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(Research Article)

Physicochemical properties of potato starch films containing natural extracts of turmeric and hibiscus

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Abstract

The development of biodegradable films is an alternative to the use of environmentally unfriendly packaging. For that, several natural polymers can be used, such as starch, which has good characteristics like inherent biocompatibility, biodegradability, and easy availability. The functional performance of starch-based biodegradable materials can be extended or improved by adding antioxidant agents, as well as by using novel preparation techniques. The addition of natural antioxidants to the starch-based films can further contribute to the development of functional films. The objective of the present work was to develop starch-based active films added with hibiscus and turmeric extracts. Important properties of the filmogenic hydrogels and the films prepared by casting such as absorption and solubility in water and oxygen permeability rate were determined. Oxidative degradation can affect the inherent color, flavor, and microbial stability of a variety of foods, then the oxygen transfer rate (OTR) was also determined. The information learnt from this study will be of value for further developments employing natural polymers or biocomposites being used as primary packaging for foods. Present tendency is to create active or smart packaging materials and starch-based materials that will play a significant role from now on.

Keywords: Potato starch films; Plant extracts; Turmeric; Hibiscus

1. Introduction

A few natural polymers are being used as raw materials for constructing biodegradable materials, like proteins, polysaccharides (such as starch), and lipids. Starch is found in plants where it is the main form of fuel storage and is a major food supply for humanity, produced in seeds, rhizomes, roots, and tubers in the form of semi-crystalline granules with unique properties for each plant (Bertoft, 2017). Differences in properties are defined by differences in amylose and amylopectin structures and contents, granular organization, presence of lipids, proteins and minerals and starch granule size (Waterschoot et al, 2015).

Natural starch exists in the form of granules, which are spherical, oval, or irregular in shape, with diameters ranging from about 0.1 to 200 µm depending on the source and maturation stage. Starch granules are insoluble in cold water, but if they are heated above a certain temperature, they would absorb water and expand; the granules will continue to absorb water, and the crystalline regions will swell, eventually leading to the disruption of the starch granules and the formation of a hydrophilic colloidal solution (Liu et al., 2020). This process is known as the gelatinization of starch. After gelatinization, when a starch solution is cooled to a sufficiently low temperature, starch molecules reorganize, hydrogen bonds reform, and ordered structures are reestablished (Li et al., 2020). At the same time, the viscosity of starch suspension increases and gel forms. This process is known as retrogradation of starch.

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Starches differ significantly in composition, morphology, thermal, rheological and retrogradation properties. According to a compilation made by Hoover (2001), the characterization of potato starch is: yield 32%, granules size: 5-110 μm, granules shape: oval, spherical; amylose content: 25.4%, total lipid: 0.19%, organic phosphorus: 0.089%, inorganic phosphorus 0.001% and nitrogen 0.1%. Potato starch exhibits higher swelling power, solubility, paste clarity and viscosity than, for instance, wheat, rice, or corn starches. For that reason, potato starches are able to be used to prepared starch based biodegradable packaging films (Hirpara et al, 2021).

Hibiscus sabdariffa L., commonly known as roselle, red sorrel or karkade, is an annual herbaceous subshrub belonging to the family *Malvaceae,* is native to Africa and grows in tropical and sub-tropical regions such as Sudan, southern Asia, and America. Hibiscus plant produces edible calyx, belonging to a large family *Malvaceae* (Sharma et al., 2016). It is annual but can be cultivated as a perennial plant in the tropical and subtropical areas worldwide. In addition to the bast fiber and paper pulp or calyx, it also produces leaves and seeds (Osman et al., 2011). Fleshy calyces (sepals) are commercially important to produce beverages, juices, jams, and syrup in the food industry and are a good source of natural food colorants because of their high pigment content (Bridle & Timberlake, 1997). Hibiscus is rich in dietary fiber and possesses high amounts of bioactive compounds or phytochemicals including anthocyanins and carotenoids and a wide range of nonstarchy polysaccharides (Özlem Tokusoglu & Clifford Hall, 2011; Borrás-Linares et al., 2015).

The phenolic compounds found in this plant include organic and phenolic acids, such as citric acid, hydroxycitric acid, hibiscus acid, and protocatechuic acid. Flavonoids such as quercetin, luteolin or gossipetin, and their respective glycosides are also present. Anthocyanins, detected in high amounts in the calyces, are responsible for the bright red color. The most frequent anthocyanins of hibiscus flowers are cyanidin-3-glucoside, delphinidin-3-glucoside, cyanidin-3-sambubioside, and delphinidin-3-sambubioside (Ali et al., 2005). The phenolic compounds extracted from hibiscuses are used in diverse ways including antioxidants and hyperlipidemia and are also effective against low-density lipoprotein (Sayago-Ayerdi et al., 2007).

