

# Encapsulation of tomato juice by freeze-drying: physicochemical and antioxidant characterization and stability

Encapsulado de jugo de tomate mediante liofilización: caracterización fisicoquímica, antioxidante y estabilidad

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## ABSTRACT

Tomatoes are an important dietary source of lycopene, a natural antioxidant compound that contributes to reducing reactive oxygen species (ROS); however, the lack of a food matrix can result in their rapid degradation during storage. Therefore, the aim of this work was to evaluate the physicochemical, antioxidant, and stability properties of lycopene and  $\beta$ -carotene in encapsulated tomato juice (TJ) obtained by freeze-drying. Maltodextrin (MD) and gum arabic (GA) were used as wall materials. Two formulations were obtained: MD:GA:TJ (3:1:14) and MD:GA:TJ (1:3:14), and were both compared with three controls: MD:GA (3:1), MD:GA (1:3), and freeze-dried TJ without wall materials. The physicochemical properties, lycopene, and  $\beta$ -carotene content were evaluated; also, antioxidant activity was measured by DPPH and ABTS assays. Carotenoid stability during storage was also evaluated. The encapsulates presented maximum concentrations of lycopene and  $\beta$ -carotene of 17.86 and 1.90 mg/100 g, respectively, and an antioxidant activity that varied from 5.14 to 20.98  $\mu$ M Trolox/g. Good hydration, water solubilization, and color properties were observed in the encapsulates. The MD:GA:TJ (3:1:14) treatment presented the best antioxidant and stability characteristics during storage.

## KEYWORDS

Carotenoids, encapsulate, freeze-drying, stability, tomato juice.

## RESUMEN

Los tomates son una importante fuente dietética de licopeno, un compuesto antioxidante natural que contribuye a reducir las especies reactivas de oxígeno (ROS); sin embargo, sin una matriz alimentaria puede resultar en su rápida degradación durante el almacenamiento. Por lo tanto, el objetivo del presente trabajo fue evaluar las propiedades fisicoquímicas, antioxidantes y estabilidad del licopeno y  $\beta$ -caroteno en encapsulados de jugo de tomate (TJ) obtenidos mediante liofilización. Se utilizaron maltodextrina (MD) y goma arábica (GA) como materiales de pared. Se obtuvieron dos formulaciones: MD:GA:TJ (3:1:14) y MD:GA:TJ (1:3:14), y se compararon con tres controles, MD:GA (3:1), MD:GA (1:3) y TJ liofilizado sin encapsular. Se evaluaron propiedades fisicoquímicas, contenido de licopeno y  $\beta$ -caroteno, actividad antioxidante mediante los en-

sayos de DPPH y ABTS y estabilidad de los carotenoides durante el almacenamiento. Los encapsulados presentaron concentraciones máximas de licopeno y  $\beta$ -caroteno de 17.86 y 1.90 mg/100 g, y una actividad antioxidante que varió de 5.14 a 20.98  $\mu$ M Trolox/g. Se observaron buenas propiedades de hidratación, solubilización en agua y de color en los encapsulados. El tratamiento MD:GA:TJ (3:1:14) presentó las mejores características antioxidantes y de estabilidad durante el almacenamiento.

#### **PALABRAS CLAVE**

Carotenoides, encapsulado, estabilidad, jugo de tomate, liofilización.

## **INTRODUCTION**

Tomato (*Solanum lycopersicum* L.) and tomato products and by-products provide high amounts of antioxidant compounds for the human diet. Their regular consumption has been correlated with a lower risk of developing various types of cancer and cardiovascular diseases. The intake of tomato benefits human health because of its high content of bioactive compounds, mainly lycopene,  $\beta$ -carotene, phenolic compounds, and ascorbic acid (Borguini and Da Silva Torres 2009; Naeem et al. 2018). Lycopene and  $\beta$ -carotene are reported to be the main micronutrients responsible for the antioxidant effect of tomato products (Raffo et al. 2006). These antioxidant properties can inhibit the action of singlet oxygen ( $^1O_2$ ), inhibit lipid oxidation, and scavenge peroxy radicals (ROO $\cdot$ ) (Oroian and Escriche 2015; Rosales et al. 2011). However, their low solubility in water (hydrophobicity), chemical instability, and low bioavailability limits their use as nutraceutical ingredients for food supplementation and fortification. Moreover, they show high instability and susceptibility to physical and chemical degradation during processing and/or storage due to chemical, mechanical, and thermal effects (Gul et al. 2015; Kha et al. 2014; Gutiérrez et al. 2019).

Encapsulation has been reported to be an effective method of overcoming these limitations. It improves the bioavailability, water solubility, and stability of hydrophobic compounds, allowing for the controlled release of active agents. It consists of enclosing a core material within an impermeable membrane (wall matrix) that protects it from mechanical stress, temperature, light, oxygen diffusion, and other pro-oxidants (Gul et al. 2015; Kha et al. 2014; Tolun et al. 2016). The most used wall materials to encapsulate fruit juices are maltodextrins with various dextrose equivalent (DE) values and gum arabic. These

biopolymers have good properties as encapsulating agents, in addition to biocompatibility, high solubility, low viscosity, and low toxicity (Ferrari et al. 2013; Oberoi and Sogi 2015; Tolun et al. 2016). Several works have reported the encapsulation of lycopene and  $\beta$ -carotene from fruits such as watermelon (Gomes et al. 2014; Oberoi and Sogi 2015; Quek et al. 2007), pink guava, spiny bitter melon (Kha et al. 2014), and tomato (Chiu et al. 2007; Goula and Adamopoulos 2005; Souza et al. 2018). These studies have focused on encapsulation efficiency, carotenoid content, and storage stability, among other characteristics of the encapsulated formulations. Gomes et al. (2014) evaluated the antioxidant activity in microcapsules of watermelon juice by spray-drying and found values of 6.43  $\mu$ M Trolox/g. Meanwhile, Souza et al. (2018) found antioxidant activity values of 6.18 to 27.24  $\mu$ M Trolox/g in microencapsulates of concentrated tomato juice obtained by atomization. There are works about the encapsulation of lycopene or tomato concentrates; however, there are no reports regarding the encapsulation of tomato juice by freeze-drying and its effect on the concentration of carotenoids and its antioxidant activity. Therefore, the aim of the present research was to obtain encapsulates of tomato juice by freeze-drying using maltodextrin and gum arabic as wall materials and to evaluate their effect on the physicochemical properties, antioxidant activity, and stability of lycopene and  $\beta$ -carotene during storage.

