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# Instant coffee: Products and packaging systems characteristics

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## ABSTRACT

The moisture gain cause instant coffee quality loss. In Brazil, these products are marketed in glass containers, with seals and screw caps, metal cans with easy-open lid and LDPE (low density polyethylene) over cap and flexible plastic packaging of multilayer film: PET (polyethylene terephthalate)/Al (aluminum) foil/LDPE. The aim of this study was to evaluate twelve instant coffees of three different types (freeze-dried, spray-dried agglomerated and powder) and their packages regarding to water activity, initial moisture, thermal analysis and moisture sorption isotherm, as well as some characteristics of the packaging systems - gas composition of the headspace, oxygen and water vapor transmission rate. All instant coffees evaluated presented similar characteristics of quality standard described in international literature. Products showed quality loss appearance with 10%-12% (d.b.) moisture content. Packaging systems presented oxygen transmission rate values greater than 1.000mL (STP)$m^{-2}$.$day^{-1}$ and water vapor transmission rate values less than 0.017g of water.$package^{-1}$.$day^{-1}$ (25 °C/75%RH) and 0.048g of water.$package^{-1}$.$day^{-1}$ (38 °C/90%RH).

**Key words:** Soluble coffee; Packaging; Moisture sorption isotherms; WVTR; OTR.

## 1 INTRODUCTION

Coffee is the beverage most consumed worldwide for traditional reasons and healthiness due to the beneficial characteristics such as: stimulating, antioxidant, blood-protective and hypoglycemic action (Samoggia; Riedel, 2019). The aroma and flavor of roasted coffee are important attributes of acceptability. They are formed by a complex mixture of approximately three hundred volatile compounds and the partition of these components between the gas phase and food is relevant to the resulting quality that is passed to coffee products made with roasted coffee (Borém et al., 2021).

Instant coffees are the second coffee product most consumed worldwide. Also called soluble coffees, they are obtained from extraction with hot water freshly ground-roasted coffee beans (FSSAI, 2022). Currently, there are two conventional techniques to manufacturer instant coffees, i.e., spray drying and freeze-drying. Spray drying (SD) is the most economical process for commercial production but freeze-drying (FD) produces products with higher quality (Ishwarya; Anandharamakrishnan, 2015).

The quality loss of soluble coffee is mainly related to the gain of moisture. Normally, the moisture content of soluble coffee is 2% to 4% (d.b.), and the moisture gain is much more critical in this product than in roasted and ground coffee. Total particle agglomeration occurs when the moisture content reaches 7% to 8% (Robertson, 2016). The intrinsic factors that influence the instant coffee' shelf life causes chemical and physical changes as oxidation reactions and physical collapse (Manzocco et al., 2016; Jannah et al., 2022).

Due to the ease of solubilization of soluble coffees in aqueous media, their packaging must protect the product against moisture gain until their expiration date. In Brazil, instant coffees are marketed in glass containers, with seals and screw caps, metal cans with easy-open lid and plastic LDPE 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.

The flexible plastic packaging has lower production cost than the glass containers and presented higher mechanical resistance, which reduces product losses in filling lines, storage, point of sale and final disposal, but they do not have sustainable appeal such as packaging made from mono-material plastic and glass materials. Therefore, the aim of this study was to characterized instant coffees in Brazilian market considering their quality loss due to moisture gain and the protection characteristics of the packages used.

## 2 MATERIAL AND METHODS

### 2.1 Material

#### 2.1.1 Packaging Systems And Instant Coffee

Twelve spray-dried (agglomerated and powder) and freeze-dried instant coffees packaged in nine different packaging systems produced in Brazil and imported were evaluated (Table 1). The samples were acquired in the commerce of the city of Campinas/SP and the analyzes were carried out at the Packaging Technology Center of the Food Technology Institute (Cetea/Ital).

