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Alternative flexible plastic packaging for instant coffees

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ABSTRACT

Instant coffees are consumed worldwide and their packages must protect them mainly from moisture gain. Flexible packaging stand-up pouches made by PET/Al foil/LDPE are currently used but, the look for alternative materials is interesting to replace the aluminum foil with reducing costs and focusing on sustainability. Therefore, the aim of this study was to evaluate the quality loss of freeze-dried and spray-dried (agglomerated and powder) instant coffees during 365 days at 25 °C/75% RH, packaged in five plastic structures: PET (polyethylene terephthalate)/Al (aluminum) foil/LDPE (low density polyethylene), LDPE/HDPE (high density polyethylene)/LDPE, BOPP (biaxially oriented polypropylene)/BOPP met (metallized)/PP, PET/PET met/LDPE and PET/BOPP met/LDPE. The results were compared with the shelf-life estimated by modeling the moisture sorption isotherms of the products by mathematical models. Results indicated that the lower the barrier to water vapor of the packaging material, the greater the gains in moisture and water activity of the instant coffees and in addition to being thermally less stable. After 365 days of storage, the three soluble coffees still had acceptable characteristics in the five packaging structures, indicating that it is possible to replace the currently used laminate, which contains aluminum foil, with recyclable structures. However, the greatest stability for the coffees was obtained using the alternative structures: BOPP/BOPP met/PP and LDPE/HDPE/LDPE, a result that was in concordance with that obtained by mathematical modeling.

1. Introduction

Instant coffee is the second coffee beverage most consumed worldwide due to its ease of consume. These products are obtained from freshly ground-roasted coffee beans by extraction with hot water (2011). There are two conventional techniques to manufacturer instant coffees: spray drying (SD) and freeze-drying (FD). The SD is the most economical process and the FD presents higher quality of aroma and flavor (Ishwarya & Anandharamakrishnan, 2015).

Instant coffees are very susceptible to quality loss due to moisture gain and requires packaging barrier against the ingress of moisture by permeation and hermetic closure system (Robertson, 2013).

The initial moisture content of instant coffees is 2% to 4% and moisture absorption is much more critical in this product than in roast and ground coffee, which can cause full agglomeration of the particles when the moisture content reaches 7% to 8%. This union of particulate matter will be greater, the greater the moisture content absorbed by the product. In addition, the high moisture content of coffee accelerates the quality loss reactions associated with oxidation (Robertson, 2013). Temperature can also accelerate the deterioration process of soluble coffee.

Severe agglomeration, which results in product hardening and leads to rejection by consumers, is a major problem in the food industry. It is a result of moisture sorption by the powdered food due to the inadequate barrier provided by the selected packaging material. The glass transition temperature (Tg) is a very important parameter used to describe and control the agglomeration of particulate foods, being characteristic of the product, which depends, among other factors, on its composition and water content. The food is expected to be stable below its Tg. Above this temperature, the difference between the Tg and the storage temperature is responsible for controlling the rate of physical and chemical changes in the product (Yamato, 2018).

Moisture gain is a critical parameter that determines the quality and shelf life of low-moisture foods. The rate and extent of moisture transfer from the environment to soluble coffee packaged in plastic containers depend on its water activity, temperature, relative humidity of the

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storage environment, the water vapor permeability of the package material as well as the integrity of the packaging closing system. The water vapor permeation model based on Fick's law and moisture sorption isotherms is often combined with mathematical simulation to predict or estimate the shelf life of products which is limited by the observed maximum or critical moisture value (Robertson & Lee, 2021).

At all the world, instant coffees are marketed in glass jars with seals and screw caps, metal cans with easy-open lid and plastic over cap and flexible plastic packaging (stand-up pouch) made of multilayer film (PET/Al foil/LDPE) where the aluminum foil is the barrier layer to avoid the permeation of gases, moisture and loss of aroma. It is important to highlight, that flexible plastic packaging has lower cost of production than the glass jars and presented higher mechanical resistance, which reduces product losses in filling lines, storage, point of sale and final disposal (Andregheti, 2015), but it does not have a sustainability appeal and represent a problem from an environmental point of view due to the separation difficulty of the materials and their incompatibility in the plastic material recycling lines (Teixeira, 2013).

