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The influence of formulation and packaging material on the rheological properties of milk chocolate

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ABSTRACT

Fiber is a substitute for sugar in chocolates but changes its hygroscopicity and rheological characteristics if their packaging doesn't avoid its moisture gain. The objective of this study was to evaluate the water vapor permeability of different packaging materials (BOPP/metBOPP (biaxial oriented polypropylene /metallized) BOPP/BOPP white and Cellophane) and the effect of the permeability on the rheological properties (by Casson model) of three types of milk chocolate: standard, with the addition of Inulin and Fructooligosaccharides during storage at 20 °C /75% RH. Due to the high hygroscopicity of the fibers, the initial moisture content of the chocolates with fibers was higher than that of standard chocolate. No increase in viscosity were observed at standard and with Inulin chocolates during 270 days at 20 °C/75% RH, regardless of the packaging material used. However, chocolate with FOS packed in cellophane showed changes in their viscosity, with values ranging from 4.63 Pa.s to 7.5 Pa.s after 210 days of storage. In addition, the Inulin chocolate in Cellophane package showed an increase at flow limit from 3 Pa to 6 Pa after 270 days. Therefore, the results indicated that chocolates with fibers should be packed in materials with medium or high barrier to water vapor, such as BOPP/metBOPP and BOPP/BOPP, to avoid rheological changes during storage.

1. Introduction

The population in general are worried with their eating habits and they are looking for foods with reduced sugar and nutritionally content which are increasing the demand for chocolates enriched with prebiotics (Engeseth & Pangan, 2018). In milk chocolates, the percentage of sugar is high and has influence in the hardness and taste. In addition, this raw material contributes to the stability of chocolate, so its reduction can cause a change in texture, viscosity, flow limit and product structure (Fu et al., 2018).

Prebiotics can be used as substitutes for sugar and lipids because they present good solubility, and sweetness with good sensory acceptability (Rolim, 2015; Singla & Chakkaravarthi, 2017). Inulin and fructooligosaccharides (FOS) are soluble fibers, which have chemical similarity. Thus, they have the same nutritional properties, resulting in possible improvement of health and reduction of the appearance of diseases (Saad, 2006). The ingestion of prebiotics such as Inulin and FOS has several health benefits, such as reduced risk of heart disease and cancer, decreased absorption of glucose and cholesterol (Cho & Dreher, 2001). In addition, according to the International Food Information Council Foundation (2017), fiber is considered the first healthy compound by the population and sugar is considered the first compound responsible for weight gain.

Fibers, such as inulin and FOS, are considered good sugar substitutes in chocolates, as they have technological properties such as thickening, emulsifying and texturizing. They also have sweetness, low calories and no aftertaste (Corradini, Lantano & Cavazza, 2013; Dominguez, Rodrigues, Lima & Teixeira, 2013; Shourideh, Taslimi, Azizi & Amin, 2012). In addition, prebiotics are considered resistant compounds to industrial processes, being degraded only under conditions involving high temperatures and low pH, parameters not used in chocolate processing (Duar et al., 2015).

Inulin plays an important technological role and it can act as a texturizing agent, increase viscosity, form gels and stabilize emulsions (Abbasi & Farzanmehr, 2009; Mensinka, Frijlink, Van Der Voort & Hinrichs, 2015). Inulin has a sweetness equivalent at 10 to 30% of sucrose, it is less caloric than sucrose and resistant to thermal processes (stable at temperatures up to 140 °C in neutral pH solutions), which made inulin to have a great potential for applicability in many products (Meyer, 2009; Vasconcelos, Silva, Teixeira, Chaves & Martino, 2010).

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FOS can be used as a texturizing and bulking agent (Haghighi, Rezaei, Labbafi & Khodaiyan, 2011), to improving the consistency of products (Fu et al., 2018), which made FOS fiber has been used as a fat substitute and sugar in food products (Morris, 2014).

Chocolate when is heated is classified as pseudoplastic fluid, meaning that it needs a shear stress to start the movement. The rheological properties of chocolate are very important in applications like to cover ice cream and bakery products, with parts of panettones and cookies and to mold confectionaries in various shapes and sizes. The rheological properties depend process parameters such as time and temperature of conching process, tempering conditions, particle diameter and the percentage of water and fat present in the product (Afoakwa, Patersona & Fowle, 2007; Bolliger, Zeng & Windhab, 1999; Faccinetto-Beltrán, Gómez-Fernández, Santacruz & Jacobo-Velázquez, 2021).