**Table 1:** Instant coffees and packaging systems evaluated.

| Identification | Process | Shape | Country | Packaging identification |
|---|---|---|---|---|
| 1 | Spray-drying | agglomerated | Brazil | A - Glass container - external diameter of 50 mm - 100 g Cold-bonded flexible laminate seal and PP twist cap |
| 2 | | powder | | |
| 3 | | powder | France | B - Glass container - external diameter of 48 mm - 100 g Heat-sealed flexible laminate seal and PP twist cap |
| 4 | | agglomerated | Brazil | C - Flexible packaging - stand up pouch - 50 g PET 13 μm/Al foil 12 μm/LDPE 55 μm |
| 5 | | | | |
| 6 | | powder | Brazil | D - Can - external diameter of 82 mm - tinplate material - aluminium seal - LDPE cap - 180 g |
| 7 | | powder | France | E - Can and cap - external diameter of 59 mm - tinplate material - aluminium seal - 90 g |
| 8 | Freeze-drying | | Brazil | F - Glass container - external diameter of 50 mm - 100 g Cold-bonded flexible laminate seal and PP twist cap |
| 9 | | | | |
| 10 | | | England | G - Glass container - external diameter of 48 mm - 100 g Heat-sealed flexible laminate seal and PP twist cap |
| 11 | | | Brazil | H - Glass container and cap with LDPE sealant - external diameter of 54 mm - 140 g |
| 12 | | | Brazil | I - Flexible packaging - stand up pouch - 50 g PET 13 μm/Al foil 12 μm/LDPE 55 μm |

## 2.2 Instant Coffee Characterization

### 2.2.1 Water activity

The water activity of instant coffees at 25 °C was determined using DECAGON - AquaLab series 4TEV Water Activity Meter hygrometer (DECAGON – AquaLab, USA) based on psychometry, with resolution of 0.0001.

### 2.2.2 Initial moisture content

The initial moisture content was determined at 70 °C by weighing samples (~3 g) in pre-weighted aluminum capsules, before and until constant weight in a vacuum chamber model VDL53 (WTB BINDER, Tuttlingen, Germany) (AOAC, 2019).

### 2.2.3 Thermal analysis

Thermal transitions were monitored using a Differential Scanning Calorimeter, model Discovery DSC 250 (TA Instruments, USA), with autosampler 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. 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 (Souza et al., 2023).

### 2.2.4 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 relative humidities: LiCl (11.3% RH), $MgCl_2$ (32.8% RH), $K_2CO_3$ (43.2% RH), Mg $(NO_3)_2$ (52.9% RH), NaBr (57.6% RH), $NaNO_2$ (63.3% RH), KI (68.9% RH), NaCl (75.4% RH), $(NH_4)_2SO_4$ (81.0% RH), KCl (84.3% RH) e $BaCl_2$ (90.3% RH) at 25 °C for 21 days (ASTM E104, 2020). Approximately 1 g of each material was weighed into weighs glass filters, 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.

## 2.3 Packaging Systems Characterization

### 2.3.1 Headspace gas composition

Headspace gas composition of the packaging was evaluated using a gas chromatograph from Agilent Technologies, model 7890A (Agilent, USA) operating with thermal conductivity detector at 150 °C, columns Molecular Sieve 13x and Porapak N at 40 °C and injector at 70 °C. The chromatographic results were processed by the Chemstation/Agilent program, version B 03.01. Gas was taken directly from headspace with a gas-tight syringe introduced into the packaging through a septum. Next, specific gas concentrations were determined qualitatively and quantitatively (Sarantopóulos et al., 2017).

### 2.3.2 Oxygen transmission rate (OTR)

OTR of the packages was evaluated in the OXTRAN equipment, model 2/60 (Ametek/MOCON, USA) at 23 °C and 0.21 atm of partial pressure of oxygen conditions (ASTM F1307, 2020).

### 2.3.3 Water vapor transmission rate (WVTR)

WVTR was determined by gravimetric method at 25 °C/75% RH and at 38 °C/90% RH conditions for rigid packages (ASTM F1249, 2020) and the PERMATRAN equipment, model W 3/33 (Ametek/MOCON, USA) at 38 °C/90% RH conditions were used for stand-up pouch packages (ASTM D4279, 2020).