## **MATERIALS AND METHODS**

### **Plant materials and chemicals**

Tomato fruits (*S. lycopersicum* var. Saladette) were obtained from greenhouses located in Tulancingo, Hidalgo, Mexico. Spray-dried gum arabic (GA)

(Cedrosa, S.A. de C.V., Naucalpan, Mexico, Mexico) and 28-32 DE maltodextrin (MD) (Reasol S.A. de C.V., CDMX, Mexico) were used as wall materials. Potassium persulfate, hexane, ethanol, and acetone were purchased from J.T. Baker S.A. de C.V. (Avantor Performance Materials, Ecatepec, Estado de México, Mexico). 2,2'-diphenyl-1-picrylhydrazyl (DPPH•), Trolox (6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid), and ABTS [2,2'-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid)] were purchased from Sigma-Aldrich (Sigma-Aldrich Química S.A. de C.V., Toluca, Estado de México, Mexico).

### Tomato juice encapsulation

Tomato fruits were washed, disinfected with a solution of sodium hypochlorite (250 ppm), and grounded (Osterizer blender, model BLSTBC4129-013, Mexico) without water addition, to obtain the juice (TJ). This was subjected to sonication (Ultrasonic Cleaner, Mod. 32V118A, Illinois, USA) for 20 min at 30°C at a frequency of 40 kHz. TJ was mixed with the wall materials using an Ultraturrax homogenizer (Ika T25, DS1, Germany) at 13,500 rpm for 5 min. The mixture was ultra-frozen (Ultra-freezer Thermo Scientific 303, USA) for 72 h and freeze-dried at  $133 \times 10^{-3}$  mBar and -40°C (freeze-drier model 79480 Labconco, Missouri, USA) to obtain two formulations: MD:GA:TJ (3:1:14) and MD:GA:TJ (1:3:14). Both formulations were compared with two controls (without TJ), MD:GA (3:1) and MD:GA (1:3), and with freeze-dried TJ without wall materials. Once freeze-dried, formulations and controls were milled in a blade mill (Grindomix, Retsch GM 200, Germany) at 500 rpm until a 150 µm powder was obtained.

**Encapsulation efficiency (EE).** Encapsulation efficiency was determined as a function of the retention of lycopene and β-carotene content in the encapsulates using equations 1 and 2:

$$\text{Encapsulated lycopene (\%)} = \frac{\text{total lycopene} - \text{superficial lycopene}}{\text{total lycopene}} \times 100 \quad (1)$$

$$\text{Encapsulated } \beta - \text{carotene (\%)} = \frac{\text{total } \beta - \text{carotene} - \text{superficial } \beta - \text{carotene}}{\text{total } \beta - \text{carotene}} \times 100 \quad (2)$$

The content of superficial lycopene and β-carotene was determined according to Kha et al. (2014). Briefly, 0.1 g of powdered sample was mixed with 10 mL of n-hexane under gentle agitation for 10 min to avoid the disruption of the capsules. The solvent was poured and used to determine the concentration of lycopene and β-carotene, according to Fish et al. (2002).

### Lycopene and β-carotene content

Lycopene and β-carotene content was determined spectrophotometrically according to Fish et al. (2002) with slight modifications. A sample of 0.1 g was dissolved in 10 mL of hexane and vortexed for 5 min. This mixture was sonicated (VEVOR Ultrasonic Cleaner Mod. 32V118A, Illinois, USA) for 40 min using pulses of 10 min with a 5-min pause at a frequency of 40 kHz. Then, samples were centrifuged at  $15,000 \times g$  for 10 min (Thermo Electron LED GmbH, Mod. ST 16R, Osterode am Harz, Germany). Lycopene and β-carotene concentration in supernatants (hexane phase) were spectrophotometrically determined. Lycopene and β-carotene standard curves were done at 503 and 478 nm, respectively.

### Antioxidant activity

Antioxidant activity was determined using the DPPH (Brand-Williams et al. 1995) and TEAC (Re et al. 1999) methods. A 0.1g sample was obtained and dissolved. Later, the samples were dissolved in 10 mL of ethanol and vortexed for 5 min. Then, samples were sonicated for 40 min at 10 min pulses with 5-min pauses at a frequency of 40 kHz. These samples were centrifuged at  $15,000 \times g$ , 10 min. Supernatants were collected for antioxidant activity determination. For the DPPH assay, 0.3 mL of supernatant was added to 2.7 mL of a DPPH• cold ethanolic solution  $6 \times 10^{-5}$  M and allowed to stand in complete darkness for 60 min at 4°C; absorbance was measured at 515 nm. For the TEAC assay, the ABTS<sup>•+</sup> radical cation was obtained by the reaction of ABTS with 2.45 mM potassium persulfate and incubated at 25°C in the darkness for 16 h. Once formed, ABTS<sup>•+</sup> was diluted with ethanol until an absorbance of  $0.700 \pm 0.02$  at 734 nm was achieved. The ethanolic tomato extract and diluted ABTS<sup>•+</sup> were mixed and stored for 6 min, and the absorbance was measured at 734 nm. The results

of antioxidant activity were expressed in  $\mu\text{M}$  Trolox equivalents per g DW.

