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Biodegradable Films with the Addition of Nanofibers: a Review Focusing on Raw Materials and Analysis

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HIGHLIGHTS

- Starch is promising biopolymer to be used as a biodegradable natural source.
- The combination of starch with nanocellulose can provide new applications.
- Glycerol is an additive widely used in the production of biodegradable packaging.
- Biodegradable films are effective to extend the shelf life of fresh vegetables.

Abstract: Starch is an important energy reserve polysaccharide for vegetables, found in large quantities in nature. From a renewable and low-cost source, it is considered an excellent raw material for the production of biopolymers. Biodegradable films can be obtained from different starch sources and can be an alternative to the damages caused by petroleum products, since they have the properties of conventional plastics and are easily degraded in the environment. However, starch-based films have limitations such as poor mechanical properties. To overcome this weakness, the addition of secondary additives is used to improve its resistance. This review highlights how different materials used in the production of biodegradable polymers, the importance of incorporating reinforcement content in the polymer matrix and as the main characterization techniques of bio composites.

Keywords: starch; reinforcement; film properties; casting**.**

INTRODUCTION

Food packaging acts as an inert barrier between food and the environment, providing safety, extension of shelf life and allowing them to have a wide distribution. For this reason, it must be designed according to each product characteristic to guarantee its durability and also the maintenance of its sensory characteristics

[1]. The non-conventional films are a class of biodegradable polymers that are becoming very promising in the world Market. They presenting themselves as an alternative to the use of petrochemical derivatives found in current plastic packaging and contributing to the reduction of plastic waste [2,3].

According to European Bioplastics, Nova Institute (2019), bioplastics can be found in several market segments such as: packaging, food services, agriculture / horticulture, consumer electronics, automotive, consumer goods and home appliances. According to the Brazilian Association of Public Cleaning Companies [4], in last decades there was an increase in urban solid waste in the country. In 2018, 79 million t of wastes were generated in Brazil and more than 40% of the garbage collected had an improper destination. In 2019, global bioplastics production capacities totaled almost 53% of the volume destined for the packaging market - the largest market segment in the bioplastics industry. Global bioplastics production capacity is expected to increase from around 2.11 million t in 2019 to approximately 2.43 million t in 2024 [5].

Biodegradable films are prepared from a blend of organic compounds and water. Normally it is added to water a polysaccharide and a plasticizer. Starch is an organic compound widely found in nature and the most studied to be used in the production of thermoplastic packaging due to the ideal properties for a good film formation [6].

Films produced using only cited basic components have high hygroscope, are not very flexible and become brittle, which makes processing difficult for the production of packaging [7]. The functionality of films produced from starchy sources depends largely on their components. The combination of starch with another polymeric material, such as nanocellulose, is an alternative to improve the physical and chemical properties of the thermoplastic and provide new applications [8].

The use of a lignocellulosic fiber of high strength and durability incorporated in the film, aims to reinforce and improve the mechanical, barrier and thermal properties due to the nanometric size and the high crystallinity of the cellulose [9]. This review provides information to better understand the important factors in the development of more resistant films based on starch and highlights the main analysis of their characterization.

RAW MATERIALS

Starch

Starch is considered a promising biopolymer to be used as a biodegradable natural source with varied applications, such as in packaging, because it is a versatile, cheap and abundant raw material in nature [10].

In an essentially linear chain formed by glucose units joined by α-1,4 glycosidic bonds is amylose, which contributes mainly to the amorphous phase of the starch granule. Formed by glucose units joined in α-1,4 and α-1,6 bonds with highly branched structure is amylopectin, which predominantly contributes to the peripheral crystalline organization of starch granules [11]. The destruction of the original semicrystalline structure of the granule by a gelatinization process must occur above 70 °C in the presence of water so that when cooled there is the formation of a resistant and translucent film, similar to the structure of a thin cellulose film with great possibility of use of this material in food packaging [12].Table 1 shows works in which starch was used for the production of biodegradable films and nanoparticles as reinforcement material.

Table 1. Recent published effects of different starch types blended with nanoparticle, plasticizers on the films properties.

