



## Starch-cashew tree gum nanocomposite films and their application for coating cashew nuts



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### ABSTRACT

Polysaccharides have been used in several studies to form films and coatings to be used as packaging materials (or to complement packaging materials) for foods. The incorporation of nanofillers such as montmorillonite (MMT) has been suggested to improve the barrier and mechanical properties of bio-materials. Films from starch and cashew tree gum (CTG) were developed according to a central composite design with two variables: MMT concentration and sonication time. MMT concentrations of 5–10 g/100 g increased tensile strength and elastic modulus, while higher MMT concentrations impaired those properties. The moisture barrier has been favored by MMT, while transparency and elongation have been impaired by the nanofiller. Sonication favored strength and modulus, probably because of a better MMT dispersion. Starch-CTG coatings with or without MMT were applied to cashew nut kernels. Coated kernels presented reduced moisture absorption during 120 days of storage (especially those coated with the nanocomposite coating), indicating that the coatings could decrease the texture changes of kernels. Moreover, the oxidation rates during storage were similarly decreased by both coatings.

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### 1. Introduction

Cashew nut, one of the most important edible nuts in international trade, is the fruit of cashew trees (*Anacardium occidentale* L.), and its kernel is the most widely industrialized cashew product. Since lipids constitute more than 40 g/100 g of cashew nut kernels (Chandrasekara & Shahidi, 2011), lipid oxidation is their main cause of deterioration, due to the formation of off-flavors (oxidative rancidity), impairing their acceptability. Moreover, the kernels are also susceptible to moisture absorption, resulting in texture changes and loss of their characteristic crispness. The application of coatings to cashew nut kernels could significantly increase their

stability, avoiding the necessity of using high barrier packaging materials (more expensive and usually thicker). Moreover, sometimes the kernels are sold in bulk, not protected by an external package while exposed to be viewed by consumers, and this is when the presence of a protective coating would be especially useful.

Cashew tree gum (CTG) is a water soluble heteropolysaccharide exuded from cashew tree (*A. occidentale* L.), whose composition has been previously reported (De Paula, Heatley, & Budd, 1998), and which has been used by Carneiro-da-Cunha et al. (2009) as coating for apples. However, the branched structure and polyanionic character of CTG result in solutions with very low viscosity (10 cPs for a 1 g/100 mL CTG solution at 25 °C, according to Gyedu-Akoto et al., 2007). An adequate viscosity for film formation usually requires CTG concentrations of at least 40 g/100 mL, or a combination of CTG with another polysaccharide with a higher viscosity, such as alginate (Azeredo et al., 2012).

Polysaccharide-based films and coatings usually have poor mechanical and barrier properties when compared to petroleum-

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based polymers. The use of nanoreinforcements, such as montmorillonite-type nanoclays (MMT), has been extensively studied to improve the physico-mechanical performance of biopolymer materials. MMT is formed by platelets which have two layers of tetrahedral silica sheets filled with a central octahedral alumina sheet (Weiss, Takhistov, & McClements, 2006). Its negative surface charge defines the interlayer spacing (Alexandre & Dubois, 2000), the imbalance of negative charges being compensated by exchangeable cations. Parallel layers are linked together by weak electrostatic forces (Tan, Zhang, Szeto, & Liao, 2008). MMT is an excellent reinforcing filler, thanks to its high surface area and large aspect ratio (Uyama et al., 2003). Moreover, the addition of MMT to polymer matrices increases the tortuosity of the diffusive path for permeants, forcing them to travel a longer path to diffuse through the matrix (Adame & Beall, 2009; Mirzadeh & Kokabi, 2007). Although MMT is not yet approved for food applications, it is generally recognized as a low toxicity material (Lee et al., 2005; Wang, Du, & Luo, 2008), and is commonly used in pharmaceutical products either as excipient or active ingredients (Wang et al., 2008). Recently, MMT has been studied as component of edible coatings (Azeredo, Miranda, Ribeiro, Rosa, & Nascimento, 2012; Cortez-Vega, Pizato, Souza, & Prentice, 2014) or films (Slavutsky, Bertuzzi, Armada, García, & Ochoa, 2014). MMT dispersion can be improved by the use of shear devices such as sonicators, which create sound waves that propagate into water and generate alternating pressure cycles, which overcome the bonding forces, breaking agglomerates and increasing interlayer distance (Vartiainen, Tuominen, & Nättinen, 2010).

The objectives of this study were to develop and characterize films from starch and cashew tree gum added with montmorillonite, and to test their application as coatings to increase stability of cashew nut kernels.