The inclusion of hygroscopic fibers can increase the food's ability to absorb moisture (Bolenz, Amtsberg & Schäpe, 2006) what change the rheological characteristics of the product if it isn't packaged in a water vapor barrier packaging. Chocolates are products that can present chemical, physical and sensory changes during their useful life, due to their sensitivity to humidity, light, oxygen, temperature changes and absorption of strange odors (Robertson, 2013).

The development of health chocolates is occurring by inclusion of functional components such fibers, vitamins, minerals and phytochemicals constituents in chocolate matrix without causing changes in sensory quality, physical and chemical structure during their shelf life (Anadón, Martínez-Larrañaga, Arés & Martínez, 2016; Ares, Besio, Gimenez & Deliza, 2010).

In this context, to the best of our knowledge, no study has been published on the evaluation of the effect of packages with different water vapor barrier values on the rheological stability of chocolates with and without fibers. Therefore, this is the main novelty of this research. Thus, the objective of this study was to evaluate the water vapor permeability of different packaging materials and their effect on the rheological properties of three types of milk chocolate (standard, with the addition of Inulin and of Fructooligosaccharides) during storage at 20 °C /75% RH.

2. Material and methods

2.1. Raw materials

The raw materials used for production of milk chocolate were: sugar (Mais Doce, SP, Brazil), deodorized cocoa butter (Olam Cocoa, Ilhéus, Brazil), CB), natural cocoa mass (Olam Cocoa, Ilhéus, Brazil), skimmed milk powder (Itambé, Uberlandia, Brazil), lecithin (Danisco, Pirapozinho, Brazil), PGPR (Polyglycerol polyricinoleate) (Concepta, Santa Bárbara D'Oeste, Brazil), FOS fiber (Ingredion, Westchester, IL, USA) and Inulin fiber (Sensus, Roosendaal, The Netherlands).

2.1.1. Characterization of powder ingredients

2.1.1.1. Moisture content. determined according to AOAC Official Method 934.06 (AOAC, 2010) using a vacuum oven (model VDL53, WTB BINDER, Tuttlingen, Germany) at 70 °C until a constant weight of samples was reached.

2.1.1.2. Water activity. measure in Aqualab hygrometer (Decagon, Pullman, United States), at 25.0 ± 1.0 °C (Decagon Devices, s.d).

2.2. Milk chocolate formulations and manufacture process

The three types of chocolates: standard chocolate, Inulin chocolate and FOS chocolate presented the composition describe in Table 1.

The chocolates were produced at the Cereals and Chocolates Technology centre (ITAL, Campinas, Brazil) in batches of 10 kg per formulation, according to Afoakwa (2010) and Silva et al. (2017) with adaptations by inverting the stages of refining and conching. In the refining step the maximum particle size was checked using a micrometer (MITUTOYO, Kawasaki Japan). After processing, the chocolates were demolded, packaged and stored at 20 ± 1 ºC for 15 days to stabilize the crystalline structure of the lipid matrix before starting the analysis.

TABLE 1
Formulations of milk chocolates.

Ingredients	Standard (%)	Inulin (%)	FOS (%)
Sucrose	45.0	29.5	29.5
Cocoa butter	22.5	22.5	22.5
Skim powdered milk	17.0	12.5	12.5
Cocoa liquor	15.0	15.0	15.0
Inulin	0.0	20.0	0.0
FOS	0.0	0.0	20.0
Soy lecithin	0.3	0.3	0.3
PGPR	0.2	0.2	0.2

2.2.1. Morphology

The microstructure of chocolates was observed with an optical microscope (BX41, Olympus, Tokyo, Japan) and a digital image capture (Q-Color3, Olympus, Tokyo, Japan) with a magnification of 1000 times (adapted from Aidoo, Appah, Dewalle, Afoakwa & Dewettinck, 2017).

2.3. Packaging materials

Three flexible packages with differences in composition (Table 2) were used to produce 100 mm x 35 mm packages that were heat sealed by electrical impulse (Haramura - A380 Regente, São Paulo, Brazil).

The BOPP/metBOPP and the BOPP/BOPP was obtained through the lamination process, the BOPP and BOPP metallized layer being responsible to the water vapor and gasses barrier, that regulates the permeation of water vapor and oxygen from the outside to the inside of the package. In both structures, the external BOPP is very transparent and is usually printed internally to protect the print and provides excellent optical properties. The BOPP in the inner layer is responsible for heat sealing. These packaging materials were being those normally used for chocolates (Verde, Alvim, Luccas & Alves, 2021). The Cellophane representing an alternative of a renewable and compostable source, since there is a worldwide tendency to search for non-fossil sources of materials (Beckett, 2009; Verde et al., 2021).