Therefore, the aim of this study was raising subsidies to select alternative materials for instant coffee flexible plastic packaging for instant coffees. Spray-dried agglomerated and powder and freeze-dried instant coffee were packaged in PET/Al foil/LDPE and alternative plastic structures: LDPE/HDPE/LDPE, BOPP/BOPPmet/PP, PET/PETmet/LDPE and PET/BOPPmet/LDPE. The instant coffee was evaluated regarding to water activity, initial moisture and thermal analysis during 365 days at 25 °C/75% RH. The results were compared to the shelf-life estimated by modeling the moisture sorption isotherms of the products at the same storage conditions.

2. Material and methods

2.1. Instant coffees

Three types of instant coffees (100% *Robusta* variety) freshly produced were evaluated:

1) **Freeze-drying** – The coffee extract was freeze-dried at a shelf temperature starting from −20 to −40 °C.
2) **Spray-drying agglomerated** – The coffee extract was atomized at a temperature of over 200 °C and agglomerated by controlled humidification.
3) **Spray-drying powder** - The coffee extract was atomized at a temperature of over 200 °C.

Table 1
Alternative flexible plastic materials evaluated and PET/Al foil/LDPE. *.

Structure	WVTR (g of water.m^{-2}.day^{-1}) at 25 °C/75% RH	O_2TR (mL (NTP). m^{-2}.dia^{-1}) at 23 °C and 1 atm of partial pressure gradient of oxygen
PET 12 µm /Al foil 13 µm/LDPE 55 µm	< 0.01^{a}	< 0.005^{a}
LDPE 33 µm/HDPE 33 µm/LDPE 32 µm	0.11 ± 0.00^{d}	0.20 ± 0.02^{e}
BOPP 29 µm/BOPPmet 16 µm/PP 26 µm	0.04 ± 0.00^{d}	0.37 ± 0.01^{d}
PET 12 µm/PETmet 12 µm/LDPE 67 µm	0.19 ± 0.03^{c}	1.07 ± 0.00^{c}
PET 10 µm/BOPPmet 11 µm/LDPE 53 µm	0.37 ± 0.07^{b}	3.95 ± 0.12^{b}

* WVTR = water vapor transmission rate, O_2TR = oxygen transmission rate, mean of four determinations ± standard deviation, ^{a,b,c,d,e} the means, followed by the same letter, in the column, do not differ at the 95% confidence level (p < 0.05).

2.2. Alternative flexible plastic materials

Four alternative flexible plastic material structures commercially available and PET/Al foil/LDPE were used in this study. Table 1 present characteristics of the materials and water vapor and oxygen transmission rate properties.

2.3. Packaging and storage

Packs with 0.0256 m^2 were manufactured with all materials presented in Table 1 and 50 g of the three types of each instant coffee were conditioned in each material and stored during 365 days at 25 °C/75% RH in a Weiss chamber model Pharma 1300 (WeissTechnik, Reiskirchen, Germany). Packaged instant coffees were evaluated for water activity, moisture content and thermal properties every 2 months.

2.4. Instant coffee evaluation

2.4.1. Water activity

The water activity of instant coffees (~2 g) was determined at 25 °C using DECAGON - AquaLab series 4TEV Water Activity Meter hygrometer based on psychometry, with resolution of 0.0001a$_w$ (Decagon Devices, INC s.l.s.d).

2.4.2. Moisture content

Moisture content was determined by weighing samples (~3 g) in preweighted aluminum capsules until constant weight using a vacuum chamber model VDL53 (WTB Binder, Tuttlingen, Germany) at 70 °C (AOAC 979.12, 2011).

2.4.3. Thermal properties

Thermal transitions were monitored using a Differential Scanning Calorimeter, model Discovery DSC 250 (TA Instruments, USA), with auto sampler and liquid nitrogen cooling attachment. Instant coffees (~5 mg) were sealed in aluminum pans to ensure minimal changes in moisture content and run from − 70 °C to 110 °C, at scan rates of 10 °C. min^{-1} with reheats being carried out (Frascareli, 2010). The machine was calibrated for temperature using the onset temperatures of melting and for heat flow using the enthalpy of transition of indium. Glass transition temperature (Tg) and heat capacity (ΔCp) values were calculated using TA Instruments TRIOS software.