Turmeric comes from the root of Curcuma longa, a flowering plant of the ginger family. Curcumin, is a polyphenol compound derived from the traditional Chinese herb, turmeric, also called curcuma. Curcumin is the most active ingredient of their curcuminoids and has been shown to be safe and non-toxic in both pharmacological trials and in vivo experiments (Prasad et al., 2014); (Soleimani et al., 2018). Curcumin is a low molecular weight polyphenol, and has received increasing attention thanks to its extraordinary biological activities, including anti-inflammatory, anti-cancer, antiviral, anti-Alzheimer's activities (Aydogdu et al., 2020). In food package applications, curcumin is often utilized to produce intelligent packages (Yildiz et al., 2021) and give antimicrobial activity (Wang et al., 2019). Notwithstanding all those useful features, curcumin is a very reactive and unstable compound.

In food industries, curcumin has been widely studied due to its well-known bioactive, such as anticancer, antioxidant and anti-inflammatory activities (Liu et al., 2015; Sonkaew et al., 2012). Previous studies showed that biocomposite films containing curcumin have good antioxidant properties (Ma et al., 2017; Musso et al., 2017).

The objective of this work was to determine important properties of the hydrogels, based on the filmogenic solutions, for the preparation by casting of active films, made of a natural polymer, potato starch, and natural rich antioxidant extracts such as turmeric (or curcuma) and hibiscus to develop active films. The rheology of the hydrogels, the water absorption and water solubility index as well as the oxygen gas transmission rate of the films were determined.

2. Material and methods

2.1. Material

Ingredients were obtained from the local food market.

Potato starch trade mark Yoki (that actually comes from a multi-step process where only the starch is extracted from a potato).

Hibiscus sabdariffa powder (sometimes known as roselle hibiscus, is a *Malvaceae* family shrub).

Turmeric powder comes from the ground-up roots of the *Curcuma longa* plant, that is dried and ground down into a powder sold as spice. Curcumin is the active component of the turmeric spice.

2.2. Extraction of polyphenols

The extraction of the bioactive compounds was carried out in ultrasonic bath for 10 minutes, in the proportion of 1:10 (w/v) sample: solvent. The solvent used for the extraction of hibiscus was methanol/water 80/20 and for the extraction of turmeric it was methanol. Both extractions took place at 85 °C. Methanol was from Sigma-Aldrich.

2.3. Preparation of hydrogels

The process of obtaining the films was carried out by homogenizing in aqueous solution at concentration of 5% of starch (w/v), 3% of glycerol (w/v), 0.5% of propionate of calcium under constant stirring until starch gelatinization. For the formulation of active films, the same formulation was prepared with the addition of 1% and 2% the solution of hibiscus extract and turmeric.

The hydrogel formed was kept at room temperature for 1h. Then, 80g were poured into acrylic plates and these were subjected to drying in a chamber with air circulation under controlled temperature (25°C) and relative humidity (50%), for a period of 72 hours or until the films dry.

2.4. Determination of paste properties

The paste properties of the starch hydrogels were determined in a Rapid Visco Analyzer device (model RVA-4500 from Perten Instuments, Warriewood, Australia), using Thermocline for Windows version 3. The Rapid Visco Analyser (RVA) is a heating and cooling viscometer that measures the viscosity of a sample over a given period while it is stirred (Balet et al., 2019). Each sample was analyzed in triplicate. Using the following parameters: temperature/time- 50 °C/1 min., heating of 50 °C at 95 °C at a rate of 6 °C.min-1, constant at 95 °C/2 min and 30 sec and cooling from 95 °C to 50 °C at a rate of 6 °C.min-1. The parameters used to interpret the results were: Paste temperature; Maximum or peak viscosity time; Peak or peak viscosity; Minimum viscosity; Final viscosity; Breakdown or break; setback.