### 2.4 Statistical Analysis

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

## 3 RESULTS

### 3.1 Instant Coffee Characterization

Table 2 presents the characteristics of instant coffees evaluated.

The most important factors for the stability of coffee products are water activity and moisture content. Freeze-drying coffees presented 0.202 - 0.326 water activity and moisture between 2.1 - 3.0% (d.b.) and the spray-drying coffees results had variation in the range of 0.186 - 0.461 $a_w$ and 2.3 - 4.0% (d.b.). No correlation among water activity and moisture content and the type of packaging and the production process was verified (at 95% confidence level ($p < 0.05$)).

The glass transition temperature (Tg) and the step-in heat capacity (ΔCp) are usually measured from the second heating to remove thermal history of the material and to obtain reliable values. The values of Tg recorded ranged from 35.8 °C up to 72.6 °C for spray-drying instant coffee and from 38.7 °C up to 67.3 °C for freeze-drying instant coffee.

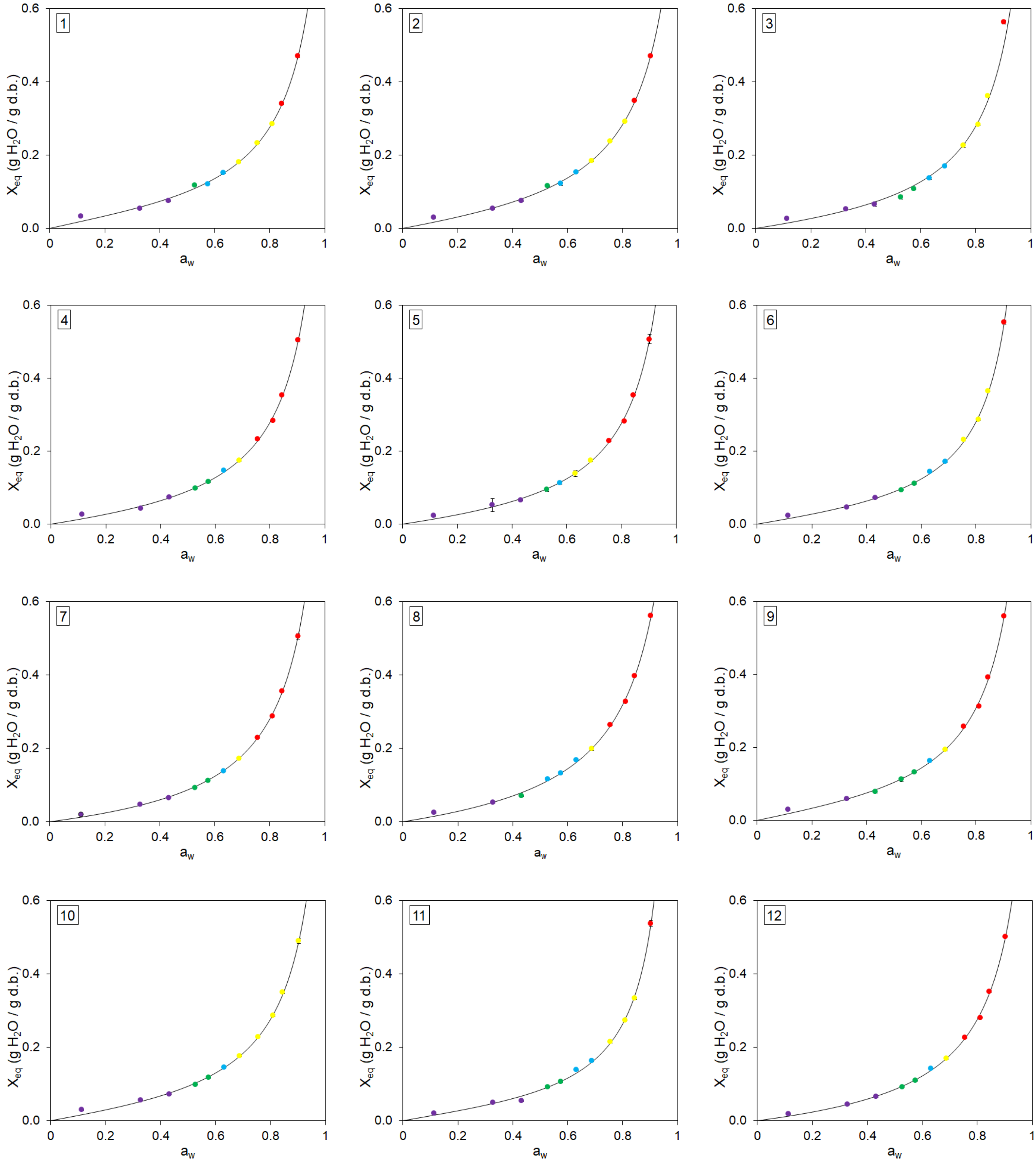
Figure 1 illustrates the moisture sorption isotherm performance of instant coffee evaluated at 25 °C.