2.5. Moisture sorption isotherm

The static gravimetric method was used to determine the adsorption isotherm. Instant coffees were kept at 25 °C for 21 days in desiccators with salt solutions that ensure different water activities (a_w): LiCl (0.113), $MgCl_2$ (0.328), K_2CO_3 (0.432), Mg $(NO_3)_2$ (0.529), NaBr (0.576), $NaNO_2$ (0.633), KI (0.689), NaCl (0.754), $(NH_4)_2SO_4$ (0.810), KCl (0.843) e $BaCl_2$ (0.903) (ASTM E104 (2012)). Approximately 1 g of each material was weighed into weighs glass filter, in triplicate. After the equilibration time, each sample was weighed again and the instant coffee was evaluated such as visually changes for definition of critical moisture.

2.6. Mathematical modeling

The experimental data obtained by moisture sorption isotherm of instant coffees were fitted to some classical mathematical models for moisture sorption predictions: GAB (Guggenheim-Andersen-de Boer), Halsey and Henderson (equations 1–3). These models provide the relation between the equilibrium content (X_{we} g of water g^{-1} d.m.) and the water activity (a_w) (Clément et al., 2018; Vega-Gálvez et al., 2008). The determination of the constants used to fit the data was carried out employing *Excel®* software. The generalized reduced gradient (GRG) method from the *Excel Solver* was performed to estimate the model parameters.

GAB $$X_{we} = \frac{X_m.C.k.a_w}{(1-k.a_w).(1+(C-1).k.a_w)} \quad (1)$$

Halsey $$X_{we} = \left(\frac{A}{\ln(\frac{1}{a_w})}\right)^{1/B} \quad (2)$$

Henderson $$X_{we} = 0.01\left[\frac{-\log(1-a_w)}{10^f}\right]^{1/n} \quad (3)$$

To evaluate the goodness of the curve fitting, in all cases the determination the minimum percent root means square error (*%RMSE*), minimum mean absolute percentage error *(E)*, and the maximum adjusted coefficient of regression (R^2) were provided (equations 4–6).

$$\%RMSE = \sqrt{\frac{1}{N}\sum_{i=1}^{N}\left(Yi - \frac{Y'i}{Yi}\right)^2} \times 100 \quad (4)$$

$$E = \frac{100}{N}\sum_{i=1}^{N}(Yi - \frac{Y'i}{Yi}) \quad (5)$$

$$R^2_{Adjusted} = 1 - \frac{\sum_{i=1}^{N}(WiYi - Y'i)^2(N-1)}{\sum_{i=1}^{N}(WiYi - Y'')^2(N-M)} \quad (6)$$

Where, according Suhag et al. (2018):
Y = experimental value of equilibrium moisture content.
Y' = predicted value of equilibrium moisture content.
Y'' = mean of experimental data of equilibrium moisture content.
N = number of observations.
W = weighting applied to each data point.
M = number of coefficients in each equation.

2.7. Wvtr prediction for alternative plastic flexible packaging selection

The water vapor transmission rate (WVTR) was prediction for instant coffee packaging materials after adjusting to the best mathematical model equation of the moisture sorption isotherms (7) (Alves & Bordin, 1998). The with critical parameters as: instant coffee weight in the packaging (50 g, 100 g, and 200 g), flexible packaging area (0.0256 m^2, 0.0481 m^2, and 0.0585 m^2) and storage time (365 days and 730 days) at 25 °C/75% RH.

$$t = \frac{Md.RHs}{100.A.WVTR}\int_{M0}^{Mc}\frac{dM}{\frac{RHs}{100} - Aw(M)} \quad (7)$$

Where:
t = shelf-life (days).
Md = dry mass of the product (g).
RHs = relative humidity of storage (%).
A = packaging area (m^2).
$WVTR$ = water vapor transmission rate (g of water.m^{-2}.day^{-1}).
A_w = product water activity as a function of moisture content, which is the moisture sorption isotherm of the product.
M_0 = initial moisture (g of water. 100 g of the dry $product^{-1}$).
M_c = critical moisture (g of water. 100 g of the dry $product^{-1}$).
This mathematical model assumes that the transfer of water vapor from outside the package to the inside is slow, then, the water vapor that passes through the packaging, it is evenly distributed in the food product and, therefore, the phenomenon that governs the moisture gain of the product is the transfer of water vapor from outside to the inside of the package (Alves & Bordin, 1998). Equation (7) was integrated using the *Excel*® software.