2.5. Water absorption and water solubility index

The determination of water-soluble fraction of the films was performed according to the methodology described by Gontard et al. (1992), in which a sample of known initial dry mass measuring 2 cm in diameter was immersed in 50 mL of distilled water at 25 °C for 24 hours, under agitation. Afterwards, the insoluble films were subjected to drying in an oven at 105 °C for 24 hours to determine the final dry mass of the sample. The water-soluble fraction was calculated according to the equation 1.

$$S(\%) = \frac{(mi-mf)}{mi} x100 \ldots\ldots\ldots\ldots\ldots \quad (1)$$

Were, S: water-soluble fraction (%), mi: initial dry mass of the sample (g), mf: final dry mass of the sample (g).

The water absorption capacity of the films was performed using the method described by Chiono et al. (2008). Film samples were cut into squares (10x10 mm) and weighed (mi). Then, they were placed in distilled water and stored at room temperature for 24h. Afterwards, the swollen films were removed from the water, dried with absorbent paper, and weighed (mf). Each assay was performed in triplicate. Thus, the film absorption calculation (A) was performed according to equation 2.

$$A(\%) = \frac{(mf-mi)}{mi} x100 \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Where, A=Water absorption in (%); mf= final sample mass and mi= initial mass.

2.6. Determination of Oxygen Transmission Rate

OTR (oxygen transmission rate) is the steady state rate at which oxygen gas permeates through a film at specified conditions of temperature and relative humidity. An indispensable element for people, oxygen is a major cause of the reactions associated with food spoilage. The Standard Test Method following the norm ASTM F1927-20 was used, permeability and permeance at controlled relative humidity through barrier materials using a coulometric detector. Assays were performed at 23 °C and 50% RH. The effective permeation area of each specimen was 50cm^2. The results obtained were corrected for 1 atm oxygen partial pressure gradient between the two surfaces of the film. This gradient corresponds to the driving force for oxygen permeation through the film. From the oxygen permeability rate, the oxygen permeability coefficient (P'O_2) was calculated follows:

$$PO_2 = \frac{TP\prime O_2 xe}{p} \text{………….(3)}$$

Where, $P'O_2$ = oxygen permeability coefficient (mL (CNTP). μm. m^{-2}.day^{-1}.atm^{-1}), $TP'O_2$ = oxygen permeability rate (mL (CNTP). m^{-2}.day^{-1}), e = average thickness of the specimen (μm), p= partial pressure of oxygen in the permeant gas chamber of the diffusion cell since the partial pressure of O_2 in the carrier gas chamber ($N_2 + H_2$) is considered null.

3. Results and discussion

3.1. Paste properties

The profiles of the RVA viscograms of the hydrogels for the preparation of the films are shown in Fig.1. It is observed that with the addition of antioxidant extracts, the viscosity of starch hydrogels increased. This suggests that these agents chemically interacted with the starch polymer matrix. The hydrogel with the addition of turmeric antioxidants had the highest viscosity value (703 cP).

The paste properties of starches are influenced by the size, stiffness, amylose, and amylopectin ratio and swelling power of the granules (Singh et al., 2007). Peak viscosity measures the water absorption capacity of the starch in terms of the strength of the swollen granules under shear and the performance of these granules. These results indicated that potato starch without or with the addition of plant extracts present different properties and the application of these formulations will depend on the convenience for the application in different food preparations.

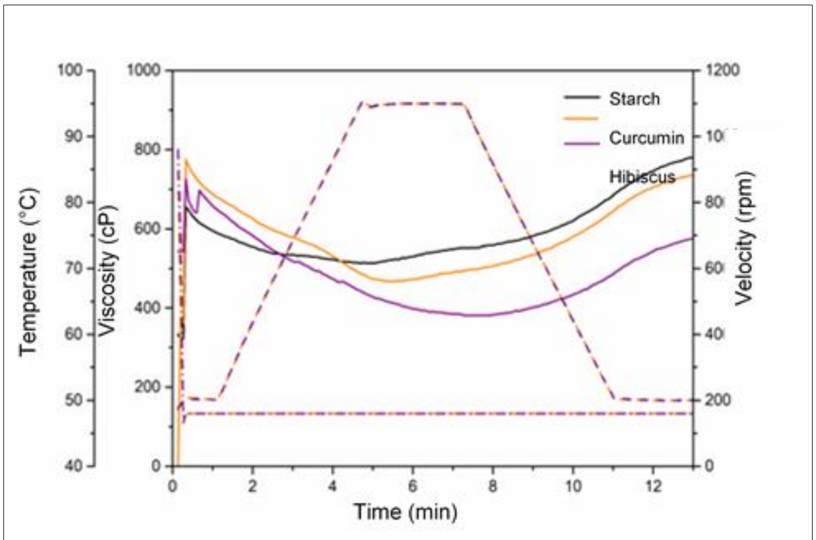

Figure 1 Viscosity profiles of potato starch hydrogels with and without turmeric (curcumin) and hibiscus extracts

