Food Chemistry Advances 1 (2022) 100046

Contents lists available at ScienceDirect

Food Chemistry Advances

journal homepage: www.elsevier.com/locate/focha

Microencapsulation of acerola (*Malpighia emarginata* DC) AND ciriguela (*Spondias purpurea* L) mixed juice with different wall materials

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ARTICLE INFO

Keywords:
Spray drying
Bioactive compounds
Stability
Xanthan gum
Gum arabic
maltodextrin

ABSTRACT

The effect of five different mixtures containing xanthan gum (XG), maltodextrin (MD) and gum arabic (GA) on microencapsulation by spray drying of mixed acerola and ciriguela juice was investigated in order to preserve its contents of ascorbic acid (AA), total phenolic compounds (PC) and total carotenoids (TC), as well as its antioxidant activity (AO) under different temperatures. All microcapsules produced with the different mixtures showed effective AA retention. Microcapsules produced using the mixtures with the highest and the lowest XG concentrations showed the greatest TC and PC retentions, respectively. As the mixture with MD and the lowest XG concentration ensured the highest AO retention, these microcapsules were stored for 30 days at 5°C, which ensured satisfactory preservation of AA, PC, TC and AO. Examination of their morphology by scanning electron microscopy showed the absence of pores or cracks, which allowed us to infer low oxygen permeability and high oxidative stability.

1. Introduction

Recent trends in the food industry show that functional foods are becoming more popular around the world and are increasingly being incorporated into the daily diet. Food companies are producing new products enriched with bioactive compounds, with the aim of promoting better health of consumers. This growth is driven not only by innovation in the food industry, but also by changing lifestyles and increasing consumer awareness about his health and nutritional benefits of foods containing bioactive compounds. Given that health is becoming an increasingly important personal and social value, it is not surprising that consumers have started to pay more attention to the benefits of good nutrition (Barauskaite et al., 2018).

Exotic tropical fruits are rich sources of bioactive compounds, such as ascorbic acid (AA), total carotenoids (TC) and phenolic compounds (PC), having the potential to be used in the production of foods with healthy and functional appeal. Acerola (*Malpighia emarginata* DC) is a fruit known for its high content of AA, which can reach 1678 mg/100 g (Belwal et al., 2018), of PC including benzoic acid derivatives, phenylpropanoids, flavonoids and anthocyanins, and of TC. They can be consumed either *in natura* or processed in the form of juices, ice cream, jellies, syrups, liqueurs, sweets in syrup and vitamin C capsules (Moura et al., 2018; Nascimento et al., 2018; Ribeiro et al., 2019). Ciriguela (*Spondias purpurea* L), originating in Central and South America and quite common in northeastern Brazil, is a red or orange drupe that, when ripe, has a pulp with pleasant aroma and flavor. It is rich in TC and micronutrients, such as calcium, phosphorus, iron, provitamin A, B complex vitamins and AA, and contains in its pulp, husk and seeds compounds such as quercetin, hydroxycinnamic acids, ellagic acid, chlorogenic acid, protocatechuic acid, flavonoids, gallic acid, vanillic acid, *p*-coumaric acid, and ferulic acid (Silva, Costa, Branco, & Branco, 2016). The purpose of preparing a mixed acerola and ciriguela juice is to obtain a product with good economic potential, as these fruits are widely produced, consumed and marketed in the rapidly developing northeast region of Brazil, and have high levels of AA, PC and TC.

As AA, PC and TC are very susceptible to degradation, especially when exposed to light, moisture, heat and oxygen, one of the techniques most used today to protect them is microencapsulation by spray

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https://doi.org/10.1016/j.focha.2022.100046
Received 20 December 2021; Received in revised form 1 May 2022; Accepted 13 May 2022

drying. Microencapsulation consists of coating solids, liquids or gaseous materials with a continuous film to produce micrometer-sized capsules (Ozkan, Franco, Marco, Xiao, & Capanoglu, 2019). Compounds used to coat these microcapsules are also known as wall materials. The selection of the appropriate wall material is an important factor for microencapsulation and protection of bioactive compounds, because it must provide adequate mechanical resistance, be chemically and thermally compatible with the product to which it will be applied, and allow the controlled release of bioactive compounds (Labuschagne, 2018; Rezende, Nogueira, & Narain, 2018).

