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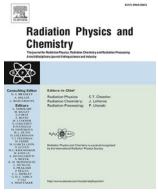

Effects of electron beam irradiation on ozone-modified potato starch film

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A R T I C L E I N F O

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A B S T R A C T

Functional starch-based films are promising materials being developed at different laboratories. Native and modified starches are two materials being employed for this purpose. Chemical modification of starch generally involves esterification, etherification, or oxidation of the units that make up the starch polymers. Ozone constitutes an advanced oxidation technology for starch modification. In this work, properties of two kinds of materials - ozone-modified potato starch and irradiated ozone-modified potato starch-based films - were evaluated. Potato starch films were submitted to electron beam irradiation with doses up to 40 kGy. Films were produced by the casting technique using native and ozonated potato starch, glycerol as the plasticizer, and water as the solvent, and characterized in term of solubility and absorption of water, determination of oxygen gas transmission rate and Fourier Transform infrared Spectroscopy (FTIR) analysis. Ozone-modified potato starch presented strong alterations on pasting properties, with drastic reduction of viscosity values, water solubility and oxygen permeability. The data reported in this work contribute to the understanding of the ozone-modification process on starch and suggests further possibilities of industrial applications of ozonation on biomaterials.

1. Introduction

The development of green materials using natural polymers has increased in the last decades, because of their benefits of being more environment-friendly. Starch is a proper low-cost substitute for polymers extracted from petroleum derivatives. It is the most abundant organic compound found in nature (after cellulose), and is found in immense quantity in plants, where it is the main form of fuel storage. Starch occurs in two forms: alpha amylose (long unbranched chains) and amylopectin (highly branched). Normal starch from maize, rice, wheat and potato, contains about 70–80% amylopectin and 20–30% amylose (Perez and Bertoft, 2010). The ultimate material properties of starch composites will depend on several factors: starch source (starches from maize, pea and potato are normal-amylose and high-amylopectin), extraction method, film formulation, processing methods, and curing procedures.

Starch has been extensively used in non-edible or edible preparations (Whistler et al., 1884), and its derivatives are widely used to provide functional properties such as gelling, thickening, bonding and adhesion. Native starches are often modified to develop specific properties such as solubility, texture, adhesion and tolerance to the heating temperatures used in industrial processes (Alcázar-Alay and Meireles, 2015). Chemical modification of starch generally involves esterification, etherification, or oxidation of the units that make up the starch polymers.

Starch food packaging, i.e., starch-based coatings and films, is a more recent area of interest (Mary et al., 2020; Zhao et al., 2021; Díaz-Montes and Castro-Muñoz, 2021). According to Bangar et al. (2021) the functionality of starch-based biodegradable substances is still technically a challenge. It can be improved by starch modification, blending starch with other biopolymers or additives, and using novel preparation techniques.

Ozone is a very powerful oxidizing agent, and it can be quickly decomposed into oxygen, leaving no residues, and meeting the global demand for sustainability. Ozone constitutes already an advanced oxidation technology for starch modification (Pandiselvam et al., 2019), although the effects of oxidant differentiation by ozone gas on some physicochemical and functional properties of starches is starting to be elucidated (Castanha et al., 2019; La Fuente et al., 2020).

Irradiation of natural or synthetic polymeric materials with ionizing radiation (gamma rays, X rays or accelerated electrons) produces oxidized products, grafts, scission of main chains (degradation) and crosslinking. Starch when irradiated with ionizing radiation is observed to produce free radicals, generate sugars owing to cleavage of amylopectin branches, and exhibit variation in enzymatic digestion, amylose content, morphology, crystallinity, thermal property, and chemical

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composition. Numerous publications ratify that ionizing radiation, in a dose-dependent way, is able to diminish starch viscosity because it provokes depolymerization or cross-linking/grafting reactions to the starch medium (Bhat and Karim, 2009; Uehara et al., 2009; Govindaraju et al., 2022; Rostamabadi et al., 2023).

Ionizing radiation is used mainly to kill microorganisms, but can also be employed for the preparation of new materials (Kharisov et al., 2013; Barroso and Del Mastro, 2019; Ashfaq et al., 2020; Nguyen et al., 2021; Teixeira et al., 2021).

The use of ozone modified potato starch can be considered a novelty in the radiation preparation of edible films to be used as primary packaging.

The present work aims at: i. Evaluating how the ozone treatment affects some properties of potato starch, mainly paste properties; ii. Establishing how electron beam irradiation affects properties of films prepared with previously ozone modified potato starch. The data reported in this work will contribute to the understanding of the ozone-modification process on starch presenting further possibilities of industrial applications of ozonated biomaterials.