The difference between the peak viscosity and minimum viscosity values defines the breakdown, thermal stability index of the starch under shear. Breakdown means the viscosity breakdown that occurs in a starch gel after going through the heating cycle. After gelatinization, the temperature of the gel is kept constant for a few minutes and the shear of the analyzer blade acting on the sample under test ends up disintegrating the starch granules and promoting the reduction (breaking) of the sample viscosity.

The Setback value is most related to the retrogradation tendency of the amylose of the starch granules (Huang et al., 2010). The temperature of the gel reduces, and, with this, the amylose retrogrades, and the starch granules reorganize. The hydrogel with highest setback was the one with turmeric added with 263 cP and the lowest with 203 cP, for the hydrogel with the addition of hibiscus. With the addition of the extracts, the gels had higher peak viscosity and higher breakdown compared to the control (Tab. 1).

Table 1 Paste properties of starch hydrogels added with hibiscus or turmeric (curcumin) extracts 1%

Hidrogels	Peak viscosity (cP)	Min. viscosity (cP)	Breakdown (cP)	Final Viscosity (cP)	Setback (cP)
Potato starch (PS)	532	510	22	750	240
PS+hibiscus	682	398	284	601	203
PS+curcumin	703	484	219	747	263

According to Gerçekaslan (2021) peak viscosity is an important characteristic to distinguish properties of starches, and an indication of water holding capacity. Also, high crystallinity is associated to lower peak viscosity. As the starch granules swell, the viscosity of the system increases, and when the number of swollen granules reaches the highest level, the viscosity reaches the peak. This indicates that physical or chemical modification significantly increases the swelling ability of the starch granules.

3.2. Water absorption, water solubility index and oxygen gas transmission rate of films

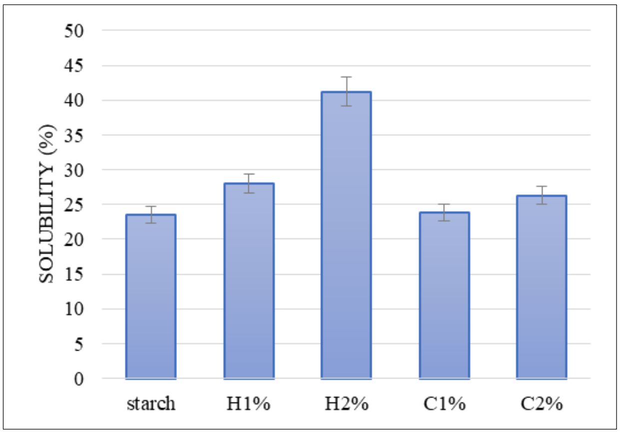

Figure 2 Solubility of films with hibiscus and curcumin extracts

Figure 2 and 3 shows that the water solubility and absorption of films with 1 and 2% of extract. It was observed the water solubility and absorption increase with added of the extracts. Potato starch films increase their solubility when hibiscus extracts were present, but no modification was found when curcumin extract was added. Wang et al. (2007), studying potato starch films added with 1:2 glycerol (glycerol/starch), found similar solubility values.

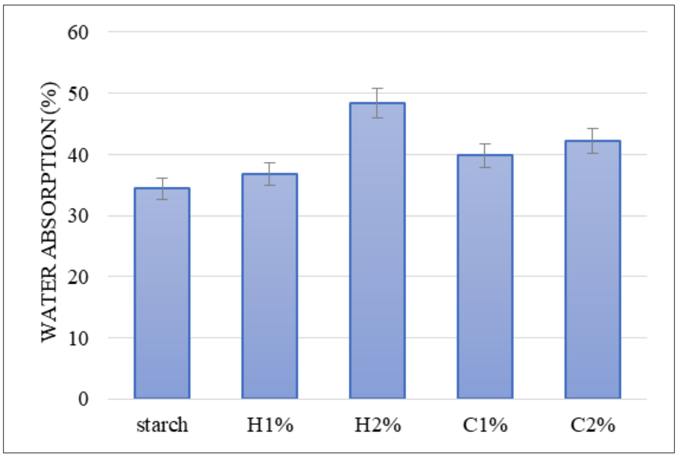

Figure 3 Water absorption of films with hibiscus and curcumin extracts

Water solubility indicates the films hydrophilicity. The determination of WS is useful for choosing a film for specific applications (Salarbashi et al., 2019). For instance, low WS is required to protect food from moisture absorption. However, in other cases, like the encapsulation of phytochemicals, WS's high value may be preferred (Liu et al., 2019).