Polymer Source	Plasticizer	Origin of reinforcement	Main results	References
Potato, cassava and chitosan	glycerol	Turmeric	significant increase in tensile strength	[13]
Maize	glycerol	polyester and cotton	tensile strength and thermal stability have been significantly improved	[14]
Regular and waxy maize	glycerol	eucalyptus nanocellulose	higher tensile strength and thermal stability, lower water vapor permeability (WVP) and solubility	[15]
Elephant foot yam	glycerol	whey protein concentrate and psyllium husk	higher tensile strength, lower water vapor permeability, and solubility	[16]
Cassava and chitosan	glycerol	montmorillonite or bamboo	increase of tensile strength and of elongation at break	[17]
Rice	glycerol	mesocarp cellulose fiber	highest thermal stability and lowest water absorption	[18]
Banana	glycerol	nanofiber of banana peel	increase of tensile strength, Young's modulus, water-resistance, opacity, and crystallinity	[19]
Wheat	glycerol	cellulose nanocrystals from rice, oat, and eucalyptus	increase of tensile strength	[20]
Pea	glycerol	waxy maize starch	increase of tensile strength, and decrease of moisture content, WVP, and water-vapor transmission rate	[21]

Plasticizer

The most important and fundamental plasticizer that is always used in the manufacture of starch films is water, called the primary agent. The most used secondary plasticizing agents are polyols, which are organic compounds with multiple hydroxyl groups, non-toxic and considered safe for use in the food industry [11].

Glycerol is an additive widely used in the production of biodegradable packaging; within the polyol class it stands out as the most important for playing a vital role in the polymer industry. It is the most used plasticizer in the production of starch-based films, it alters the interface between adjacent molecules in the polymeric starch chain [22]. Other polyols that also stand out in their use are xylitol and sorbitol.

In the interstices within the starch molecule, the plasticizer replaces the intra and intermolecular hydrogen bond in the system with that of its functional group, thus increasing the intermolecular spacing. The least organized phase of the starch (amorphous phase) is the most prone to these strong interactions. This action results in a greater extension of the starch matrix, which improves the functional properties and produces less fragile, but less rigid films [22, 23].

Nanofibers

Fibers are bio-based materials, making it a fully biodegradable source. When in nanometer scale these fibers have characteristics of being highly resistant and crystalline. In starch composites, nanocellulose has promising properties such as increased mechanical performance and remarkably high transparency.

The composition of lignocellulosic fibers is very dependent on the source of the fibers. It is basically composed of cellulose that represents the crystalline portion and an amorphous portion corresponding to lignin and hemicellulose, in addition to other compounds present in lesser proportion such as pectin, ashes and waxes [24].

They are considered nanofiber when these structures have dimensions smaller than 100 nanometers. Nanofibers extracted from plant sources have potential characteristics for providing improved mechanical properties such as increased stiffness, strength and flexibility of the films, in the thermal properties of starch, ensuring greater thermal stability and for decreasing the sensitivity to water due to the fact that cellulose nanofibers have lower affinity for water [19].

Main methods and instrumental techniques for the characterization of biocomposites

Field Effect Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM)

Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) analysis are carried out to verify the microstructures' characteristics. Field effect scanning electron microscopy is a technique used to visualize the morphology of a material through enlarged images with high depth of focus and resolution [25]. The technique consists of emitting a thin beam of electrons (<1 nm) of high energy, in parallel lines, focused on the sample surface. TEM images are obtained using a power filter transmission electron microscope with an acceleration voltage equal or higher than 80 kV. To study the morphology of the films, a thin section is cut with a diamond knife in an ultra-microtome. The trimmed section is placed on a carbon-coated copper grid and stained with chemical like uranyl acetate and phosphotungstic acid [26].

Thermal analysis

Thermogravimetry (TG)

Thermogravimetric analysis is an adequate experimental technique to provide important information on polymers exposed to heating. Information such as thermal stability, thermal decomposition, identification of impurities and moisture. The equipment measures the temperature difference between the sample and a standard and the loss of mass as a function of time and / or temperature to monitor physical and chemical changes [27].

The thermogravimetric curve resulting from the analysis provides data on the mass losses of the samples and it is possible to quantify the mass variation that occurred as a function of the increase in temperature. In films these data include the evaporation of volatile constituents, such as water and glycerol volatilization, drying, moisture, decomposition and depolymerization of organic substances. The main parameters used are the heating rate (° C / min), the furnace atmosphere (inert or oxidizing gas) and the type of crucible used [27].

Differential Scanning Calorimetry (DSC)

DSC is a widely used technique, consisting of measuring the temperature and heat flow in a sample associated with controlled time and / or temperature variation. This analysis allows, for example, to verify the start, peak and completion temperature of the sample gelatinization and points out enthalpy variations with respect to a thermally inert reference material. The DSC curve is formed from peaks obtained from temperature variations onset (To), peak (Tp) and conclusion (Tc) of the event, in addition to the enthalpy (ΔH) of the event, which is identified by the peak area in relation to the baseline. Moreover, considering that enthalpy is a transition related to crystalline phase, it is directly correlated to the crystallinity degree. Physical and chemical changes are measured quantitatively and qualitatively through endothermic and exothermic processes [28].

X-Ray Diffraction (XRD)

The semicrystalline behavior of the starch granules varies with the type of plant and the processing conditions. X-ray diffraction is a method that distinguishes different types of starch patterns (A, B or C) [29].