2.3.1. Assessment of barrier properties

2.3.1.1. Water vapor transmission rate (WVTR). The WVTRs of BOPP/metBOPP and BOPP/BOPP were determined in equipment with an infrared sensor - PERMATRAN - (3/31, MOCON, Minneapolis, USA) according to the procedure described in ASTM F1249–13 (2013). The effective permeation area of each specimen was 50 cm^2 and the test was conducted at 38ºC/90% RH in four repetitions. The WVTR of Cellophane was determined according the ASTM E 96/E 96 M-16 - Standard test methods for water vapor transmission of materials (ASTM, 2016). All WVTR were determined in four repetitions.

2.3.1.2. Oxygen transmission rate (OTR). OTRs were determined according to the ASTM D 3985–05 - Standard test method for oxygen gas transmission rate through plastic film and sheeting using a coulometric sensor), in OXTRAN equipment (2/20, MOCON, Minneapolis, USA), operating with oxygen (99.99%) as permeant gas. The readings were corrected for 1 atm of partial pressure gradient of the permeant gas. The permeation area was 50 cm^2 and the tests were performed at 23 °C and dry conditions, in three repetitions

2.4. Viscosity and flow limit

The viscosity and flow limit of the chocolate samples were determined in triplicate in a rheometer (Brookfield, RVDIII, Middleboro,

Table 2
Packaging materials used in the study.

Packaging material	Description	Total thickness* (μm)
BOPP/metBOPP	Biaxially oriented polypropylene laminated to metallized biaxially polypropylene, heat sealed (Coverplast, Brazil)	37.65 ± 0.8[a]
BOPP/BOPP	Biaxially oriented polypropylene laminated to white biaxially polypropylene, heat sealed (Vitopel, Brazil)	45.57 ± 0.4[b]
Cellophane	White Regenerated Cellulose Film (Futamura, United Kingdom)	50.3 ± 0.0006[c]

**Mean ± standard deviation (n* = twenty-five determinations in five specimens *3).*
[a,b,c] Means followed by different letter in the column differ statistically from each other ($p > 0.05$).

United States), using a cylindrical spindle (specification: S15). Shear stress was measured at 40 ± 0.5 °C. The results obtained for shear rate and shear stress were adjusted according to the Casson model (Vissotto et al., 1997) to express the plastic viscosity (ηCa) and the yield stress (τCa).

2.5. Hardness (snap test)

Hardness (snap test) of chocolate: was determined using a texturometer (TA-XT2i, Stable Micro Systems, England, Three Point Bend Rig and platform OR Stand - HDP/3 PB). The parameter evaluated was the force of rupture applied to the center of the bar/cross-sectional area of the bar, with a test speed of 1.7 mm/s and this test were repeated ten times. The bar dimensions were: (80×25×10) mm (adapted from Luccas, Bonomi & Kiechbusch, 2014; Silva & Conti-Silva, 2017).

2.6. Statistical analysis

The results were analysed according to the following tests: the Shapiro-Wilk and Anderson-Darling normality tests, Bartlett's and Levene's tests, ANOVA and Welch's tests, the non-parametric Kruskal-Wallis test to verify the equality between means, the multiple paired comparisons of the Tukey averages, the Games-Howell test, the T2 of Tamhane test and the Dunn's non-parametric test, all using the software Addinsoft - XLSTAT - Version 2015.6.01.24494.

3. Results and discussion

3.1. Characterization of powder ingredients

The FOS and inulin presented 1.77 ± 0.18% (w.b.) and 1.99 ± 0.09% (w.b.) of moisture and 0.32 ± 0.01 and 0.36 ± 0.01 of water activity (Aw), respectively. The refined sugar presented moisture of 0.25 ± 0.05% (d.b.) and 0.241 ± 0.06 Aw. The powdered milk moisture was 3.11 ± 0.77% (d.b.) and 0.255 ± 0.06 Aw (ASTM 2020).

3.2. Formulation, production and characterization of chocolates

The substitution of sugar by the fibres promoted a 34.4% reduction in sugar in the fiber formulations compared to the standard (Table 1). Thus, according to Brazilian legislation, the products can be declared "light in sugar" (minimum reduction of 25%, RDC No. 54, Brazil, 2012).

The sweetness of the samples was not adjusted, taking advantage of the sweet taste of the fibres to compensate for this sensorial characteristic. The percentages of butter and cacao liquor remained the same in the three formulations (Table 1) according to Brazilian legislation (minimum of 25% of cocoa derivatives in milk chocolate, RDC No. 264, Brazil, 2005) and to avoid possible color variations in products.