2. Material and methods

2.1. Material

Native potato starch was obtained in bulk from a local retail food market, so no precise amylose or moisture content were available. We estimated a moisture content in the range of 11–15% and apparent potato starch content in the range of 28.8–39.4% (Pineda-Gomez et al., 2021). Also, Glycerol P.A grade (Sigma-Aldrich, Brazil), milliQ water and propionate of calcium were employed.

2.2. Starch modification (ozone treatment)

The modification of potato starch with ozone was carried out at a local startup *Brasil Ozônio Laboratory* (CIETEC/IPEN) facility, according to the methodology proposed by Castanha et al. (2019) with some modifications. The ozone was produced by an ozone generator (BRO3 brand, PLUS1.2 model – benchtop ozone generator), fed by an oxygen concentrator (LUMIAR healthcare brand), generating ozone gas from the corona effect (Fig. 1). The system consists of a dielectric, in which oxygen is inserted, and through a constant electrical discharge, generated by an electrical system, the oxygen molecule (O_2) is broken, generating ozone (O_3). The ozone-rich gas was directed to an Erlenmeyer with starch suspensions (10% w/w), prepared with milliQ water.

The gas flow in the reactor was kept constant at 2.0 L min^{-1} and the gas concentration was kept at 6 × 10^3 mg O3. L^{-1} for 5 min. The native starch sample was the control. The suspension was sedimented and the supernatant liquid was discarded. The starch was dried in an oven at room temperature (~25 °C). After 24 h, starch samples were ground with a pestle and mortar, and stored at 25 °C in closed glass containers for further analysis.

A B

Fig. 1. (A) Oxygen concentrator and (B) BRO_3/Ozone generator.

2.3. Preparation of starch films

The process of obtaining the films was carried out by dispersing native or ozone-modified starch in distilled water, 5% of starch (w/v), 3% of glycerol (w/v), 0.5% of propionate of calcium heating under constant stirring at 85 °C until starch gelatinization. The films were produced by casting technique: 80 g of the starch-containing mixtures were poured into acrylic plates and these were dried in a chamber with air circulation under controlled temperature (25 °C) and relative humidity (50%), for a period of 72 h.

2.4. Determination of paste properties

The paste properties of the native and ozone-modified potato starch were determined in a Rapid Visco Analyzer device (model RVA-4500 from Perten Instuments, Warriewood, Australia), using Thermocline for Windows version 3. Starch concentration was 5% w/v and each sample was analyzed in duplicate, 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 s and cooling from 95 °C to 50 °C at a rate of 6 °C.min-^{1}. Viscosity was expressed in centiPoise (cP). The parameters used to interpret the results were: Maximum or peak viscosity time; Peak or peak viscosity; Minimum viscosity; Final viscosity; Breakdown or break; setback.

2.5. Irradiation

The samples of native or the ozone-modified potato starch films contained in acrylic plates (4 plates for each determination) were subjected to electron beam irradiation (EBI) process using a Dynamitron II electron accelerator, Radiation Dynamics Inc. at room temperature with doses of 5, 10, 20 and 40 kGy. The parameters were: energy 1.202 MeV, beam current 0.62 mA, dose rate 2.81 kGy s-^{1}, tray speed of 6.72 m min^{-1}, dose related per passage 5 kGy. For each treatment, a non-irradiated sample was maintained as a reference (0 kGy). After irradiation, the samples were stored in a desiccator until analysis. Dosimetry was performed using alanine dosimeters calibrated according to the international standard ISO/ASTM l51607 (2004).

2.6. Films characterization

2.6.1. Solubility in water and water absorption

The percent of solubility was the percentage of dry matter of the film solubilized after 24 h immersion in water. The percentage of initial dry matter of each film was determined at 105 °C for 24 h. Two discs of film (2 cm diameter) were cut, weighed, immersed in 50 mL of distilled water and slowly and periodically agitated for 24 h at 25 °C. The pieces of film were then taken out and dried (105 °C for 24 h) to determine the weight of dry matter which was not solubilized in water. The weight of dry matter solubilized was calculated by subtracting the weight of dry matter not solubilized from the weight of initial dry matter and reported as initial dry weight basis. The water-soluble fraction was according to the equation below:

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

Wherein: *mi* and *mf* are the initial and the final discs' mass (g), respectively.

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

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

2.6.2. Determination of oxygen gas 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 50 cm^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:

$$PO_2 = \frac{TP'O_2 xe}{p} \quad (3)$$

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. This test was performed with control and 40 kGy samples.

2.6.3. Fourier Transform infrared spectroscopy (FTIR) analysis

Irradiated and unirradiated starch films were submitted to FTIR analysis. A PerkinElmer Spectrum One FTIR, equipped with the Universal ATR Sampling Accessory (ZnSe cell), was used to obtain 4 cm^{-1} resolution spectra in the 400 - 4000 cm^{-1} region, scanned 25 times (Absorbance vs. wavenumbers mode) at room temperature and humidity. The FTIR data shown in the figures were shifted onto the vertical axis to permit convenient comparisons.