2.8. Statistical analysis

The results were statistically evaluated by means of analysis of variance (ANOVA) and the Tukey test to compare the averages ($p < 0.05$).

3. Results and discussion

3.1. Instant coffee evaluation

Fig. 1 present water activity and moisture content results for all instant coffees conditioned in the 4 alternative flexible plastic materials and a standard material for 12 months at 25 °C/75% RH.

Over 12 months of storage at 25 °C/75%RH, instant coffees showed higher water activity and moisture content (%d.b.) when packaged in PET/BOPPmet/LDPE, followed by coffees in PET/PETmet/LDPE, LDPE/HDPE/LDPE and BOPP/BOPPmet /PP.

Since the closure of the packaging was hermetic, all instant coffee' moisture increase because the water vapor permeation of the packaging materials was comparatively superior respectively in 370, 190, 110 and 40 times in PET/BOPPmet/LDPE, PET/PETmet/LDPE, LDPE/HDPE/LDPE and BOPP/BOPPmet/PP compared with PET/AI foil/PE.

These comparisons considering an ideal condition, in which the instant coffee packaging is exposed to 25 °C/75%RH, which in practice does not occur, due to variations in the temperature and relative humidity conditions during the storage time.

However, during 12 months of storage no water activity above 0.5 was observed for any of the three types of instant coffees packaged in the four structures studied. the three types of instant coffees. For moisture content, only the spray-drying agglomerated instant coffee exceeded 7% (d.b.) when packaged in PET/BOPPmet/LDPE.

Water activity and moisture content are considering the most important factors for the stability of dry foods, they are closely linked to the shelf life of coffee products. Similar to the data obtained in this study, Deotale et al. (2020) reported values of 0.41 for spray-drying instant coffee and 0.35 for freeze-drying instant coffee newly produced.

Instant coffee produced by freeze-drying normally presents shelf-life higher than spray-drying process (Robertson, 2013), probably because the initial moisture of the product by freeze-drying is around 2% (Pintaro, 1975), while by atomization is 4.5% (Labuza, 1982). However, the initial moisture of the instant coffees depends on the type of packaging and the closure system.

The thermal properties (Tg and ΔCp) results are presented at Fig. 2 for the instant coffees packaged in the five materials and storage during 12 months at 25 °C/75% RH.

Over 12 months of storage at 25 °C/75%RH, instant coffees showed greater increase in glass transition temperature (Tg) and decrease in the step-in heat capacity (ΔCp) when packaged in PET/BOPPmet/LDPE, followed by coffees in PET/PETmet/LDPE, LDPE/HDPE/LDPE and BOPP/BOPPmet /PP.

Tg and ΔCp are normally measured from the second heating to remove thermal history of the material and to obtain reliable values. For instance, Yu et al. (2012) reported Tg of 45 ± 1.0 °C and ΔCp of 0.34 J g^{-1} °C^{-1} for a freeze-dried instant coffee evaluated with a composition of 35% of carbohydrate (mono and disaccharides), with the remaining 25 % being long chain arabinogalactan and galactomannan. This indicates that the thermal properties of soluble coffee depend on the type and amount of carbohydrates in the product. The glass transition temperature of instant coffees increases by adding high molecular weight polysaccharides (Manzocco et al., 2016).

Spray-dried agglomerated instant coffee packaged in PET/BOPPmet/LDPE reached the temperature of 25 °C before 10 months of storage follow by freeze-dried instant coffee (11 months). Greater the difference between Tg and temperature of storage, the more stable the product. Then if instant coffee collapses when Tg is overcome during storage, temperature and relative humidity are critical parameters to control.