### Characterization of encapsulated tomato juice

**Moisture content.** Moisture content was determined gravimetrically in a drying oven at  $105^\circ\text{C}$  until constant weight was achieved (AOAC 2006). Moisture content was expressed as the percentage of weight loss before and after drying.

**Water activity.** Water activity ( $a_w$ ) was measured using an Aqualab instrument (Aqua Lab 4TE, Decagon Devices, United States of America), at  $25 \pm 5^\circ\text{C}$ .

**Hygroscopicity.** Hygroscopicity was determined according to Fernandes et al. (2014). 1 g of sample was placed in a desiccator containing a saturated solution of NaCl (75% relative humidity [RH]) at  $25^\circ\text{C}$  for a week. Afterward, the samples were weighed to determine the adsorbed moisture content. Hygroscopicity was defined as the weight of water in grams per 100 g of dry sample (g/100 g).

**Water solubility index and water absorption index.** The water solubility index (WSI) and water absorption index (WAI) were determined according to Ahmed et al. (2010). Samples of each powder (1 g per sample) were dissolved in 12 mL of distilled water. Then, samples were incubated in a water bath at  $30^\circ\text{C}$  for 30 min (Doihan Labtech CO, LTD, Mod. LCB-11D, Kyonggi, Korea) and centrifuged at  $10,000 \times g$  for 10 min. The supernatant was dried at  $105^\circ\text{C}$  (Thermo Electron LED GmbH, VWR Oven Mod. 89511-404, Langensfeld, Germany) until constant weight was achieved. WSI and WAI were calculated according to equations 3 and 4, where  $DW_{SUP}$  is the dry weight of the supernatant,  $DW_{IE}$  is the initial weight of the encapsulated (dry basis), and  $W_{SED}$  is the weight of the sediment after centrifugation.

$$WSI = \frac{DW_{SUP}}{DW_{IE}} \times 100 \quad (3)$$

$$WAI = \frac{W_{SED}}{DW_{IE}} \quad (4)$$

**Bulk density.** Bulk density was determined according to Fernandes et al. (2014). Five grams of powdered sample were transferred into a 25 mL graduated cylinder. Afterward, the test tube was gently tapped so that the encapsulates were compacted under their own weight until volume measurements with insignificant differences were obtained. The weight of the powder was divided by the volume that it occupied in the cylinder ( $\text{g}/\text{cm}^3$ ).

**Color.** Color attributes ( $L^*$ ,  $a^*$ , and  $b^*$  values) of the samples of each powder were measured using a HunterLab colorimeter (Minolta, CM508d, Minolta Camera. Co., Ltd., Osaka, Japan). The  $a^*$  (red-green) and  $b^*$  (yellow-blue) values were used to calculate chroma ( $C^*$ ) values and hue angle ( $h^\circ$ ), using equations 5 and 6.

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (5)$$

$$h^\circ = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (6)$$

### Degradation kinetics of lycopene, $\beta$ -carotene

For the assessment of stability, the tomato encapsulates were stored in resealable, opaque, 200-gauge polyethylene bags at  $25 \pm 0.5^\circ\text{C}$  and  $4 \pm 0.5^\circ\text{C}$ . The lycopene and  $\beta$ -carotene content were measured every 5 days for 40 days. The degradation kinetics were determined using the first-order reaction model and according to the following equation (7), where  $C$  is the carotenoid concentration ( $\text{mg}/100 \text{ g}$ ) at time  $t$ ,  $C_0$  is the initial carotenoid concentration ( $\text{mg}/100 \text{ g}$ ),  $t$  is time in days, and  $k$  is the reaction rate constant.

$$\ln\left(\frac{C}{C_0}\right) = -kt \quad (7)$$

### Statistical analysis

The data obtained were analyzed using a completely randomized experimental design with five replicates

and a test of multiple comparisons of Tukey means with  $P \leq 0.05$ . The SAS v.9.4 software was used for all analyses.

## RESULTS AND DISCUSSION

### Encapsulation efficiency

Table 1 shows the encapsulation efficiency (EE) of the main carotenoids present in tomato juice. In general, it is observed that the MD:GA:TJ (3:1:14) treatment presents a higher content of lycopene and  $\beta$ -carotene within the encapsulates with respect to the MD:GA:TJ treatment (1:3:14). Through these results, it can be observed that the higher the concentration of maltodextrin in the wall material, the more significantly improved is the encapsulation of the carotenoids. On the other hand, it has been reported that MD facilitates the encapsulation of fruit juices because the  $\beta$ -D-glucose units reduce the problems of stickiness and agglomeration (Oberoi and Sogi 2015; Quek et al. 2007). In addition, MD has good properties to protect core materials from oxidative damage, such as oils and oleoresins (Fuentes-Ortega et al. 2017). This may explain a higher EE of the MD:GA:TJ (3:1:14) treatment compared to the MD:GA:TJ (1:3:14) treatment. However, the proportion of GA in the wall materials offers good emulsifying properties, facilitating the protection of lipid substances (Cano-Higuera et al. 2015; Lourenço et al. 2020). On the other hand, the presence in the wall materials also favors the encapsulation of tomato juice. Some studies have reported that maltodextrin-arabic gum give better results in the EE of bioactive compounds than separately. It has even been reported that, together, MD-GA can support a load concentration of up to 30% (Adejoro et al. 2019; Tolun et al. 2016).