The results of the oxygen permeability rate of starch-based films are shown in Fig.4. The oxygen permeability rate is, by definition, a permeant flux that passes through an area of the material, due to the difference in the partial pressure of the permeant between the surfaces (Moreira, 2007). Oxygen to permeate must open space between the matrix, because it is a more nonpolar vapor, it needs a larger free volume between the chains and a lower cohesive density. All films can be considered as moderate transmitter. Nevertheless, the presence of the hibiscus extract reduced notoriously the transmission, increasing their barrier capability. This behavior was no evident in the films containing curcumin extracts.

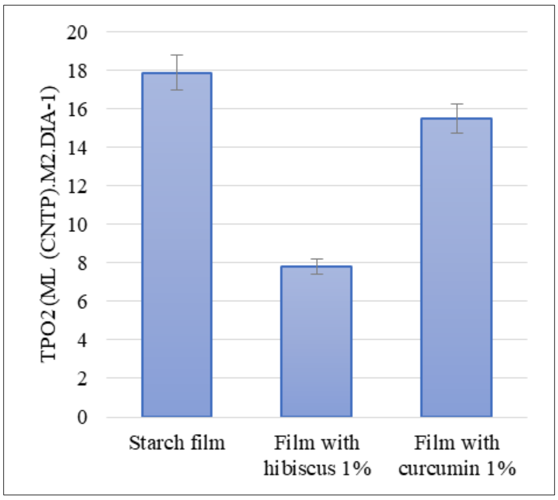

Figure 4 Oxygen gas transmission rate the film with extracts of 1%

The presence of hydrophilic functional groups in the backbone of polysaccharides facilitates hydrogen bonding between polysaccharides and water molecules, which in turn increases WS and WVP of polysaccharides-based films. For starch films, there was a slight tendency to increase water absorption with the addition of hibiscus and with the curcumin extracts.

An indispensable element for people, oxygen is a major cause of the reactions associated with food spoilage. Oxidative degradation can affect the delicate color, flavor, and microbial stability of a variety of foods such as coffee & tea, nuts, chocolate, cheese, meat, gourmet snacks, and retorted products. Usually, a general comparisons of barrier capabilities are as follows:

Food products are prone to be oxidized during processing, transport, and preservation. The oxygen barrier property of the films is also important for maintaining the quality of food products. Wang et al. have reported that the OP value of the chitosan/EGCG films reinforced with nano-bacterial cellulose is from 3 to 4 × 10^{-3} g/m s Pa (Wang et al., 2018). For both the 3% and 5% PP films, addition of BC (0–10%) gradually decreased the OP due to a more indirect path for transmission of oxygen molecules. Just as a complement, in Table 2 appeared compilated data from the literature about definition of relative oxygen barrier capability of materials.

Table 2 Range of Relative Oxygen Barrier Capabilities in terms of their units

Relative Oxygen Barrier Capability	**cc/m²/24h**	**cc/100 in²/24h**
High-Barrier	~ 1-10	~ 0.06-0.65
Moderate Transmitter (of O_2)	~ 1000	~ 64.50
High Transmitter (of O_2)	~ 10,000	~ 645.20

4. Conclusion

The presence of the plant extracts rich in flavonoids, such as hibiscus, turmeric or curcuma, alter substantially the properties of the potato starch films or hydrogels. Bioactive ingredients are the most promising substitute for synthetic chemical agents. Paste properties of the hydrogels made of potato starch added or not the plant extracts present differences in terms of Maximum or peak viscosity time; Peak or peak viscosity; Minimum viscosity; Final viscosity;

Breakdown or break; setback. The differences reflected the chemical differences of the formulations. Also, water absorption and water solubility present substantial differences. The desired solubility for a film is directly linked to the destination of its application, so turmeric embedded films could act as protection for foods with high water activity, as their solubilities are lower compared to hibiscus added films. In terms of the relative oxygen barrier capability, the presence of the hibiscus extract reduced notoriously the oxygen transmission of the films, increasing their barrier capability.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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