The conventional sequence for chocolate production initially involves the refining of the mass in a roller mill followed by conching (Afoakwa, 2016).

For this work, the production of traditional (control) or fiber-added chocolates was carried out with conching first and then refining in a ball mill. In this order, the refining step allowed the correction of particle size defects (eventual formation of aggregates due to the absorption of moisture by the fibres during conching) and guaranteed the adequate granulometry of the chocolate masses produced with fibres.

At the end of the refining step, the maximum particle size obtained in the control and fiber formulations was between 20 and 25 μm, which is within the range of values lower than 30 μm recommended by Talbot (2009) and Beckett (2013) and avoiding the possibility of a gritty sensation when consuming the product.

3.2.1. Moisture content and water activity (Aw) of chocolates

The chocolates presented Aw values in the range of 0.32 to 0.36. Values lower than 0.60 indicate that the products do not present a risk of developing fungi and yeasts. The Aw values were within the range of 0.2 to 0.4, which meant the oxidation reaction was low and suggests there was no favoured lipid oxidation (Labuza, 1975).

The Aw of the chocolates with FOS was the same as the control chocolate, with no significant difference (p <0.05). The water activity for chocolates with inulin was similar to that obtained by Farzanmehr & Abbasi (2009) and Konar et al. (2018) who obtained values of 0.34 and 0.395, respectively. Despite the hygroscopic characteristics, inulin has a high binding capacity in products with low Aw values (Farzanmehr & Abbasi, 2009).

Standard, inulin and FOS chocolates presented moisture contents below 2% within the recommendation by Beckett (2013) for greater fluidity of the product and to avoid aggregation of the sugar particles. On an industrial scale probably the humidity of the chocolates would be smaller because the conches are closed.

Chocolates with inulin and FOS showed higher moisture contents than the control chocolate. The processing methods used were the same for the three formulations so the increase in moisture may have been due to the hygroscopic fibres added to the formulation. The results obtained were lower than those observed by Shah (2010) for milk chocolates with fibres and higher than those obtained by Aidoo (2014) for chocolates with inulin.

3.2.2. Morphology

The appearance of the chocolate samples is shown in Fig. 1. The control sample presented a typical appearance of solid lipid dispersion with evidence of cocoa solids (dark particles) and sugar crystals (hexagonal particles). Aidoo et al. (2017) reported similar characteristics for chocolate products with added polydextrose and inulin. The samples containing the fibres presented typical material particles produced by spray drying, in addition to the cocoa and sugar solids, which could indicate the presence of the fibres; this drying process is commonly used to obtain these ingredients in the form of powder.

3.3. WVTR and OTR of the packaging materials

The WVTR results of the packaging materials, shown in Table 3, demonstrate in that the materials that presented the greatest barrier to water vapor were BOPP/metBOPP due to metallization on the BOPP film directly, without the presence of printing between the BOPP film and the metallization, which minimizes discontinuity in the aluminum deposition layer on the BOPP film. The BOPP/BOPP present a medium water vapor barrier and the Cellophane higher water vapor permeation. This water vapor permeability of these materials will protect more or less the chocolate during storage against moisture gain.

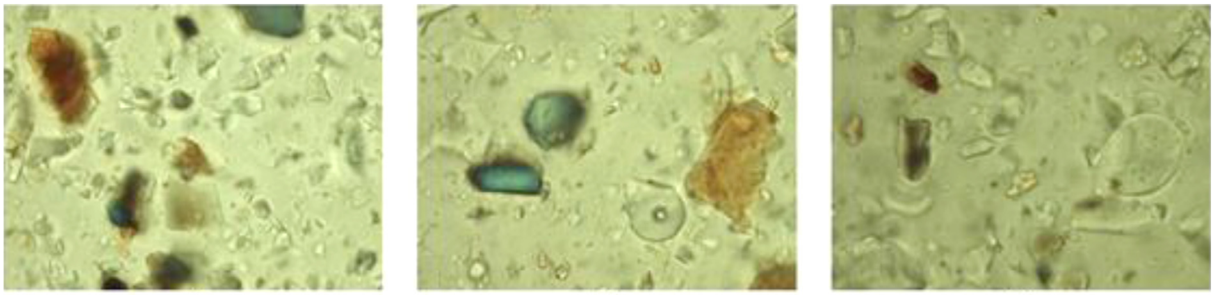

Fig. 1. Optical microscopy showing the morphology of chocolate. A: Standard chocolate; B: Inulin chocolate; C: FOS chocolate. 1000 × magnification.