Also, Adejoro et al. (2019) evidenced the adherence of MD-GA on acacia extracts in layered forms as a coating of the extract. Moreover, according to the results, a higher content of lycopene was encapsulated than  $\beta$ -carotene. On the other hand, the findings of this research are very similar to those reported in the literature where lycopene and  $\beta$ -carotene from tomato and other sources such as gac oil and watermelon have been encapsulated by freeze-drying and spray-drying (Goula and Adamopoulos 2005; Kha et al. 2014; Oberoi and Sogi 2015).

### Lycopene and $\beta$ -carotene content

The content of lycopene and  $\beta$ -carotene in the encapsulate of tomato juice is observed in Table 2. The results show a higher concentration of these carotenoids in freeze-dried tomato juice compared to encapsulated tomato; however, it should be considered that wall materials contribute to the total solids content by reducing the concentration of the encapsulated material. On the other hand, in the MD:GA:TJ (3:1:14) treatment, a significantly higher concentration of lycopene and  $\beta$ -carotene was found with respect to the MD:GA:TJ treatment (1:3:14). This difference can be explained by the properties of the wall materials, as already mentioned. Some similar studies (Quek et al. 2007) have obtained lycopene and  $\beta$ -carotene concentrations of 72.44-95.40 mg/100 g DW and 3.14-2.30 mg/100 g DW, respectively, in spray-dried watermelon juice. Meanwhile, Oberoi and Sogi (2015) reported a lycopene content of 62.3 mg/100 g in freeze-dried watermelon powder. The authors of both studies used MD as the encapsulating agent at maximum concentrations of 5% and 10%, respectively. On the other hand, Gomes et al. (2014) found 22.89 mg/100 g DW of lycopene in watermelon juice microcapsules,

**Table 1. Encapsulation efficiency (EE%) of tomato juice.**

| Mixture           | Encapsulated lycopene (%)     | Encapsulated $\beta$ -carotene (%) |
|-------------------|-------------------------------|------------------------------------|
| MD:GA:TJ (3:1:14) | 97.81 $\pm$ 0.23 <sup>a</sup> | 85.73 $\pm$ 0.42 <sup>a</sup>      |
| MD:GA:TJ (1:3:14) | 77.08 $\pm$ 0.57 <sup>b</sup> | 63.64 $\pm$ 1.25 <sup>b</sup>      |

Data are expressed as mean value  $\pm$  standard deviation ( $n = 5$ ). Values tagged with the same letter in each column showed no significant differences according to the Tukey test with  $P \leq 0.05$ . MD: maltodextrin; GA: gum arabic; TJ: tomato juice.

**Table 2. Lycopene and  $\beta$ -carotene content and antioxidant activity as measured by DPPH and ABTS assays in encapsulated tomato juice obtained by freeze-drying.**

|   | Mixture                       |                               |                              |                              |                               |
|---|-------------------------------|-------------------------------|------------------------------|------------------------------|-------------------------------|
|   | MD:GA:TJ<br>(3:1:14)          | MD:GA:TJ<br>(1:3:14)          | MD:GA (3:1)                  | MD:GA (1:3)                  | TJ                            |
| <b>Lycopene</b><br>(mg/100 g DW)                    | 17.86 $\pm$ 0.09 <sup>b</sup> | 16.15 $\pm$ 0.20 <sup>c</sup> | -                            | -                            | 24.22 $\pm$ 0.18 <sup>a</sup> |
| <b><math>\beta</math>-carotene</b><br>(mg/100 g DW) | 1.90 $\pm$ 0.07 <sup>b</sup>  | 1.57 $\pm$ 0.10 <sup>c</sup>  | -                            | -                            | 3.17 $\pm$ 0.14 <sup>a</sup>  |
| <b>DPPH</b><br>( $\mu$ M Trolox/g DW)               | 10.38 $\pm$ 0.84 <sup>b</sup> | 5.14 $\pm$ 0.22 <sup>c</sup>  | 0.27 $\pm$ 0.01 <sup>d</sup> | 0.20 $\pm$ 0.01 <sup>d</sup> | 16.10 $\pm$ 0.32 <sup>a</sup> |
| <b>ABTS</b><br>( $\mu$ M Trolox/g DW)               | 20.98 $\pm$ 0.36 <sup>a</sup> | 10.31 $\pm$ 0.21 <sup>b</sup> | 0.45 $\pm$ 0.02 <sup>c</sup> | 0.40 $\pm$ 0.01 <sup>c</sup> | 20.80 $\pm$ 0.54 <sup>a</sup> |

Data are expressed as mean value  $\pm$  standard deviation ( $n = 5$ ). Values tagged with the same letter in each row showed no significant differences according to the Tukey test with  $P \leq 0.05$ . MD: maltodextrin; GA: gum arabic; TJ: tomato juice.

using MD and GA at a concentration of 22% solids as wall materials. It is important to consider that in this research, the final concentration of solids was set at 30%, so that large amounts of encapsulating agents diluted the concentration of bioactive compounds in the final products.

### Antioxidant activity

The antioxidant activity of the samples was determined according to their capacity to scavenge the DPPH $\cdot$  and ABTS $^{2+}$  radicals. The results are shown in Table 2. According to the ABTS assay, the freeze-dried tomato juice, and the MD:GA:TJ (3:1:14) encapsulates present higher antioxidant activity with respect to the MD:GA: TJ treatment (1:3:14). On the other hand, as expected, the wall materials show low antioxidant activity by both assays. Regarding the DPPH assay, the highest anti-radical activity of the DPPH $\cdot$  radical was registered in the freeze-dried TJ, followed by treatments MD:GA:TJ (3:1:14) and MD:GA:TJ (1:3:14), presenting significant differences between them. In general, greater antioxidant activity is observed in treatment MD:GA:TJ (3:1:14) with respect to MD:GA:TJ (1:3:14). These results can be attributed to a higher concentration of MD and GA in the first and second treatments, respectively. This behavior could be explained by considering the presence of other bioactive compounds in the tomato juice as phenolic compounds and vitamin C, as these molecules have well-documented antioxidant properties, along with lycopene and  $\beta$ -carotene (Naeem et al. 2018; Raffo et al. 2006; Rosales et al. 2011). These substances were