The analysis is based on the principle of interaction between the incident X-ray beam and the electrons of the material to be studied. Diffracted photons are detected when the radiation hits the sample, and the phenomenon occurs in the scattering direction. This technique is used to study the differences between the semicrystalline behavior in starches and to identify traces of crystalline phases of the films [30].

The relative crystallinity (RC) % can be calculated according to Equation 1 [31]:

$$\mathrm{RC}(\%) = \frac{\mathrm{Ac}}{(\mathrm{Ac}+\mathrm{Aa})} \text{ x } 100 \qquad (1)$$

Where: RC = relative crystallinity, Ac = crystalline area and Aa = Amorphous area in the diffractogram.

The differences found in the relative crystallinities in the different sources can be attributed to the higher content of amylopectin, which is the molecule that presents greater granular structural organization [32].

Rapid Visco Analyzer (RVA)

Starch granules when heated to a specific temperature and sufficient water exhibit a behavior known as gelatinization. Acquiring high viscosity due to the swelling of the granules and gradual rupture of its crystalline structure. As it cools, a gel forms, due to the retrogradation of the starch [33].

The typical RVA profile of starches provides a plethora of information. The paste temperature is the temperature at which the equipment detects a measurable viscosity. Peak viscosity is the highest viscosity reached by the starch during the heating cycle, it corresponds to the maximum swelling of the granules without their rupture; the viscosity drop decreases after this peak due to the continuous shearing that occurs, where the highly swollen granules disintegrate. This variation between peak viscosity and minimum viscosity is known as breakage, it also gives information about the stability of the paste during the cooking process. During cooling, the chains begin to regroup, again increasing the viscosity (final viscosity). The difference between the final and minimum viscosity, called Setback, is associated with the tendency of starch retrogradation [33].

These starch paste properties are commonly monitored through rheological techniques on a Rapid Visco Analyzer (RVA) equipment, displaying the viscoamylographic profile of each starch.

Moisture, Solubility and Water Vapor Permeability in Biodegradable (WVP) Films

The moisture analysis allows to verify the amount of water present in the films, while the water solubility analysis indicates the behavior of the films in aqueous environments [17]. Water vapor permeability (WVP) is an important property evaluated in thermoplastics, as it allows to predict the water gain or loss of a product, since one of the main functions of food packaging is to prevent or reduce the transfer of moisture from the external environment to the inside of the package [34].

Traction Properties

The parameters most analyzed in tensile tests to assess the resistance of thermoplastics are tensile stress (TS), the elastic or Young's modulus and elongation at break [35].

The elastic or Young modulus evaluates the degree of stiffness of the material. It is equivalent to the ratio between tensile stress and deformation in the elastic region. It is a measurement of the mechanical effort to deform the sample: the higher the value of this parameter, the greater the material stiffness. The tensile stress, also called ultimate tensile strength, is the maximum tension with standed by a sample in an tension effort.

On the other hand, the elongation at break is a percentage ratio (%) between the elongation of the sample and its initial length in the moment of rupture. This parameter determines the point at which the film breaks under the tensile test and reflects the flexibility and stretching capacity of the material. The elongation can be also related to the material’s toughness because tough materials are capable of absorbing mechanical energy by deforming themselves. The higher the percentage of elongation at break, tougher the material [36].

The Table 2 shows, respectively, the values found before and after the addition of filler in the polymeric matrix of starch-based films.

Table 2. Values found before and after the addition of filler in the polymeric matrix of starch-based films

Starch + filler	Moisture (%)	Solubility (%)	WVP (g/msPa)	TS (MPa)	Reference
Regular maize + Eucalyptus	29.06-25.87	22.86-16.02	1.39-1.16	3.99-6.58	[15]
Waxy maize + Eucalyptus	27.54-24.70	32.85-27.02	1.42-1.11	2.06-2.42	[15]
Wheat + rice	27.03-24.43	37.87-30.71	4.74-2.81	2.65-3.67	[20]
Wheat + oat	27.03-16.03	37.87-31.42	4.74-2.42	2.65-5.07	[20]
Wheat + eucalyptus	27.03-20.81	37.87-34.57	4.74-2.54	2.65-4.32	[20]
Cassava + Bamboo	13.40-6.30	34.20-33.90	2.56-2.45	1.60-2.40	[17]
Cassava + Montmorillonite	13.40-6.10	34.20-31.30	2.56-2.60	1.60-2.10	[17]
Pea + waxy maize	38.74-23.17	Unrealized	11.18-4.26	5.76-9.96	[21]
Maize + sugar beet Pulp	Unrealized	Unrealized	4.73-3.00	21.90-28.87	[37]
Maize + starch nanoparticles	24.40-20.40	Unrealized	4.21-3.04	2.32-4.48	[6]
Banana + banana peels	15.90-14.50	32.30-23.90	10.70-3.50	7.30-11.10	[19]
Rice + rice straw	13.80-9.50	Unrealized	Unrealized	10.00-26.80	[38]

Rheological measurements of Film Forming Solutions (FFS)

Rheological measurements can be related to many attributes of the film. The flow properties depend upon materials structure and interactions. The viscosity is a measurement of flow resistance. It’s depended with shear stress reveals the material’s behavior in flowing situations: Newtonian, dilatant, or pseudoplastic. It is important the knowledge of this behavior for processability purposes [39,40].