Table 3
Barrier properties of the packaging materials.

Packaging material	WVTR*(g water.m^{-2}.day^{-1})	OTR**(mL (STP).m^{-2}.day^{-1})
BOPP/metBOPP	0.31 ± 0.01^{a}	18.69 ± 0.64^{a}
BOPP/BOPP white	3.51 ± 0.06^{b}	910.84 ± 19.27^{b}
Cellophane white	575.82 ± 9.40^{c}	0.005 ± 0^{c}

Mean ± standard deviation (n = *four and ** three determinations).
[a,b,c] Means followed by different letter in the column differ statistically from each other (*p*>0.05).
WVTR - Water vapor transmission rates at 38 °C/90% RH; OTR – Oxygen transmission rate at 23 °C/75% RH and 1 atm of partial oxygen pressure gradient.
BOPP - Biaxially oriented polypropylene; met – metallized; Cellophane - Regenerated Cellulose.

The OTR results presented in Table 3 show that those with the greatest oxygen barrier were Cellophane and the medium oxygen barrier was the BOPP/metBOPP due to the presence of metallization on the BOPP film directly, without the presence of printing between the BOPP film and the metallization as discuss previously. The BOPP/BOPP present height oxygen permeation.

The chocolates were wrapped in packages without headspace modification and, therefore, the fact that the materials have distinct oxygen permeability rates should not interfere with the quality of the product since the packages had the same composition as the atmospheric air in the storage (21% oxygen in the headspace).

3.4. Viscosity and flow limit

The viscosities of the traditional chocolates and those containing inulin, packed in the three types of packaging were very close, and did not present differences or significant increases after 270 days at 20ºC/ 75% RH (Figs. 2a, 2b). The results obtained for the traditional chocolate differed from those observed by Kiumarsi, Majchrzak, Yeganehzad, Jäger and Shahbazi (2020), who used aluminum foil to wrap the chocolates and found an increase in viscosity after 30 days at 18 ºC. However, in the formulations of chocolates with higher concentrations of inulin, these same authors also found no significant changes in the viscosity during storage, similar to that observed in the present study.

The chocolate with FOS (Fig. 2c) in BOPP/metBOPP differed significantly (p<0.05) from those packed in BOPP/whiteBOPP and Cellophane after 120, 150 and 270 days of storage at 20ºC/75% RH. During storage the values for the viscosity of the chocolates were similar, although there were some statistical variations. However, the chocolates with inulin and FOS in Cellophane packages presented higher viscosities than the traditional chocolate after 270 days of storage, corroborating the results obtained by Konar et al. (2018). The increase in viscosity is directly related to the increase in moisture content of the chocolates with fibers packed in packaging with high water vapor permeability, principally in Cellophane, due to the fact that the sugar particles absorb the moisture and agglomerate forming sandy lumps which migrate to the surface of the product (sugar bloom), causing greater resistance to the flow of the chocolate (Afoakwa, Paterson & Fowler, 2008). Saputro et al. (2017) also showed the formation of amorphous sugar in chocolate with higher moisture content, causing agglomeration of the particles and greater viscosity due to the fact that larger particles need the availability of greater fat percentages to reduce the particle-particle interactions in the chocolate. Chocolates with inulin and FOS in BOPP/metBOPP showed similar viscosities (p>0.05) to the traditional chocolate during storage at 20ºC and 75% RH, as observed after the production of chocolates with 25% inulin by Shourideh et al. (2012). According to Aidoo, Depypere, Afoakwa and Dewettinck (2013), the fibers form films with the emulsifiers, coating the solid particles and thus increasing the viscosity of the chocolates, making them functional compounds adequate to substitute sugar.

The flow limit of the traditional chocolate increased during storage at 20ºC/75% RH (Fig. 3a), similar to that observed by Kiumarsi et al. (2020). However, the increase in flow limit was greater in the chocolates containing the fibers inulin and FOS, as also observed by Rodriguez Furlán, Baracco, Lecot, Zaritzky & Campderrós, 2017. The chocolate with inulin in BOPP/metBOPP showed no statistical difference (p <0.05) in the flow limit during storage but differed significantly from the same product in BOPP/whiteBOPP and in cellophane (Fig 3b). Inulin is a good gel former in aqueous systems due to the strong interaction between its molecules in water (Ronkart, Paquot, Fougnies, Deroanne & Blecker, 2009). Despite chocolate showing a low moisture content, interactions of the charges between the solid particles dispersed in the lipid base of the product still occur, and, since inulin is a polymer that favors interactions, when associated with the gradual increase in moisture content, could have contributed to increasing the flow limit of the product in the packaging more permeable to water vapor (Fig. 3c). An increase in the flow limit of chocolates with inulin was also observed by Kiumarsi, Rafe and Yeganehzad (2017), due to the formation of a crystalline structure with the addition of inulin. Note in Fig. 2c that the flow limit of the chocolates with FOS in cellophane was similar to that of the chocolates in the other packaging types. After 270 days at 20ºC/75% RH, the chocolate with FOS in cellophane showed a flow limit