more susceptible to encapsulation in wall material mixtures containing a higher MD proportion. Some studies available where phenolic compounds were encapsulated showed that MD provided a higher encapsulation capacity and stability than GA (Ferrari et al. 2013). Tolun et al. (2016) found that an MD:GA 8:2 ratio gave better results than a 6:4 ratio for encapsulating phenolic compounds. On the other hand, the samples show a higher antioxidant activity by the ABTS assay than by the DPPH assay. According to the literature, the ABTS assay can measure the antioxidant activity of compounds of a hydrophilic and lipophilic nature. Meanwhile, DPPH has a higher affinity for hydrophilic compounds (López-Palestina et al. 2019). Therefore, the difference observed between ABTS and DPPH may be due to the antioxidant activity provided by the content of carotenoids present in the encapsulates. In addition, it has been reported that carotenoids, due to their ability to inhibit the action of singlet oxygen, decrease lipid oxidation and scavenge free radicals (Oroian and Escriche 2015; Rosales et al. 2011). Finally, the values found in the samples are similar to those reported in the literature; Gomes et al. (2014) found 6.43  $\mu$ mol Trolox/g in lycopene-rich encapsulated watermelon juice. Meanwhile, Souza et al. (2018) report values of 6.2 and 27.2  $\mu$ mol Trolox/g in tomato concentrate microencapsulated by atomization.

### Characterization of encapsulated tomato juice

Table 3 shows the moisture content of powders obtained in this research. The results of the moisture analysis of the freeze-dried powders ranged from 1.15

to 7.87%. Significantly, the highest moisture values were found in tomato juice powders and the lowest in polymer mixtures MD:GA (3:1) and MD:GA (1:3). On the other hand, no significant differences were found with respect to this parameter between the MD:GA:TJ (3:1:14) and MD:GA:TJ (1:3:14) treatments. Moreover, the data show that the addition of MD-GA to tomato juice reduces the moisture content in the final product. This is due to the increase of solids in the tomato juice before the drying process; therefore, the water content is reduced to evaporate during freeze-drying (Kha et al. 2014; Quek et al. 2007). Moreover, by reducing the moisture of tomato juice through the effect of biopolymers addition as wall material, its stability against microbiological attack and lipid oxidation can be increased (Edris et al. 2016). The moisture content values found in this study are similar to those reported in other research, in which tomato juice has been encapsulated by spray-drying and watermelon juice by freeze-drying (Oberoi and Sogi 2015; Quek et al. 2007). In addition, the moisture values reported herein were below 4%, which is the minimum specification for a food-grade product (Edris et al. 2016). Lower moisture values in the encapsulated are desirable to prevent stickiness, high particle viscosity, and powder agglomeration. Agglomeration could cause the encapsulated to collapse, reducing its capacity to retain the active material and leading to its oxidation (Fernandes et al. 2014).

The analysis of the water activity in the tomato juice encapsulates is shown in Table 3. The non-encapsulated tomato juice samples present significantly higher  $a_w$ . On the other hand, the  $a_w$  behavior in the tomato encapsulates was significantly higher in the MD:GA:TJ (1:3:14) treatment than in the MD:GA:TJ (3:1:14) encapsulates. According to Alves et al. (2014), the use of maltodextrin in encapsulation is effective in reducing  $a_w$  values because it reduces the hygroscopicity of the final powders. On the other hand, the  $a_w$  results of this study are similar to those reported in encapsulated fruit juices with high lycopene content. Oberoi and Sogi (2015) reports  $a_w$  values from 0.153 to 0.439, while Quek et al. (2007) report amounts from 0.22 to 0.29. The  $a_w$  results of tomato encapsulates also suggest stability against biochemical reactions and microbial growth, positively impacting the shelf life of powders (Kha et al. 2014)

The results of the evaluation of the hygroscopicity parameter are shown in Table 3. According to the analysis, the encapsulates of tomato juice of the treatments MD:GA:TJ (3: 1:14) and MD:GA:TJ (1:3:14) showed significantly higher hygroscopicity, followed by non-encapsulated tomato juice, while the mixture of the biopolymers presented the lowest values. In this research, it was expected that, when the biopolymers were added to tomato juice, the hygroscopicity values would decrease; however, these increased. This may be due to the lower moisture content in MD:GA:TJ (3:1:14) and MD:GA:TJ (1:3:14) compared to freeze-dried tomato juice. Some studies have reported that low-moisture powders present higher hygroscopicity values (Tonon et al. 2008; Fernandes et al. 2014), as could be observed in this research. In addition, the TJ is high in carotenoids that are of non-polar origin (Gutiérrez et al. 2019; López-Palestina et al. 2019) and therefore, the TJ absorbs less water. On the other hand, the hygroscopicity values reported in this study were like those found in studies in which MD:GA ratios of 3:1 and 1:1 were used to encapsulate bioactive compounds as anthocyanins from berry and sweet potato (Akhavan et al. 2016; Mohd et al. 2015). Hygroscopicity is a critical factor in determining the shelf life of a product, as water percentage impacts lipid oxidation, powder rheology, and compound degradation (Vidović et al. 2014). The hygroscopicity values found in this study make it advisable to handle the encapsulates in environments with low relative humidity, and to pack them in materials impermeable to water vapor (Silva et al. 2012).

