*L. innocua*). The smaller nanoparticles exhibited higher antibacterial activity. Regarding catechin nanoparticles stability, they presented higher stability in the pH range 2.0–4.5, with nanoparticle precipitation occurring at pH above 5.0.

There are a few studies about the encapsulation of phenolic compounds and the incorporation of these nanoparticles in starch/gums edible films. Nanoencapsulation of polyphenols for food packaging application is an emerging area.

Polysaccharide films are a good alternative to common packaging, being healthier, eco-friendly and economical. In order to enhance food quality and safety, phenolic compounds could be used as an alternative to decrease or substitute synthetic preservatives, as well food indicators. However, polysaccharide matrices present some disadvantages relative to common packaging such as instable tensile strength, water-vapour barriers, solubility and others. The addition of phenolic compounds, in some cases, improves the physico-chemical properties of film matrix. In addition, in order to enhance film resistance, plasticizers are added. Polyphenols are obtained from natural sources but it is also from by-products (from agricultural wastes, for example) [130], where the latter is more economic. The diversified structures of polyphenols allow them to have two fundamental roles in edible packaging: used in active edible packaging due to their biological properties, namely antioxidant and antimicrobial; and in intelligent packaging, such as pH indicators, controlling and monitoring the quality and safety of food. Moreover, polyphenols have the capability to modulate the physico-chemical properties of starch and gums films. In some cases, they improve the mechanical characteristics of films as well barriers properties. Most phenolic compounds have no toxicity in human health [135]. On the other hand, phenolic compounds are unstable with respect to variations in pH, temperature and UV light exposure; thus, care must be taken when preparing extracts and incorporating them into films. The studies described in this review also show that some polyphenols can act as pro-oxidants when they are above certain concentrations.

7. Legislation

In the European Union, the legislation for food packaging is described in Regulation (EC) No 1935/2004 and its amendments. According to article 3 of this regulation [136], food packaging should not release any constituents for the food at a toxic level for humans as well as at levels that result in physicochemical and organoleptic changes in food composition, and they must have the correct information about the contained product [5]. For active and intelligent materials, the rules are described in Commission Regulation (EC) No 450/2009 [126,137] and its amendments. The active materials can release substances into the food acting as preservative or absorb substances from food packaging, such as liquids and gases (e.g., oxygen) [136]. In United States, the approval for food packaging is the responsibility of the Food and Drug Administration (FDA). According to this entity, for the approval of an edible packaging, the packaging must not only be safe but also listed as GRAS and used according with Good Manufacturing Practices (GMP) [138]. According to the regulations EU no. 10/2011, on plastic materials, and EC 450/2009, on active and intelligent materials, if components are not available in the positive list of the regulation EC no. 10/2011 (the list of approved compounds to be used in the manufacture of plastic-based packaging) and not specifically recognized as a food additive (e.g., catechins and caffeine), they must be below 10 µg per kg of food or food simulant [112]. When the biopolymer material is not listed as GRAS, the producer must apply for its approval, demonstrating product safety. In some cases, active edible packaging has essential oils in its composition; therefore, its toxicity and allergenic effects must be evaluated in order to grant food safety [138].

Due to the recent search and increase in natural preservatives use, the European legislation (EC/1334/2008) [139] has been amended in order to include information regarding natural extracts. According to FDA, natural compounds are accepted as safe and are allowed for food use in the case of the following: the dose required to produce the intended

effect is minimum, within the good manufacturing practice principles, and is applied in its forms (e.g., plant extracts and essential oils) [112].

The development of edible films or coatings is encouraged by the US Environmental Protection Agency's (EPA) due to its environmental properties. This agency aims to design new food packaging with less environmental toxic materials and its possibility to reuse or compost [138]. The International Organization for Standardization (ISO) establishes legislation for the production, distribution and use of packaging materials such as ISO 18604:2013(E). Moreover, this organization also has regulations concerning the reusability and biodegradability properties of packaging materials [5].

8. Conclusions

Natural polysaccharides are good alternatives to conventional packaging as they are generally more economical, environmentally friendly and biodegradable (at least to some extent). These polysaccharides form an edible matrix capable of coating food, thereby reducing the use of synthetic and non-biodegradable packaging. Given the demand for healthier and less processed foods, the addition of natural compounds, such as phenolic compounds, can reduce or replace synthetic additives, preventing lipid oxidation and contamination by microorganisms, improving the preservation of food quality and extending the expiration date. In addition to these biological properties, phenolic compounds also modulate the physical and chemical properties of films, which can improve or worsen them. More studies are needed in order to improve the physical properties of edible films or coatings. Active films or coatings containing polyphenols are an alternative with less environmental impact and also healthier for human consumption.

9. Future Perspectives

More studies are needed to improve the physico-chemical characteristics of films without plasticizer addition. Due to their diversity, phenolic compounds must be studied in order to improve the physico-chemical structure of polysaccharide films and achieve better biological properties. Polyphenols present in plant extracts are mostly glycosylated. Some studies mentioned here refer to genins as active compounds in the tested films. Thus, it is important to assess whether glycosylation and its degree interfere with the biological properties (antioxidant and antimicrobial) and physicochemical characteristics of the films.

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