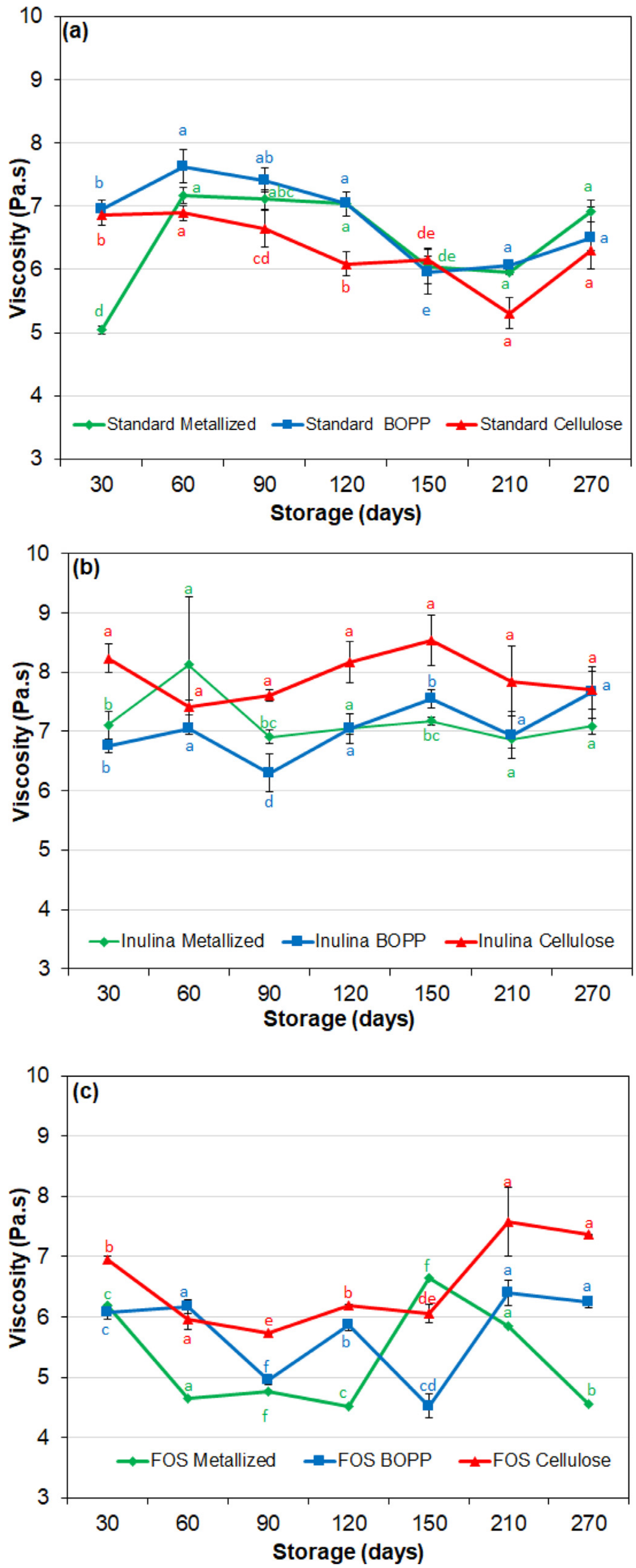

Fig. 2. Viscosity of the three types of chocolates during the storage. A: standard chocolate; B: Inulin chocolate and C: FOS chocolate; green line: BOPP/metBOPP; blue line: BOPP/whiteBOPP; red line: Cellophane.