According to Figure 1 all instant coffees presented an initial change in color characteristic aspect at a minimum 7% equilibrium moisture content (green point). After this point at water activity 0.4 - 0.5 $a_w$ and 10% - 12% (d.b.) equilibrium moisture content the coffee aspect changes and the degradation continues until agglomeration, dissolution, and solubilization. This value demonstrates the ease this type of food gains moisture.

**Table 2:** Instant coffees characterization.

| Identification | Water activity ($a_w$)* | Initial moisture (% dry basis)** | Glass transition*** | |
|---|---|---|---|---|
| | | | Tg (°C) | ΔCp ($g^{-1}.J^{-1}.°C^{-1}$) |
| Spray-drying instant coffee | | | | |
| 1 | $0.418 \pm 0.001^{b}$ | $4.0 \pm 0.0^{a}$ | 49.5 ± 3.8 | 0.520 ± 0.082 |
| 2 | $0.461 \pm 0.000^{a}$ | $3.0 \pm 0.1^{b}$ | 49.0 ± 1.0 | 0.375 ± 0.081 |
| 3 | $0.254 \pm 0.003^{e}$ | $2.6 \pm 0.0^{d}$ | 51.7 ± 1.0 | 0.386 ± 0.065 |
| 4 | $0.353 \pm 0.007^{c}$ | $3.0 \pm 0.0^{b}$ | 35.8 ± 1.2 | 0.630 ± 0.007 |
| 5 | $0.363 \pm 0.001^{c}$ | $2.8 \pm 0.0^{c}$ | 72.6 ± 0.7 | 0.447 ± 0.066 |
| 6 | $0.186 \pm 0.002^{f}$ | $2.7 \pm 0.1^{c}$ | 57.9 ± 1.5 | 0.449 ± 0.052 |
| 7 | $0.304 \pm 0.002^{d}$ | $2.3 \pm 0.1^{e}$ | 47.3 ± 0.6 | 0.416 ± 0.012 |
| Freeze-drying instant coffee | | | | |
| 8 | $0.204 \pm 0.004^{b}$ | $2.1 \pm 0.1^{e}$ | 62.3 ± 2.1 | 0.317 ± 0.005 |
| 9 | $0.326 \pm 0.003^{a}$ | $2.5 \pm 0.0^{c}$ | 41.5 ± 1.4 | 0.506 ± 0.033 |
| 10 | $0.220 \pm 0.002^{b}$ | $3.0 \pm 0.1^{a}$ | 48.7 ± 1.8 | 0.481 ± 0.146 |
| 11 | $0.216 \pm 0.002^{b}$ | $2.8 \pm 0.0^{b}$ | 67.3 ± 2.3 | 0.456 ± 0.023 |
| 12 | $0.202 \pm 0.015^{b}$ | $2.3 \pm 0.0^{d}$ | 38.7 ± 1.4 | 0.529 ± 0.021 |

Values referring to (*) two (**) five and (***) three determinations ± standard deviation.
Tg - Glass transition temperature and ΔCp - Heat capacity.
$^{a,b,c,d,e,f}$ the means, followed by the same letter, in the column for the same coffee production process, do not differ at the 95% confidence level ($p < 0.05$).
Packaging' characteristics and types of instant coffees are described in Table 1.