Maltodextrin (MD) is a wall material widely used in encapsulation by spray drying because it has a low cost and good ability to form amorphous solids, improves the aqueous solubility of the encapsulated material, and has low viscosity at high concentrations. Like MD, gum arabic (GA) is also widely used as a wall material because it has favorable attributes such as film-forming capacity, high water solubility, low viscosity, good retention of volatile components and emulsifying properties (Labuschagne, 2018; Rezende, Nogueira, & Narain, 2018). There are few studies on the use of xanthan gum (XG) as a wall material for microencapsulation by spray drying, although it is recognized as an efficient thickening agent capable of providing, when used in combination with other gums, better retention of active compounds in the polymer matrix (Labuschagne, 2018).

The combination of different wall materials is of special commercial interest, as it allows for new functionalities, on the one hand, and a reduction in the level of inputs, on the other. Therefore, the objective of the present work was to evaluate the combination of XG, MD and GA in the preservation of AA, PC and TC present in the mixed acerola and ciriguela juice during its microencapsulation by spray drying, as well as to study the stability of these compounds during storage at different temperatures, in order to offer a good cost-benefit relation of the new microencapsulated product.

2. Materials and methods

2.1. Sample preparation

Fruits of ciriguela (*Spondias purpurea* L) and acerola (*Malpighia emarginata* DC), cultivated in small rural farms of the Agreste and Mata zone of the state of Pernambuco, Brazil, were purchased at the Supply and Logistics Center of Pernambuco and handled at the Physicochemical Analysis and Food Processing Laboratory located in the Department of Consumer Sciences of the Federal Rural University of Pernambuco. After discarding the unsuitable, rotten or injured fruits as well as pieces of leaves and stems, those selected were washed in water to remove any dirt and sanitized by immersion for 15 min in a sodium hypochlorite solution at a concentration of 200 ppm. A pulper (model NB10, Bonina Compacta, Itabauna, BA, Brazil) was used for pulping. Pulps were packaged in low-density polyethylene bags, frozen and stored at -20 °C until use. For the experiments, pulps were mixed in the proportion of 60% acerola and 40% ciriguela (g/g), according to the methodology described by Ribeiro et al. (2019).

2.2. Physicochemical characterization of in natura pulps

Pulps were characterized for moisture, soluble solids, pH, titratable acidity, proteins, lipids and ash according to the methodologies described by the Association of Official Analytical Chemists (AOAC, 2016), while the total sugar content was calculated by difference and expressed in g/100 g.

2.3. Color of in natura pulps

The color of *in natura* pulps was measured with a manual Color Reader colorimeter (CR400/410, Minolta, Osaka, Japan) and evaluated according to the CIELab system, where L^* is the luminosity (0 = black and 100 = white), a^* the intensity of the green-red component (green in negative and red in positive directions) and b^* the intensity of the blue-yellow component (blue in negative and yellow in positive directions) (Choudhury, 2014). After placing the pulps on Petri plates until covering the bottom, five readings were performed on the surface of pulps using a xenon lamp (Standard Illuminant C), a 10° observation angle and a measurement area with 8-mm diameter.

2.4. Ascorbic acid content of in natura pulps

The ascorbic acid content (AA) was determined by a titrimetric method using 2,6-dichlorophenol-indophenol (AOAC, 2016). To do so, a standard solution of 1% ascorbic acid was prepared. After weighing 5 g of each sample in Erlenmeyer, 50 mL of distilled water were added. Distilled water was used as a blank and a solution of 0.01% 2,6-dichlorophenol-indophenol as a titrant. The content of ascorbic acid, expressed in mg/100 g, was calculated through the equation:

$$\mathrm{AA} = \frac{(VA - VB)}{VP} * 100$$

where VA, VB and VP are the volumes of titrant solution used in the titrations of sample, blank and standard, respectively.