3. Results

3.1. Paste properties

Key rheological properties of starches include pasting property, viscosity of starch paste, and rheological features of the starch gels. In the present work, pasting properties of ozone-modified and native potato starches were investigated. Data from the Rapid Visco Analyzer (RVA) are shown in Table 1.

Paste properties of ozone-modified starch showed lower viscosity than the native starch: the viscosity values (peak viscosity, minimum viscosity, and final viscosity) suggest little resistance to pasting. Chan et al. (2009) had already reported that the intrinsic viscosity of oxidized starches decreased significantly, and the extent of starch oxidation by ozone varied among different types of starch.

Viscosity reduction found in this work can be attribute to weakened hydrogen bonding between the starch molecules and partial depolymerization of starch molecules by ozonation (Çatal and İbanoğlu, 2014). Present results are also in agreement with those from Klein et al. (2014): they considered that ozone oxidation of cassava starch in aqueous solution was able to reduce the crystallinity, promoting strongest alterations on pasting properties.

Table 1
Paste properties (RVA) of hydrogels of native and ozone-modified starches.

Hydrogels	Viscosity (cP)	Minimal Viscosity (cP)	Breakdown (cP)	Final viscosity (cP)	Setback (cP)
Native Potato starch	532	510	22	750	240
Modified potato starch	140	117	23	163	46

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

Starch processing by radiation leads to the formation of radiolytic products and some radiation degradation can be expected as ionizing radiation acts on polysaccharides by the abstraction of H atoms that can lead to the direct scission of the backbone (Ashfaq et al., 2020; Nguyen et al., 2021).

Fig. 2 displays the water solubility of films prepared with native and ozone-modified starches as a function of electron beam irradiation (EBI) dose. Films made of native potato starch present, as expected, water solubility increasing with the increase of the irradiation dose. In the case of ozone-modified starch, water solubility presented another pattern of behavior: up to 10 kGy presented higher solubility than the native starch and a pronounced reduction of solubility at 20 kGy. In an already affected structure by ozone treatment, it can be expected that the action of irradiation presents some differences, although the explanation for this fact remains to be investigated. Predominance of one or another radiation-induced phenomenon - scission of main chains (degradation) or crosslinking - at certain level of energy absorption from the irradiation process could be considered.

Water absorption of the potato starch films is displayed in Fig. 3. Oxidized starch obtained by ozone treatment showed a reduction in water absorption capability in comparison to native samples prepared with native starch. In the case of the sample irradiated at 20 kGy an unexpected behavior was also found, such as in the case of solubility discussed below.

Table 2 displays the results of the oxygen permeability essays of EB irradiated potato starch films with doses of 0 and 40 kGy. The highest dose was chosen to point out just important differences.

For native starch films, there was no significant difference between doses of 0 and 40 kGy in terms of oxygen permeability. On the other hand, for the previously ozone-modified starch films, the oxygen permeability of the control (0 kGy) was much higher (20.34) and the irradiated with 40 kGy presented a notorious decline (16.59). As mentioned before, the effects of oxidant differentiation by ozone gas on functional properties of starches is not yet completely elucidated. Castanha et al. (2019) considered, that similarly to for RVA analysis, that difference in behavior is probably due to the fact that the native starch granules are intact: the bonds of the molecules are undamaged and their hydroxyl groups were not replaced by electronegative groups, bonding strongly to each other. Thus, their molecules are more tightly bonded, which hinders their solubility in water if compared to the ozonated starches. Also, the exact action of ionizing radiation onto the ozone already modified starch is still to be elucidated.

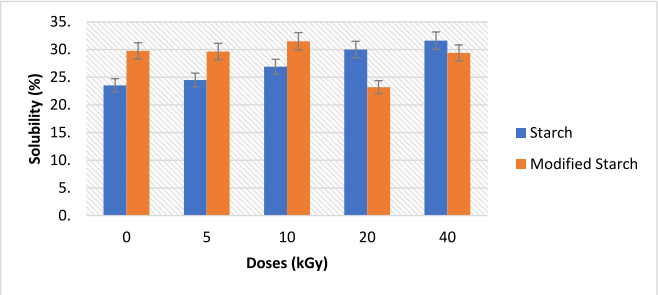

Fig. 2. Water solubility % of films of native and ozone-modified starch as a function of irradiation dose.