Legend: ● Characteristic ● Color Change ● Volume reduction ● Total agglomeration ● Solubilization

**Figure 1:** Equilibrium moisture sorption isotherm of instant coffee at 25 °C produced at spray dryer (1 - 7) and freeze dryer (8 - 12) by different companies.

Freeze-dryer products showed the most variable behavior, for example, coffees number 8 and 9 (Figure 1) presented color change at 0.43 $a_w$. These products were packaged in glass containers with cold-bonded flexible laminated seal and PP twist cap. Coffee number 10 presented total agglomeration at 0.69 $a_w$. This coffee was packed in modified atmosphere in glass container with heat-sealed flexible laminated seal and PP twist cap.

For spray-dryer coffees the same behavior could be observed for coffees numbers 1 and 2 - same packaging (glass containers with cold-bonded flexible laminated seal and PP twist cap); samples 3 and 6 - hermetical seal (heat-sealed flexible laminated seal and aluminum seal, respectively); samples 4 and 7 - aluminum barrier (flexible packaging - stand up pouch - PET/Al foil/LDPE and can cap tinplate material - aluminum seal, respectively). These results indicate that it is possible to establish a relationship between the influence of the type of product and the packaging system on the behavior of moisture gain of the instant coffee products.

### 3.2 Packaging Systems Characterization

Table 3 presents characteristics of packaging systems evaluated in this study.

The flexible plastic packaging (stand up pouch) - C and I - presented an excellent gas barrier. The soluble coffees from France (B and E) and England (G) presented a reduced percentage of $O_2$ and $CO_2$ gases and a higher percentage of $N_2$ gas in the headspace gas composition, indicating they have modified atmosphere. Brazilian instant coffee products are packaged in air.

Rigid packaging systems showed values of OTR > 1.000 mL (STP).m$^{-2}$.day$^{-1}$ indicating that $O_2$ inside packaging is not a critical parameter for instant coffee products if the moisture of the product does not increase.

Table 3 shows that glass containers with seal presented WVTR values up to 0.017 g of water. package$^{-1}$.day$^{-1}$ at Brazilian storage conditions and 0.048 g water. package$^{-1}$.day$^{-1}$ at European storage conditions. Both values were obtained for packaging system B. For packaging without seal, and when the seal is removed, the barrier properties of the packaging depend on if the dimensions of the cap/packaging finish produce a hermetic closure. Flexible plastic packages (C and I) presented an excellent water barrier with values lower than the quantification limit of the method/equipment.

## 4 DISCUSSION

### 4.1 Instant Coffee Characterization

Deotale et al. (2020) reported values of 0.41 $a_w$ for spray-drying instant coffee and 0.35 $a_w$ for freeze-drying instant coffee, which are similar to the data obtained in this study (Table 2).