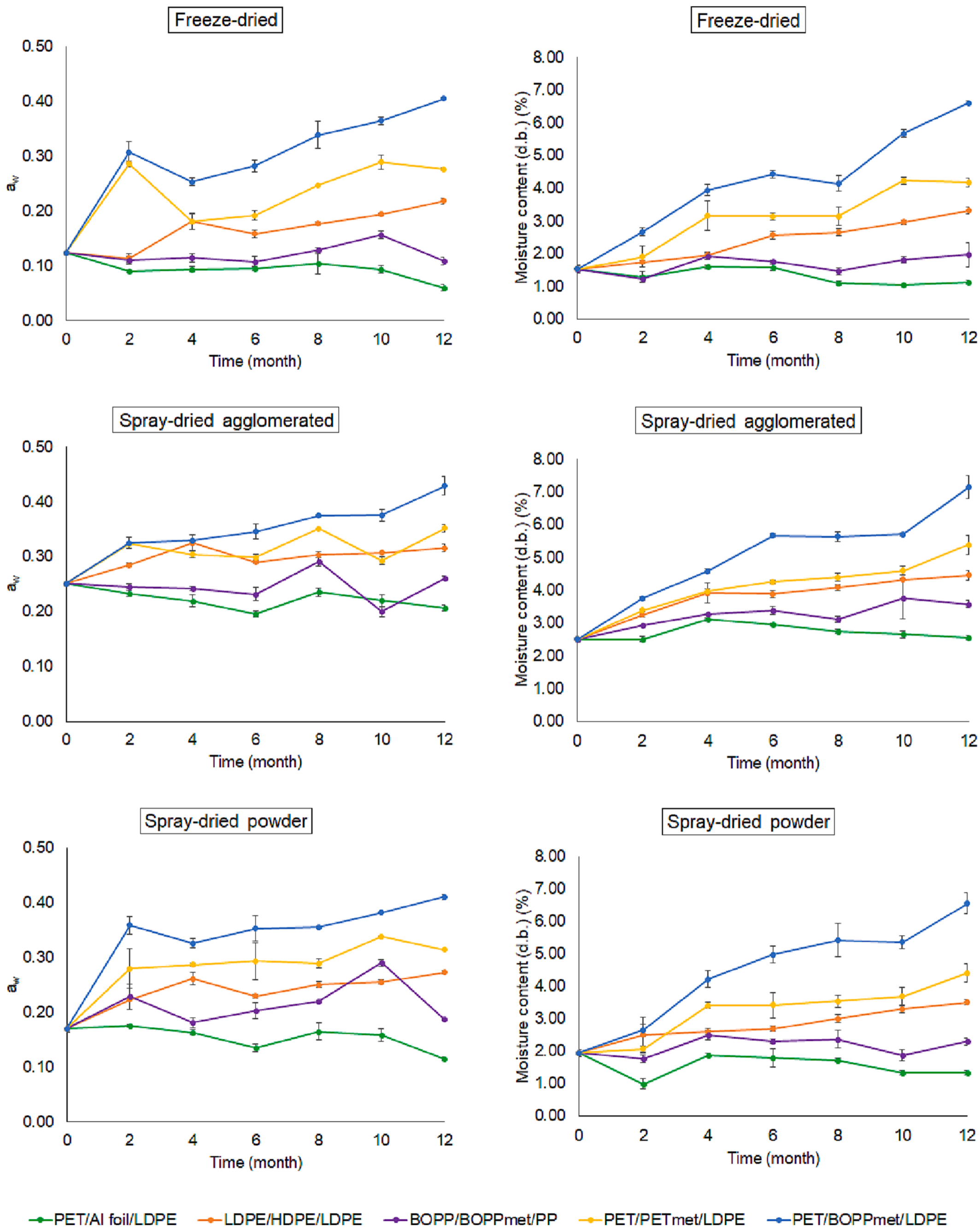

Fig. 1. Water activity and moisture content of instant coffees evaluated during 12 months at 25 °C/75% RH.

Concerning relative humidity Anese et al. (2005) reported that instant coffee stored at room temperature at equilibrium relative humidity lower than 35% is a glassy state while over this critical value, the glass-rubber transition might allow the initiation of matrix collapse.

3.2. Moisture sorption isotherm

Moisture sorption characteristics have an important role in the stability of dehydrated foods. The moisture sorption isotherm of food products at a particular temperature represents the non-linear relationship between moisture content and water activity (a_w) at equilibrium (Ishwarya & Anandharamakrishnan, 2015).