Oscillatory properties like shear modulus are important to verify viscoelastic behavior of the materials, so that elastic and viscous fractions can be determined. The relations between storage modulus (related to elastic portion), loss modulus (related to viscous portion) and angular frequency of deformation consists in measurement of molar mass, distribution of molar mass, molecular interaction and formation of crosslinked structure, for example [39,41].

Applications of nanofiber films in food packaging

In recent decades, numerous studies have been done on biopolymers for food packaging applications. Food packaging must efficiently guarantee safety and preserve product quality, from production, handling, storage and, finally, reaching the consumer. The absence of the packaging or any damage in it, will cause the loss of the quality and safety of the food, leading to commercial losses and damage to the consumer [42].

According to research on packaging, bioplastic materials can be divided into groups, based on their origin. Starch, cellulose and proteins are a category of materials that fall into the group of polymers directly extracted or removed from biomass [43].

The mentioned polymers have the particularity of being hydrophilic and with a certain crystallinity, which can generate difficulties in production. One of the weaknesses when it comes to starch-based bioplastics is their poor performance when these packages are intended for food products with a high moisture content and poor mechanical properties [11].

On the other hand, they have an excellent gas barrier, optical and organoleptic capacity, properties that are suitable for application in packaging in the food industry [44].

To overcome the limitations found, several studies indicate that the addition of co-biopolymers or other secondary additives can improve the mechanical and tensile properties of the films, the addition of cellulose nanoparticles being very promising [11]. The Table 3 presents relevant studies of biodegradable packaging applications in foods.

Table 3. Relevant reports about the use of biodegradable films in foods

Polymeric matrix	Reinforcing agent	Applied food	Results	Reference
Rice starch	-	Plum fruit	Effective in reducing both weight loss and respiration rate	[45]
Chitosan–rice starch	Ag and ZnO nanoparticles	Peach fruit	Decreased the overall surface microbial load, enhanced the shelf-life and have lowest percentage loss in weight	[46]
Chitosan-quinoa protein	Chitosan thymol nanoparticles	Blueberries and cherry tomatoes	Films with high antimicrobial activity and effective in reducing water vapor permeability	[47]
Pectin	Cellulose Nanocrystals	Strawberries	The edible coatings were effective in minimizing of the weight loss, without worsening the physical chemistry attributes	[48]
Potato starch	Nano-SiO_2	White mushrooms	Remarkable preservation properties of the films packaging were obtained	[49]
Cassava starch	Sodium bentonite clay nanoparticles	Meatballs	Showed physical, mechanical and antibacterial potential	[50]
Montmorillonite clay-polyvinyl alcohol	Rice straws	Mangoes	Exhibited a shelf life extension, reduced mass loss, increased CO2 and low O2 level	[51]
Cellulose	-	Mangoes	The moisture vapor transmission rate and of gas transmission were higher and prolonged the storage and shelf life	[52]
Cassava starch	Zinc nanoparticles	Tomatoes	Lower oxygen permeability, hardness, elastic and plastic works	[53]
Corn starch	Chitosan nanoparticle na thymol	Cherry tomatoes	Films were efficient in maintaining the firmness and reducing the weight loss	[54]

The use of biodegradable films and edible coatings has emerged as an innovative and effective solution to extend the shelf life of fresh vegetables. These materials can extend post-harvest life by regulating gas exchange and decreasing moisture loss, they can also improve the quality of the fruit's texture and reduce the incidence of bruising on the skin during handling [45].

However, new materials for film production and coating from more compatible biopolymer need to be developed to overcome current limitations and maintain the post-harvest quality of fruits, such as improving permeability and tensile strength [45].

CONCLUSION

The development of biodegradable polymers can be an alternative to reduce discards generated in the environment by conventional plastics. Starch biopolymers reinforced with secondary additives in order to reinforce the polymer matrix has gained importance in research worldwide. The incorporation of an expanded additive in the use of these biopolymers may be related to the improvement in the general performance of the material, such as its mechanical, thermal and barrier properties. However, substitutions of the green type by oil derivatives are still very small, due to the scale of production, processing operations, stability and durability issues in relation to common plastics, combined or not with food. There is need for more research with greater focus on improving the performance of the material, reducing costs and improving the ease of production and applicability of biopolymers.

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