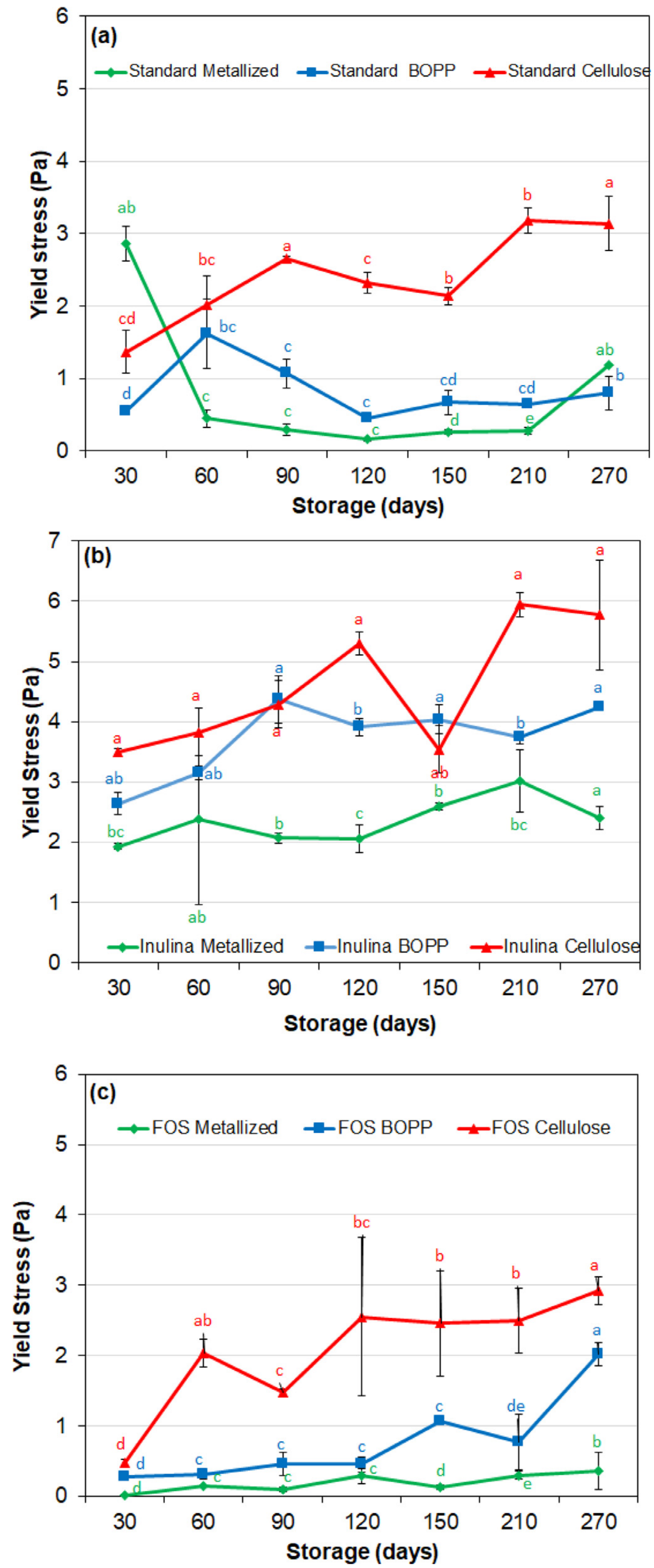

Fig. 3. Yield stress of the three types of chocolates during the storage. A: standard chocolate; B: Inulin chocolate and C: FOS chocolate; green line: BOPP/metBOPP; blue line: BOPP/whiteBOPP; red line: Cellophane.

of 2.92 Pa, although it was only 0.47 Pa after 30 days of storage, due to the increase in moisture content of the chocolate with FOS during storage. This phenomenon was also observed by Zyzlewicz, Nebesny, Motyl and Ibudzisz (2010) and by Rad et al. (2019) soon after the production of chocolates with hygroscopic ingredients with high moisture contents.

3.5. Hardness (snap test)

There was an increase in the hardness of the chocolates during storage, however this increase did not show any significant difference ($p>0.05$). between the three packages, according to the Figs. 4a, 4b and 4c.

Standard chocolate showed greater hardness than chocolates with fibers at the end of the study, the same behavior was observed by Shourideh et al. (2012) shortly after the production of chocolates with inulin. According to Pimentel, Garcia and Prudencio (2012) inulin has the function of making products soft and creamy. Contrary to what was observed by Silva and Conti-Silva (2018) and Silveira et al. (2015) after production, in which products with the addition of inulin showed greater hardness in relation to the standard, which increased with the addition higher percentage of fiber.

During storage, standard and fibers chocolates showed variations in textures, but a greater increase occurred after 210 and 270 days of storage at 20C/75%RH, however the increase was less in chocolates with fibers compared to standard chocolate, as observed by Kiumarsi et al., 2020. As discuss by Verde et al. (2021), the change in the hardness of chocolates during the storage was not sensorially perceived.