Fig. 3 illustrates the moisture sorption isotherm of products evaluated at 25 °C. All instant coffees presented an initial change in color characteristic aspect in green point. After this point the coffee aspect changes and the degradation continue until agglomeration, dissolution, and solubilization.

Visual evaluation showed instant coffees did not present changes until equilibrium moisture (d.b.) of 3.34% (freeze-dried); 6.63% (spray-dried agglomerated) and 6.76% (spray-dried powder). These values demonstrate the ease this type of food gains moisture. Robertson (2013) reported that absorption of moisture in soluble coffee is much more critical than in roasted and ground coffee, which can increase the agglomeration of particles when the humidity reaches 7% to 8%. The greater the adhesion, the higher the moisture content absorbed by the product. The high humidity speeds up the reactions of loss of quality associated with oxidation.

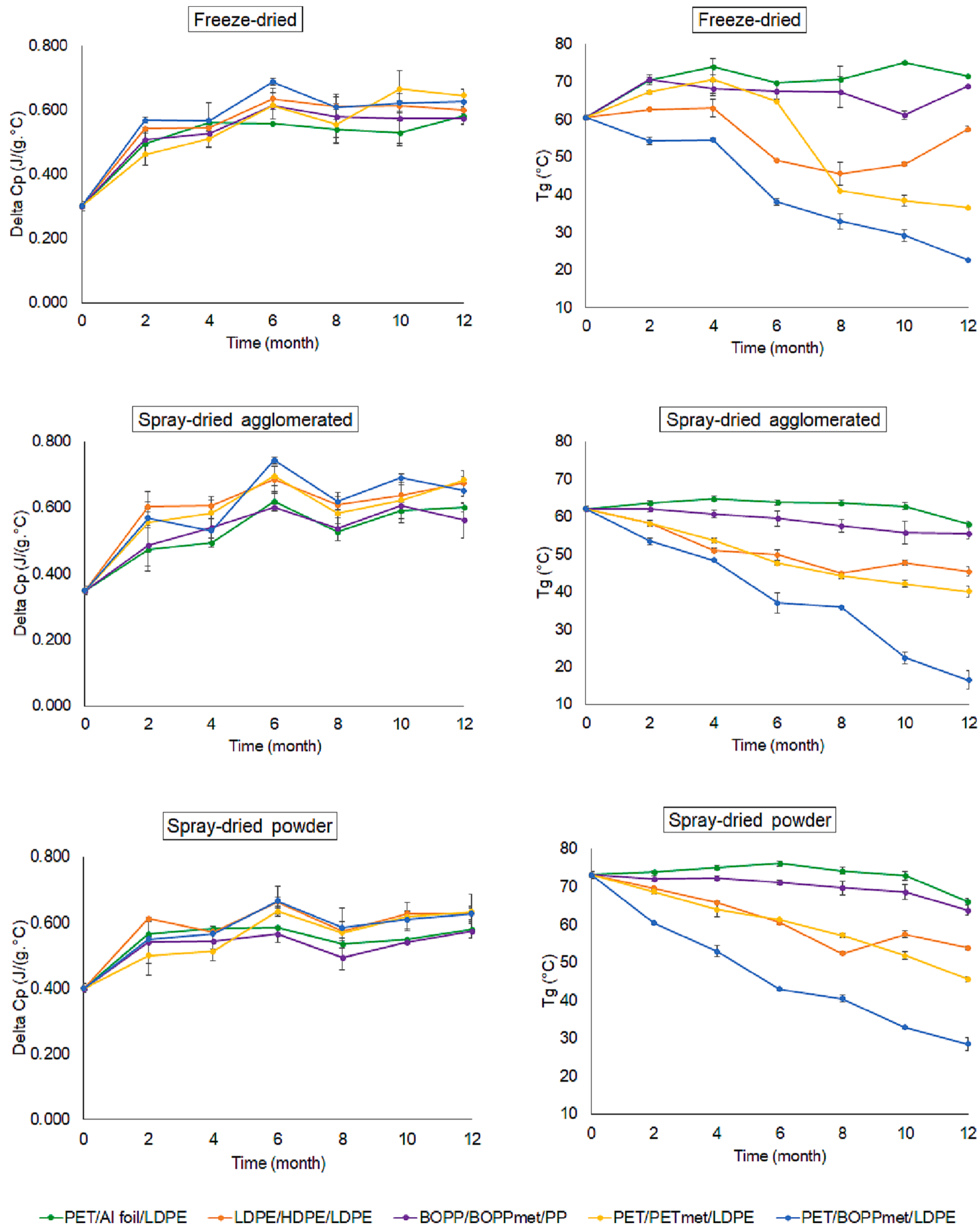

Fig. 2. Thermal properties (Tg and ΔCp) of the instant coffees evaluated during 12 months at 25 °C/75% RH.