Some rehydration properties of the powdered products evaluated in this study are shown in Table 3, expressed in terms of water solubility index (WSI) and water absorption index (WAI). The results show that the MD-GA (1:3) polymer blend has a higher WSI, together with the MD:GA:TJ (3:1:14) treatment. This could be due to the type of MD used (28-32 dextrose equivalents [DE]), as MD products with lower molecular weight have shorter chains and more abundant hydrophilic groups, which allow for better solubility in water (Cai and Corke 2000; Lopera et al. 2009).

On the other hand, the MD-GA (3:1) polymer blend has a significantly lower WSI than MD-GA (1:3). This could be attributed to a higher concentration of GA in the blend. This effect is also observed in the en-

capsulates of the MD:GA:TJ (1:3:14) treatment, as a higher concentration of GA considerably reduced the solubility capacity in water. This may be due to the complexity of the GA structure and its amphiphilic nature, limiting the inclusion of water immediately (Lopera et al. 2009). In general, it is observed that the inclusion of MA-GA in tomato juice improves the solubility properties compared to tomato juice without cover material. On the other hand, the WSI for the encapsulated TJ values were like those reported in other studies (Kha et al. 2014; Silva et al. 2012). According to the literature, high WSI values are favorable and provide desirable properties to powders, as they allow for easier rehydration of the encapsulates when used as food ingredients (Fernandes et al. 2014; Vidović et al. 2014).

According to the literature, water solubility and water absorption are inversely related (Ahmed et al. 2010; Vidović et al. 2014). Therefore, significantly higher WAI values are observed in tomato juice and lower in the polymer mixture [MD-GA (3:1)], with an inverse behavior to the WSI results, confirming what has been reported in the literature. On the other hand, in the encapsulations of the MD:GA:TJ (3:1:14) and MD:GA:TJ (1:3: 14) treatments, no significant differences were found. The WAI results in the treatments were similar to those found by Paini et al. (2015), who reported values from 0.33 to 0.58 g/g DW in MD-encapsulated bioactive compounds. Ahmed et al. (2010) reported WAI values

ranging from 0.86 to 1.20 g/g DW in encapsulated purple potato-derived bioactive compounds. The encapsulated showed optimal hydration properties, as high WSI and low WAI values allow a powder to hydrate rapidly and completely, sinking in the liquid instead of floating, and dispersing-dissolving with no lump formation (Vidović et al. 2014).

The bulk density values of the powders are shown in Table 3. These results ranged from 0.24 to 0.61 g/cm<sup>3</sup>. It was found that the powders with the highest concentration of MD, such as MD:GA:TJ (3:1:14) and MD:GA (3:1), presented the highest values of apparent density. On the other hand, tomato juice presented the lowest values of this parameter, which means that tomato juice occupied a larger volume for each gram of sample. The apparent density of the tomato encapsulates in this study is congruent with those reported by Sarabandi et al. (2017), who reported the effect of MD and GA in spray-drying of cherry tomato; in his findings, they showed a reduction from 0.655 g/cm<sup>3</sup> (MD-GA 4:1) to 0.579 g/cm<sup>3</sup> (MD-GA 1:4). On the other hand, Edris et al. (2016) reported a bulk density of 0.5 g/cm<sup>3</sup> in microencapsulated of *N. sativa* oleoresin, while Bazarria and Kumar (2017) reported the bulk density values in samples of encapsulated beetroot juice in the range from 0.516 to 0.578 g/cm<sup>3</sup>. Those authors noted that large amounts of higher-density powders can be stored in smaller containers compared

**Table 3. Physicochemical properties of encapsulated tomato juice obtained by freeze-drying.**

|  | Mixture                   |                           |                           |                           |                           |
|--|---------------------------|---------------------------|---------------------------|---------------------------|---------------------------|
|  | MD:GA:TJ<br>(3:1:14)      | MD:GA:TJ<br>(1:3:14)      | MD:GA (3:1)               | MD:GA (1:3)               | TJ                        |
| <b>Moisture content (%)</b>                | 3.78 ± 0.22 <sup>b</sup>  | 3.85 ± 0.15 <sup>b</sup>  | 1.15 ± 0.10 <sup>c</sup>  | 2.08 ± 0.16 <sup>c</sup>  | 7.87 ± 0.41 <sup>a</sup>  |
| <b>Water activity (aw)</b>                 | 0.10 ± 0.01 <sup>c</sup>  | 0.14 ± 0.01 <sup>b</sup>  | 0.07 ± 0.01 <sup>d</sup>  | 0.07 ± 0.01 <sup>d</sup>  | 0.22 ± 0.01 <sup>a</sup>  |
| <b>Hygroscopicity (g/100 g DW)</b>         | 20.70 ± 0.11 <sup>a</sup> | 21.06 ± 0.09 <sup>a</sup> | 13.43 ± 0.35 <sup>c</sup> | 12.93 ± 0.18 <sup>c</sup> | 19.38 ± 0.21 <sup>b</sup> |
| <b>Water solubility index (g/100 g DW)</b> | 91.47 ± 1.12 <sup>b</sup> | 81.74 ± 2.05 <sup>c</sup> | 93.48 ± 2.03 <sup>a</sup> | 89.04 ± 0.86 <sup>b</sup> | 41.25 ± 1.35 <sup>d</sup> |
| <b>Water absorption index (g/g DW)</b>     | 0.59 ± 0.01 <sup>b</sup>  | 0.59 ± 0.05 <sup>b</sup>  | 0.39 ± 0.01 <sup>c</sup>  | 0.56 ± 0.09 <sup>b</sup>  | 2.74 ± 0.39 <sup>a</sup>  |
| <b>Bulk density (g/cm<sup>3</sup>)</b>     | 0.58 ± 0.03 <sup>b</sup>  | 0.55 ± 0.01 <sup>b</sup>  | 0.61 ± 0.01 <sup>a</sup>  | 0.43 ± 0.02 <sup>c</sup>  | 0.24 ± 0.01 <sup>d</sup>  |

Data are expressed as mean value ± standard deviation ( $n = 5$ ). Values tagged with the same letter in each row showed no significant differences according to the Tukey test with  $P \leq 0.05$ . MD: maltodextrin; GA: gum arabic; TJ: tomato juice.

to products with lower density values. In addition, a higher bulk density could indicate lower amounts of air occluded in inter-particle space, which could prevent lycopene and  $\beta$ -carotene oxidation in the formulations under study.