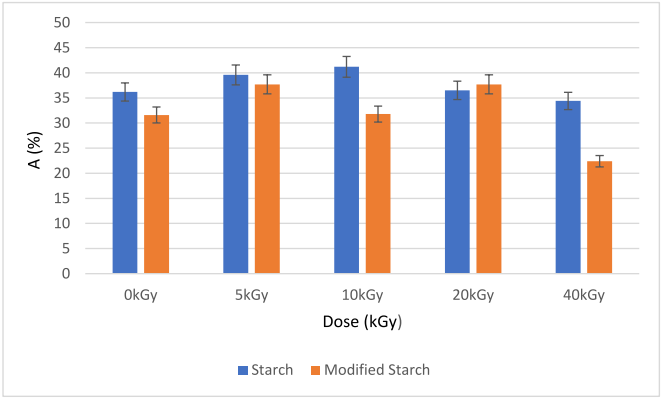

Fig. 3. Capability of water absorption of native potato and ozone-modified starch films.

Table 2
Oxygen permeability coefficient of irradiated films with doses of 0 and 40 kGy.

Irradiation Dose	TPO_2(ml CNTP) $m^{-2}day^{-1}$
Native starch 0 kGy	17.9 ± 0.2
Native starch 40 kGy	18.0 ± 0.1
Modified starch 0 kGy	20.3 ± 0.2
Modified starch 40 kGy	16.6 ± 0.2

High oxygen permeability is desired for films used in the packaging of fresh red meats, for example, as oxygen is responsible for the formation of oxymyoglobin, the pigment that gives the product its characteristic color. For foods rich in lipids, packaging with low oxygen permeability is desired, as oxygen leads to the formation of free radicals and the development of rancidity.

3.3. Fourier-transform infrared spectroscopy (FTIR)

Spectra of native and ozone-modified potato starch films were developed by using Fourier-transform infrared (FTIR) spectroscopy to characterize ordered structure (Warren et al., 2016). To identify functional groups, wavelengths from 3500 to 1500 cm^{-1} were used. Regions between 600 and 1500 cm^{-1} are referred to as the fingerprint. The functional groups identified from the FTIR of the native and ozone modified potato starch are presented in Table 3. The major functional groups were alcohol, alkane, and alkene. FTIR spectra of starches are presented in Fig. 4 whereas the interpretation of each peak is given in Table 3. The presence of absorption band at around 3396, ~29260, ~1650 cm^{-1} in the spectra indicated that both starches possess an OH, C–H, C–O–C, and C–O functional group, respectively. In addition, the characteristic C–O–C ring vibration on starch lead to an absorbance peak at around 700-900 cm^{-1}.

No detectable modification of peak position or peak intensity was evident for the potato starch molecules upon the ozone treatment.

4. Conclusion

Present results indicate that potato starch processed with ozone, a green technology, presented some modifications at molecular level resulting in significant differences with native starch mainly in terms of pasting properties, finding obtained also by others. Pasting properties of ozone-modified starch were drastically reduced. When ozone-modified starch was used to prepared edible films using also electron beam irradiation, those made of native potato starch present, as expected, water solubility increased with the increase of the irradiation dose. In the case of ozone-modified starch films, water solubility presented another pattern of behavior: up to 10 kGy presented higher solubility than those made of the native starch and a pronounced reduction of solubility at 20 kGy that remained at 40 kGy. Starch treated previously by ozone to develop edible films using electron beam irradiation had not been reported until the present work, so more detailed analysis is required. The observed properties can be useful for different technological applications, especially regarding processes/products that demand specific characteristics. Present results signalize a good trend of research to be followed.

Table 3
FTIR spectrum of the potato starches.

	Frequency(cm^{-1})	Absorbing Feature	Compound class	Intensity
1.	3396.00	O–H stretch	Alcohol	Strong, broad
2.	2926.30	C–H stretch	Alkane	Weak
3.	1651.00	C = C stretch	Alkene	Weak

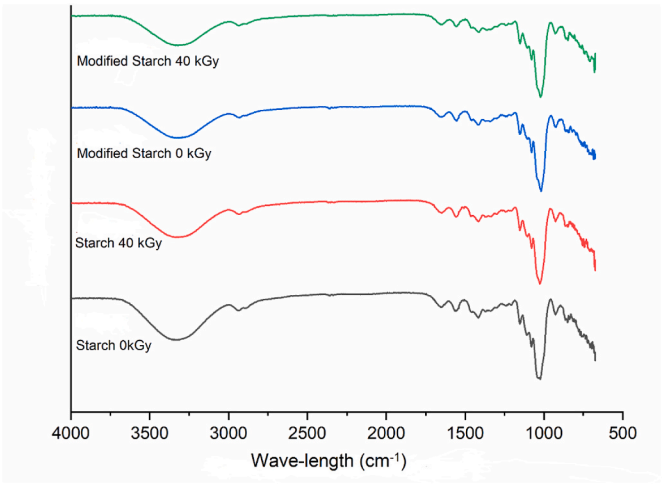

Fig. 4. FTIR spectra of native and ozone modified potato starches films irradiated with 0 and 40 kGy.

Author statement

None.

Declaration of competing interest

The authors declare that there is no conflict of interests regarding the publication of this paper.

Data availability

Data will be made available on request.

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