3.3. Mathematical modeling

Moisture sorption isotherms of instant coffees are fitted to mathematical models. The results of the regression analysis for fitting the experimental data to the different models: GAB, Halsey and Henderson are summarized in Table 2.

According to Table 2, the GAB model showed the best profile (considering the entire range of water activity - a_w) to express the experimental data for all instant coffees evaluated. It is possible to observe this model provided the highest R^2 value, the lowest *% RMSE* value, and the lowest *E (%)*, considering each single case. According to Sormoli and Langrish (2015) *E* (%) value indicates good fitting when its value is lower than 10%, but only this model choice criterion cannot be considered exclusively. Moreover, although the parameters adjusted for the other models (Halsey & Henderson) can also be considered relatively satisfactory, the GAB model was chosen to simulate the moisture sorption isotherm profiles of this work because it is well-known this model is more suitable for values of a_w up to 0.9 (Marangoni Júnior et al., 2021), which would provide to estimate very accurate results at low values.

3.4. WVTR prediction for alternative plastic flexible packaging selection

Using data from adjusted equations by the GAB model and considering the critical moisture value defined for each instant coffee it was possible to predict the water vapor transmission rate (WVTR) required for alternative plastic flexible packaging selection. Table 3 shows WVTR predictions using critical parameters pre-established such as coffee weight in the packaging, flexible packaging area and time of storage with 5% and 7% critical moisture at storage conditions (25 °C/75% RH).

The critical parameters for predicting the WVTR were selected based

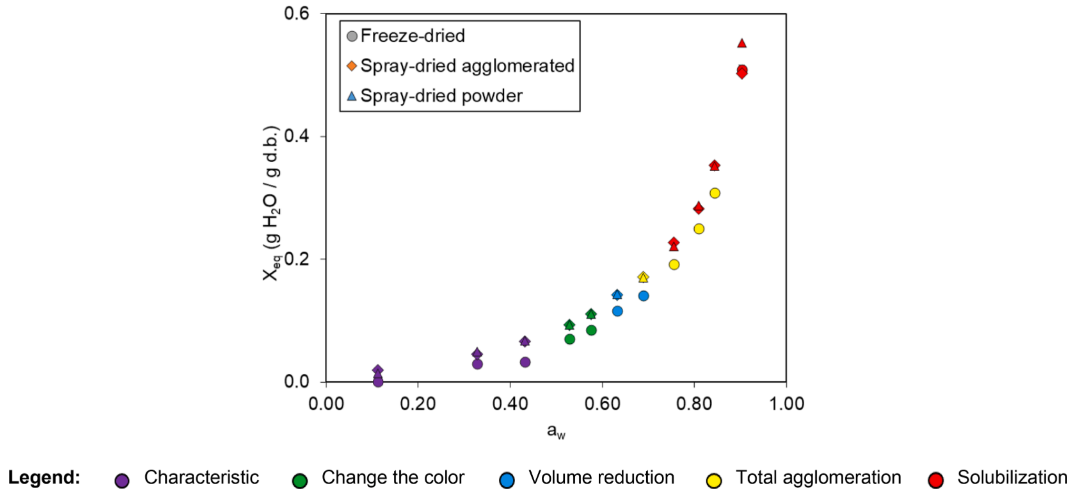

Fig. 3. Equilibrium moisture sorption isotherm of instant coffee at 25 °C.

Table 2
Moisture sorption isotherm of instant coffees at 25 °C fitted to mathematical models.