## 2. Materials and methods

### 2.1. Elaboration and characterization of films

Cashew tree gum (CTG) was obtained from exudates from cashew trees (Embrapa Tropical Agroindustry, Experimental Station at Pacajus, CE, Brazil) and purified by a method adapted from that described by Torquato et al. (2004). The exudate was ground, dissolved in distilled water at room temperature ( $24\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ), vacuum filtered, and precipitated with commercial 96°GL ethanol (ethanol:exudate ratio, 3 g: 1 g). The precipitate was drained and dried on Petri dishes in a fume hood. The dried gum was ground by using an analytical mill (A11 Basic, Ika, Staufen, Germany) and passed through a 212  $\mu\text{m}$  sieve.

The experiment was conducted according to a central composite design, carried out on eleven randomized runs from Table 1, with

**Table 1**  
Independent variables and their real (uncoded) values according to the central composite design.

Treatment	MMT (g/100 g)	Sonication time (min:s)
1	2.18	00:44
2	12.82	00:44
3	2.18	04:16
4	12.82	04:16
5	0.00	02:30
6	15.00	02:30
7	7.50	00:00
8	7.50	05:00
9	7.50	02:30
10	7.50	02:30
11	7.50	02:30

two independent variables: MMT concentration (0–15 g/100 g, on a dry starch + CTG basis) and sonication time (0–5 min). Cassava starch (Hikari, Brazil) was gelatinized in water (10 g starch/100 mL water) at 90  $^{\circ}\text{C}$  for 10 min. The other film components were then added - namely, CTG (starch/CTG weight ratio, 1:1), MMT (MMT-K10, Sigma–Aldrich, USA, concentration determined by the experimental design), glycerol (Dinamica, Brazil, at 30 g/100 g on a dry starch + CTG basis), and sorbitan polyoxyethylene monooleate (Tween 80, Sigma–Aldrich, St. Louis, USA, at 1 g/100 g on a dry starch + CTG basis, in order to improve the wettability of the film when applied on cashew kernels as a coating). The mixture was dispersed by using a Ultra-Turrax T-50 (Ika, Staufen, Germany) at 15,000 rpm for 15 min, followed by sonication in a 400 W ultrasonic processor (UP400S, Hielscher, Teltow, Germany) working at a 24 kHz for the time determined by the respective treatment. The dispersions were vacuum degassed by using a vacuum pump V-700 (Büchi Labortechnik AG, Flawil, Switzerland) at 3 kPa for 40 min, cast on glass plates, leveled with a draw-down bar to a thickness of 0.8 mm, and placed on a lab bench (25  $^{\circ}\text{C}$ , 50% RH, 24 h) to dry.

Dried samples were cut and detached from the surface. Before film characterization, the detached, free-standing samples were conditioned for 40 h at 24  $^{\circ}\text{C}$  in desiccators containing a saturated solution of calcium nitrate tetrahydrate ( $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ), in order to maintain the a constant relative humidity of 50%.

The film opacity was determined (in triplicate) according to a method described by Irissin-Mangata, Bauduin, Boutevin, and Gontard (2001). Films were cut into rectangular (1  $\times$  4 cm) shaped strips and placed onto the internal side of a Varian Cary 50 UV–vis spectrophotometer test cell (Agilent Technologies, Santa Clara, USA), perpendicularly to the light beam. The absorbance spectrum (400–800 nm) of the samples was recorded, and the film opacity was defined as the area under the recorded curve (calculated by the linear trapezoidal rule) and expressed as absorbance units  $\times$  nanometers (A nm).

The water vapor permeability (WVP) determination, with eight replicates, was based on the method E96-05 (ASTM, 2005) at 25  $^{\circ}\text{C}$  and 85% RH, using silica gel as the desiccant material. Eight measurements were made within a 24-h period.

Tensile properties of 100 mm  $\times$  6.3 mm film strips (with ten replicates) were measured according to D882-09 (ASTM., 2009), using a Universal Testing Machine (Emic DL-3000, São José dos Pinhais, Brazil) with a load cell of 100 N, initial grip separation of 50 mm, and crosshead speed of 12.5 mm/min.

Experimental data was analyzed using Minitab® v15 and fitted to second-order polynomial models.

The scanning electron microscopy (SEM) images of film samples from runs 7 and 8 (Table 1) were taken using a Hitachi TM 3000 scanning electron microscope (Tokyo, Japan), with the samples mounted on an aluminium stub with a double side adhesive. The samples were examined using an accelerating voltage of 5 kV, and a magnification of 1500 times.

### 2.2. Application of coatings to cashew kernels

Cashews kernels from the same batch were provided by the Companhia Industrial de Óleos do Nordeste (Cione, Fortaleza, CE). Two film dispersions were formed (according to the methodology described previously), corresponding to the treatments 5 and 9 (Table 1), namely, with no MMT added and with 7.5 g MMT/100 g (starch + CTG basis), respectively. Cashew kernels (200 g per treatment) were immersed in the respective film forming dispersions for 30 s, then removed and left to dry for 72 h. The coated kernels were then packaged in low density polyethylene (LDPE) bags, and stored at 25  $^{\circ}\text{C}$ , as well as non-coated kernels (control treatment). The stability tests (moisture contents, water activity,

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