The color of powdered products is one of the most important parameters in the food industry, as it conveys sensorial attractiveness and quality to dried powders (Kha et al. 2014; Mohd et al. 2015). As shown in Table 4, color parameters showed significant differences in the samples of this study. The  $L^*$  values were significantly higher in the MD:GA polymer mixtures (3:1 and 1:3) due to the white tone characteristic of both encapsulating agents. On the other hand, the encapsulated formulations MD:GA:TJ (3:1:14) showed higher  $L^*$  values than MD:GA:TJ (1:3:14). This could be attributed to a higher content of MD in the wall material, as it shows a whiter color and, thus, provides a better luminosity to the product. Meanwhile, the  $^{\circ}h$  value that defines the color tone was significantly lower in freeze-dried TJ. The  $^{\circ}h$  values vary from  $0^{\circ}$  (pure red) to  $90^{\circ}$  (pure yellow). Thus, freeze-dried TJ showed the lowest  $^{\circ}h$  value, followed by the encapsulated MD:GA:TJ (3:1:14) and by MD:GA:TJ (1:3:14). This means that the latter exhibited a hue further from pure red than the former. A more intense reddish hue in the encapsulated MD:GA:TJ (3:1:14) with respect to MD:GA:TJ (1:3:14) confirms that it contained higher lycopene and  $\beta$ -carotene concentrations (Table 4). This is because lycopene and  $\beta$ -carotene are the pigments responsible for the red

color of tomatoes (López-Palestina et al. 2019; Raffo et al. 2006; Rosales et al. 2011). Similar behavior was reported by Quek et al. (2007) and Oberoi and Sogi (2015), who evaluated the effect of MD concentration on pigments from watermelon juice. These authors found similar color parameters; in addition, they indicated that this is due to the presence of lycopene,  $\beta$ -carotene, and total carotenoids. The powder mixtures in this study were attractive in color, showed a high luminosity provided by wall materials, and exhibited a characteristic soft color denoting the incorporation of bioactive compounds. In general, the concentration of the core has a significant influence on the color of the encapsulates (Sarabandi et al. 2017).

### Storage stability at different temperatures

The degradation kinetics of lycopene and  $\beta$ -carotene in the tomato juice encapsulates are shown in Figure 1. The lycopene content in the encapsulates decreased from 53.43-67.75% after 40 days of storage at  $25^{\circ}\text{C}$  and  $4^{\circ}\text{C}$ ; lycopene reduction varied with values of 85.51-88.48%. Regarding the reduction of  $\beta$ -carotene in the samples, there was a variability of 51.84-66.74% and 81.14-88.55% during storage at  $25^{\circ}\text{C}$  and  $4^{\circ}\text{C}$ , respectively. In general, the encapsulates of the MD:GA:TJ (3:1:14) treatment showed greater retention of carotenoids at different storage temperatures than MD:GA:TJ (1:3:14). Souza et al. (2018) indicated that maltodextrin is one of the most suitable encapsulating agents to protect lycopene during the

**Table 4. Color properties of encapsulated tomato juice obtained by freeze-drying.**

|               | Mixture                       |                               |                               |                               |                               |
|---------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|
|               | MD:GA:TJ<br>(3:1:14)          | MD:GA:TJ<br>(1:3:14)          | MD:GA (3:1)                   | MD:GA (1:3)                   | TJ                            |
| $L^*$         | 78.51 $\pm$ 0.31 <sup>b</sup> | 76.90 $\pm$ 0.42 <sup>c</sup> | 87.37 $\pm$ 0.34 <sup>a</sup> | 86.77 $\pm$ 0.09 <sup>a</sup> | 44.47 $\pm$ 0.47 <sup>d</sup> |
| $a^*$         | 12.72 $\pm$ 0.31 <sup>b</sup> | 12.88 $\pm$ 0.47 <sup>b</sup> | 0.70 $\pm$ 0.01 <sup>c</sup>  | 0.96 $\pm$ 0.07 <sup>c</sup>  | 32.74 $\pm$ 0.10 <sup>a</sup> |
| $b^*$         | 11.56 $\pm$ 0.30 <sup>b</sup> | 12.31 $\pm$ 0.48 <sup>b</sup> | 7.97 $\pm$ 0.16 <sup>c</sup>  | 8.65 $\pm$ 0.12 <sup>c</sup>  | 26.79 $\pm$ 0.34 <sup>a</sup> |
| <i>Chroma</i> | 17.18 $\pm$ 0.42 <sup>b</sup> | 17.82 $\pm$ 0.67 <sup>b</sup> | 8.00 $\pm$ 0.16 <sup>c</sup>  | 8.71 $\pm$ 0.12 <sup>c</sup>  | 42.31 $\pm$ 0.18 <sup>a</sup> |
| $^{\circ}h$   | 42.26 $\pm$ 0.31 <sup>c</sup> | 43.71 $\pm$ 0.18 <sup>d</sup> | 84.95 $\pm$ 0.09 <sup>a</sup> | 83.65 $\pm$ 0.43 <sup>b</sup> | 39.29 $\pm$ 0.41 <sup>e</sup> |

Data are expressed as mean value  $\pm$  standard deviation ( $n = 5$ ). Values tagged with the same letter in each row showed no significant differences according to the Tukey test with  $P \leq 0.05$ . MD: maltodextrin; GA: gum arabic; TJ: tomato juice.