**Table 3:** Packaging systems characterization.

| Packaging system | Headspace gas composition * (%) | | | OTR * (mL (STP). m$^{-2}$.day$^{-1}$) | WVTR** (g of water. package$^{-1}$.day$^{-1}$) | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | 25 °C/75% RH | | 38 °C/90% RH | |
| | $O_2$ | $N_2$ | $CO_2$ | | with seal and cap | with cap (without seal) | with seal and cap | with cap (without seal) |
| Spray-drying instant coffee | | | | | | | | |
| A | 15.0 ± 6.0 | 80.2 ± 4.5 | 2.6 ± 2.3 | 0.384 ± 0.078 | 0.001 ± 0.001 | 0.051 ± 0.024 | 0.005 ± 0.001 | 0.138 ± 0.074 |
| B | 3.2 ± 0.1 | 95.2 ± 1.2 | 2.8 ± 0.1 | <0.005*** | 0.017 ± 0.032 | 0.052 ± 0.017 | 0.048 ± 0.089 | 0.127 ± 0.037 |
| | | | | | WVTR** (g of water. m$^{-2}$.day$^{-1}$) at 38 °C/90% RH | | | |
| C | 16.3 ± 0.2 | 78.6 ± 0.1 | 3.6 ± 0.2 | <0.005*** | <0.01*** | | | |
| D | 19.2 ± 0.9 | 77.9 ± 0.4 | 2.1 ± 0.9 | <0.005*** | -- | 0.074 ± 0.018 | -- | 0.225 ± 0.044 |
| E | 9.9 ± 2.3 | 88.3 ± 2.6 | 1.9 ± 0.1 | <0.005*** | -- | 0.199 ± 0.038 | -- | 0.595 ± 0.153 |
| Freeze-drying instant coffee | | | | | | | | |
| F - 1 | 19.4 ± 2.3 | 74.2 ± 5.6 | 0.4 ± 0.4 | 0.780 ± 0.014 | 0.002 ± 0.001 | 0.010 ± 0.001 | 0.005 ± 0.001 | 0.032 ± 0.005 |
| F - 2 | 20.8 ± 0.1 | 78.2 ± 0.3 | 0 | > 1.000 | 0.004 ± 0.001 | 0.019 ± 0.003 | 0.010 ± 0.001 | 0.050 ± 0.003 |
| G | 2.5 ± 1.5 | 95.5 ± 1.3 | 1.4 ± 0.1 | <0.005*** | 0.003 ± 0.001 | 0.047 ± 0.012 | 0.002 ± 0.001 | 0.126 ± 0.024 |
| H | 20.4 ± 0.2 | 77.5 ± 0.6 | 0.6 ± 0.1 | <0.005*** | -- | 0.002 ± 0.001 | -- | 0.007 ± 0.005 |
| | | | | | WVTR** (g of water. m$^{-2}$.day$^{-1}$) at 38 °C/90% RH | | | |
| I | 17.4 ± 0.6 | 77.9 ± 0.4 | 2.9 ± 0.6 | <0.005*** | <0.01*** | | | |

Values referring to (*) three and (**) four determinations ± standard deviation; (***) quantification limit of the method / equipment.
Packaging system details on Table 1, F - 1 and F - 2 = same packaging system and different design.
Although the oxygen transmission rate is usually expressed in mL (STP).m$^{-2}$.day$^{-1}$, in the international system of units, it is expressed in mol. m$^{-2}$.s$^{-1}$, where 1 mL (STP) is equivalent to 44.62 μmol and 1 day is 86.4x103 s.

Soluble coffee produced by freeze-drying has a shelf-life longer than the product obtained by spray-drying process (Robertson, 2016), probably because the initial moisture of the freeze-drying product is lower (approx. 2%) than the atomized product (approx. 4.5%) (Labuza, 1982).

However, initial moisture experimental values obtained for products taken from the point of sale are greatly influenced by the type of packaging and the closure system. Moisture content plays a critical role in instant coffee products, being an instantized cohesiveness due to inter-particle liquid bridges leading to cacking and flow impairment (Barbosa-Canovas et al., 2005).

The Tg and ΔCp values do not depend on the process, these parameters are related to the product composition. For instance, Yu et al. (2012) reported Tg of 45 ± 1.0 °C and ΔCp of 0.34 $g^{-1}.J^{-1}.°C^{-1}$ for a freeze-dried instant coffee evaluated with a composition of 35 % of carbohydrate (mono and disaccharides), with the remaining 25 % being of long chain arabinogalactan and galactomannan.

The glass transition temperature of instant coffees increases by adding high molecular weight polysaccharides (Manzocco et al., 2016). Greater the difference between Tg and temperature of storage, more stable the product and then, the instant coffee collapses when Tg is overcome during storage. Concerning relative humidity Anese, Manzocco and Maltini (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 may allow the initiation of matrix collapse with confirm that temperature and relative humidity are critical parameters to soluble coffee.