4. CONCLUSIONS

The replacement of sugar by fibers promoted a sugar reduction of 34.4% in chocolates. The rheological stability of chocolates packed in Cellophane material was altered due to the higher permeability to water vapor, where changes in the viscosity of milk chocolate were observed after 210 days of storage and an increase in the flow limit of chocolate with inulin was observed after 270 days of storage. Chocolates packaged in BOPP/metBOPP and BOPP/BOPP white showed no changes in viscosity, flow limit and texture during 270 days of storage. These results indicated that chocolates with the addition of hygroscopic fibers need packaging with high and medium moisture barrier properties in order not to present rheological changes during storage in places with high relative humidity.

Ethical statement - Studies in humans and animals

The authors declare that the research presented does not involve any animal or human study.

Declaration of Competing Interest

The authors declare that they have no conflict of interest.

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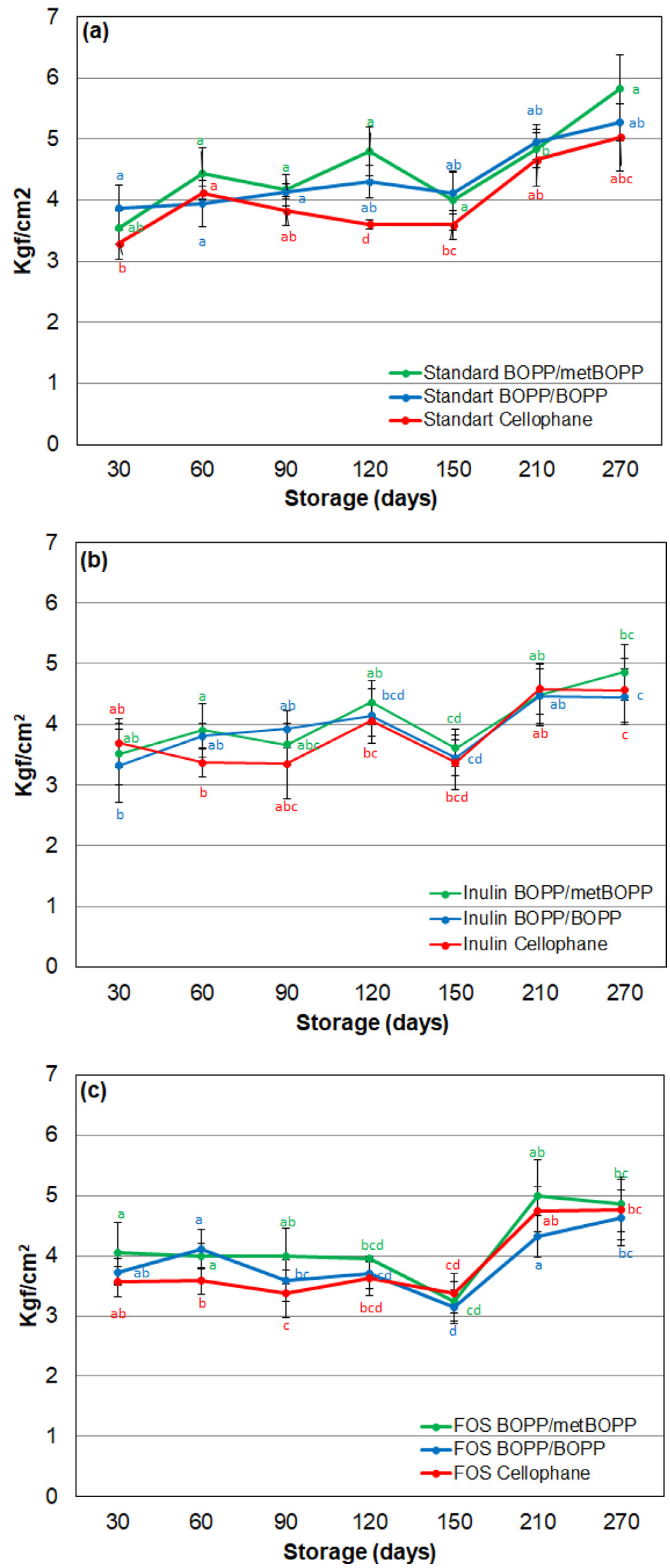

Fig. 4. Hardness of the three types of chocolates during the storage. A: standard chocolate; B: Inulin chocolate and C: FOS chocolate; green line: BOPP/metBOPP; blue line: BOPP/whiteBOPP; red line: Cellophane.

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