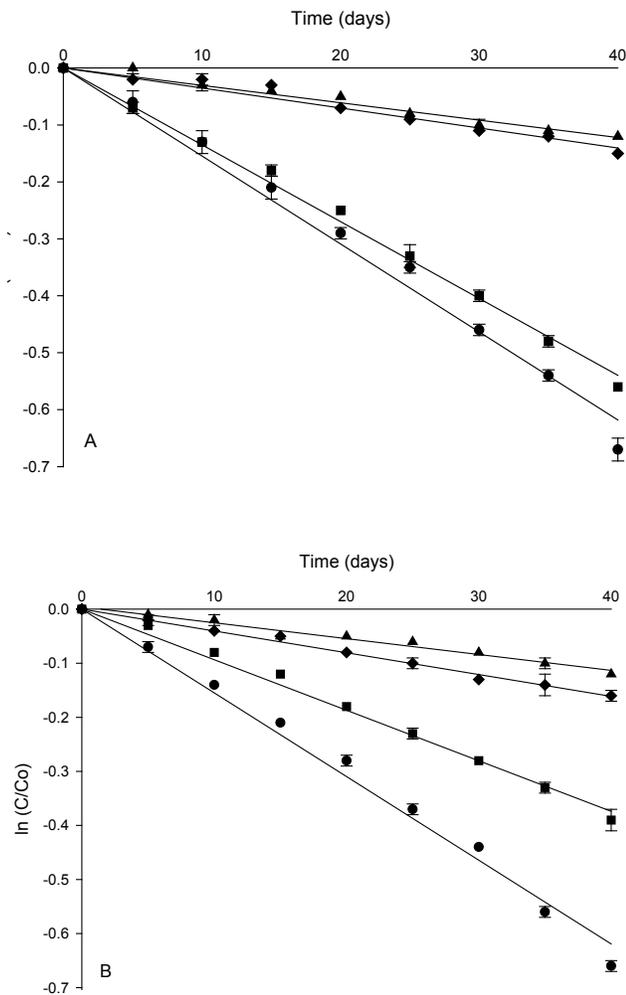


Figure 1. Degradation kinetics of lycopene (A) and  $\beta$ -carotene (B) in encapsulated tomato juice stored at 4°C (▲MD:GA:TJ (3:1:14), ◆MD:GA:TJ (1:3:14)) and 25°C (●MD:GA:TJ (3:1:14), ■MD:GA:TJ (1:3:14)). Data are expressed as mean value  $\pm$  standard deviation (n = 5).

drying and storage process of concentrated tomato juice microencapsulates obtained by atomization. This may be because MD forms wall systems with higher density and lower permeability to oxygen, providing carotenoids with better storage stability (Cai and Corke 2000; Ferrari et al. 2013). On the other hand, the

degradation rate of lycopene in the encapsulates was greater than the degradation of  $\beta$ -carotene, as shown in the degradation constant of these compounds (Table 5). The literature mentions that lycopene degrades faster than  $\beta$ -carotene (Meléndez-Martínez et al. 2004). On the other hand, the degradation rate constants (k) found in this research agree with those reported by Matioli and Rodriguez-Amaya (2002), who reported degradation rate constants of isolated and encapsulated lycopene in MD and GA of 0.0106-0.1223 with a higher degradation rate of the compound in encapsulates obtained by spray-drying. Souza et al. (2018) reported lycopene decreases of 7 to 33% during 28 days of storage in concentrated tomato microcapsules and with lycopene degradation constant values of 0.0362-0.102. It has been reported that the high degradation rates of these carotenoids may be due to oxygen contact with the encapsulated material through the pores in the wall material (Matioli and Rodriguez-Amaya 2002; Shu et al. 2006).

## CONCLUSIONS

The tomato juice encapsulates obtained by freeze-drying showed good hydration and solubilization properties in water, which allowed for rapid and complete hydration of the encapsulates. The apparent density characteristics indicate greater protection of lycopene and  $\beta$ -carotene, as less air is entrapped in the walls of the encapsulates. Good colorimetric properties were observed in the powders due to the presence of the tomato juice pigments. This study showed that the encapsulates of the MD:GA:TJ (3:1:14) treatment presented a higher encapsulation efficiency and, consequently, a concentration of lycopene and  $\beta$ -carotene, greater antioxidant activity, and greater stability of these carotenoids, mainly of  $\beta$ -carotene during storage at 4°C. The physicochemical

Table 5. Degradation rate constant (k) of lycopene and  $\beta$ -carotene in encapsulated tomato juice.

|                                    | MD:GA:TJ (3:1:14)    |                      | MD:GA:TJ (1:3:14)    |                      |
|------------------------------------|----------------------|----------------------|----------------------|----------------------|
|                                    | 25°C                 | 4°C                  | 25°C                 | 4°C                  |
| <b>Lycopene</b>                    | 0.01397 <sup>b</sup> | 0.00314 <sup>d</sup> | 0.01640 <sup>a</sup> | 0.00379 <sup>c</sup> |
| <b><math>\beta</math>-carotene</b> | 0.00994 <sup>b</sup> | 0.00296 <sup>d</sup> | 0.01621 <sup>a</sup> | 0.00401 <sup>c</sup> |

Values tagged with the same letter in each row showed no significant differences according to the Tukey test with  $P \leq 0.05$ . MD: maltodextrin; GA: gum arabic; TJ: tomato juice.

and antioxidant properties identified in this research suggest the possible incorporation of tomato juice encapsulates in food matrices.

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#### **CONFLICTS OF INTEREST**

The authors declare there are no conflicts of interest in the present investigation.

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