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 at equilibrium (Ishwarya; Anan dharamakrishnan, 2015).

Brazilian Resolution RDC nº 277, which approves the Technical Regulation for coffee, barley, tea, yerba mate and soluble products (Brasil, 2005), establishes the specific quality requirements for this class of product, being maximum moisture content of 5% wet basis for instant coffee newly produced. Robertson (2016) reported that absorption of moisture in soluble coffee is much more critical than in roasted and grounded 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 oxidation associated with loss of quality. All instant coffees studied presented change in characteristic aspects from a minimum 7% equilibrium moisture content in agreement with the literature.

### 4.2 Packaging Systems Characterization

The use of a modified atmosphere is necessary to control the rate of oxidation in coffee derivates and improve the shelf life of the products. Despite being more usual for roasted whole and grounded coffee packages with and without an application of vacuum, modified atmosphere is usually used to reduce the oxygen level up to 1 - 2% by replacing the air inside packaging with inert gas, such as $N_2$ or $CO_2$ (Manzocco et al., 2016) as found in instant coffee packaging systems.

From the OTR results (Table 3), it was also possible to conclude that the best closer system for rigid packaging of soluble coffee is the heat-seal flexible laminate with aluminium foil on the finish of the glass packaging because offers barrier and hermeticity more efficient than cold-bond flexible laminate (Ortiz et al., 2000).

Jaime (2002) evaluated the changes in headspace gas composition in glass packages containing soluble coffee and different closure systems: seal composed of an ionomer and aluminium foil (heat sealed) and a flexible laminate (cold-bonded) both with plastic over caps. In two storage conditions, 23 °C/70% RH and 35 °C/80% RH, headspace gas composition typical of air (21% $O_2$) was not altered when the cold-seal was used, that does not allow the system to be closed tightly. When heat seal was used, they observed a reduction in $O_2$ level from 21% to 13% at 23 ºC/70% RH and from 21% to 2% at 35 ºC/80% RH. These results demonstrate soluble coffees consume oxygen during storage and achieve "zero" level after 9 and 12 months for spray-dryer and freeze-dryer coffees packaged in ambient air conditions, respectively (Harris et al., 1976).

WVTR of commercial packaging was evaluated in four situations at 25 °C/75% RH – Brazilian storage condition and at 38 °C/90% RH – European storage condition both with closure system complete (seal and cap) and without seal. WVTR is the most important parameter for instant coffee packaging due to the sensibility of the products to moisture uptake (Alves; Milanez; Padula, 2000) as discussed previously. Results obtained showed that the hermeticity and the closure system of rigid packaging and the integrity of the sealing of flexible plastic packaging are fundamental requirement to guarantee the barrier to water vapor for the systems regardless of storage condition.

## 5 CONCLUSIONS

In the present study, all instant coffees evaluated (spray-dried agglomerated and powder and freeze-dried) acquired in the Brazilian market presented characteristics (water activity, initial moisture and thermal analysis values) similar to standard quality reported in the literature. Moisture sorption isotherm indicated that products lost their characteristic appearance with 10% - 12% (d.b.) equilibrium moisture content.

Modified atmosphere was verified only in the headspace of imported products packaging. Packaging systems presented oxygen transmission rate values greater than 1.000 mL (STP) $m^{-2}.day^{-1}$ and water vapor transmission rate values less than 0.017g of water. $package^{-1}.day^{-1}$ at 25 °C/75% RH – Brazilian conditions and 0.048g of water.package-1.day-1 at 38 °C/90% RH – European conditions. The water vapor transmission rate of the rigid packaging without seal depends on cap' dimension tight with the packaging finish' dimensions to make a closure integrity after packaging opened.

The results of this study presented the protection requirements of different types of soluble coffees and which types of packaging/closing systems meet these needs.

## 6 AUTHORS' CONTRIBUTION

RMS wrote the manuscript, performed the experiment and conducted all statistical analyses, CQM performed the experiment and co-work the manuscript, and RMVA supervised, reviewed and approved the final version of the work.

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