Identification	Model	Parameters C_1	C_2	C_3	*E (%)*	*RMSE (%)*	R^2
Freeze-dried	GAB	6.823	0.916	0.977	75.7	4.4	0.998
	Halsey	4.488	0.959	–	219.4	16.3	0.994
	Henderson	0.281	0.542	–	20.3	10.3	0.996
Spray-dried agglomerated	GAB	8.593	1.268	0.942	4.8	2.1	0.999
	Halsey	9.022	1.134	–	20.6	16.5	0.991
	Henderson	0.175	0.664	–	11.2	7.1	0.997
Spray-dried powder	GAB	6.768	2.003	0.980	2.5	0.7	0.999
	Halsey	7.263	1.060	–	16.4	7.6	0.997
	Henderson	0.205	0.612	–	15.9	19.8	0.993

C1, C2 and C3 – parameters of mathematical models; E - minimum mean absolute percentage error; RMSE - minimum percent root mean square error; R^2 - maximum adjusted coefficient of regression.

Table 3
WVTR prediction for alternative plastic flexible packaging selection.

Identification	Coffee weight (g)	Packaging area (m^2)	Time of the storage (days) 365		730	
			Critical moisture (%)			
			5	7	5	7
			WVTR predicted ($g.m^{-2}.d^{-1}$)			
Freeze-dried	50	0.0256	0.24	0.54	0.12	0.27
	100	0.0481	0.26	0.58	0.13	0.29
	200	0.0585	0.43	0.95	0.21	0.47
Spray-dried agglomerated	50	0.0256	0.17	0.37	0.09	0.19
	100	0.0481	0.19	0.39	0.09	0.20
	200	0.0585	0.31	0.65	0.15	0.32
Spray-dried powder	50	0.0256	0.19	0.36	0.10	0.19
	100	0.0481	0.20	0.39	0.11	0.20
	200	0.0585	0.33	0.64	0.17	0.33

on the characteristics of the packaging available in the market and the product requirements. Aluminum foil, present in PET/Al foil/LDPE traditional structure, is the barrier layer that gives protection against water vapor and gas penetration inside packaging and aroma loss. Based on information from producers and in the labels of the products, the shelf-life of soluble coffee varies from 1 year (365 days) up to 2 years (730 days) depending on the type of packaging in which it is packaged and the storage conditions.

The results presented at Table 3 shows that packaging area is the most critical parameter as the increase in weight of coffee is not proportional to the increase in the packaging area. For example, critical moisture of 5%: packaging area of 0.0256 m^2 and product weight of 50 g the minimum WVTR requested is 0.17 $g.m^{-2}.d^{-1}$ for 365 days shelf-life and 0.09 $g.m^{-2}.d^{-1}$ for 730 days shelf-fife. For critical moisture of 7% the minimum WVTR requested is 0.37 $g.m^{-2}.d^{-1}$ for 365 days shelf-life and 0.19 $g.m^{-2}.d^{-1}$ for 730 days shelf-life. Only for 200 g of product weight, the packaging area doubled compared to 50 g of product, then, WVTR estimated values also doubled.

This data indicates it is possible to use alternative flexible plastic packaging structures LDPE/HDPE/LDPE and BOPP/BOPPmet/PP for instant coffee keeping the quality of the product until the end of its shelf-life.

4. Conclusions

The best protection performances were observed for flexible plastic material structures: PET/Al foil/LDPE, PET/BOPP/BOPPmet/PP and LDPE/HDPE/LDPE. After 365 days of storage at 25 °C/75% RH, the three types of instant coffee evaluated (freeze-dried, spray-dried agglomerated and powder) presented adequate water activity, moisture and thermal properties in all packaging structures studied.

These results indicate that it is possible to use flexible packaging without aluminum foil that are recyclable.

The results obtained during the shelf-life storage at 25 °C/75% RH was in line with the obtained by GAB mathematical modeling when considering 5% as critical moisture.

CRediT authorship contribution statement

Raquel Massulo Souza: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Writing – original draft.

Christiane Quartaroli Moreira: Data curation, Formal analysis, Methodology. **Roniérik Pioli Vieira:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Leda Coltro:** Conceptualization, Data curation, Writing – review & editing. **Rosa Maria Vercelino Alves:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Funding acquisition, Project administration, Resources, Supervision, Writing – original draft, Writing – review & editing.

Declaration of Competing 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.

Data availability

No data was used for the research described in the article.

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