2.5. Total carotenoid content of in natura pulps

The total carotenoid content (TC) was determined according to the methodology described by Rodriguez-Amaya (1999). Carotenoids were extracted with ethyl ether by macerating 2 g of sample until it became colorless. After washing the extract with 2 L of distilled water, carotenoids were quantified by UV-VIS spectrophotometry (model UV-1650PC, Shimadzu, Kyoto, Japan) at 450 nm wavelength, using the absorption coefficient of 2500. The results were expressed in μg of β-carotene equivalent per g of sample(wet weight).

2.6. Total phenolic compounds content of in natura pulps

Hydroalcolic extracts of the pulps were prepared using 60 mL of distilled water, 40 mL of 95% ethyl alcohol and 20 g of pulp. After homogenization in Erlenmeyers using a magnetic stirrer for 1 h without heating, the mixtures were centrifuged at 2000 rpm for 10 min, and the supernatants were filtered in qualitative filter paper with basis weight of 80 g/m^2 and stored in amber glass bottles under refrigeration at approximately 5°C, for further analysis. The content of total phenolic compounds (PC) was determined by the same spectrophotometer described above at a wavelength of 725 nm, after addition of Folin-Ciocalteau reagent and water as solvent, according to the methodology described by Wettasinghe and Shahidi (1999). For this purpose, the experimental absorbance values of the samples were interpolated into a calibration curve constructed with gallic acid, and the results expressed in mg of gallic acid equivalent (GAE) per 100 g of sample.

2.7. Obtaining microcapsules of mixed acerola and ciriguela juice

Three different wall materials were used for the microencapsulation, i.e., maltodextrin 10DE provided by Ingredion Incorporated (São Paulo, SP, Brazil), gum arabic manufactured by Dinamica Chemical Contemporanea Ltda (São Paulo, SP, Brazil) and xanthan gum provided by Federal University of Pelotas (Pelotas, RS, Brazil). To perform the atomization by spray drying, a suspension containing 30% of total solids was prepared, being 21% wall material and 9% mixed acerola and ciriguela pulp. Due to the high viscosity of xanthan gum suspensions, preliminary tests were carried out to identify concentrations that would allow its use, in particular regarding its transport to the atomizing nozzle and the inlet temperature of the spray dryer, whose results indicated the following mixtures as the best ones: 99.7:0.3 (g/g) maltodextrin:xanthan gum, 99.9:0.1 (g/g) maltodextrin:xanthan gum, 99.7:0.3

(g/g) gum arabic:xanthan gum, 99.9:0.1 (g/g) gum arabic:xanthan gum, and 49.9:49.9:0.2 (g/g/g) maltodextrin:gum arabic:xanthan gum, referred to as MX3, MX1, AX3, AX1 and MAX, respectively. Microencapsulation by spray drying was performed in a mini-spray dryer (model LM-MSD 1.0, LabMaq do Brasil Ltda, Ribeirão Preto, SP, Brazil) under the following conditions: liquid flow rate of 0.6 L/h, atomizing nozzle with a diameter of 1.2 mm, air flow rate of 30 m^3/h, air pressure of 0.6 bar, and drying air inlet temperature of 140°C (Ribeiro et al., 2019). After drying, the powders were placed in hermetically sealed glass jars and stored at a temperature of -20°C until analyses.

2.8. Moisture content of microcapsules

The moisture content of microcapsules was determined gravimetrically (AOAC, 2016) using a moisture meter with an infrared heat source (model ID50, Marte Científica, Santa Rita do Sapucaí, MG, Brazil), and the results were expressed as a percentage (%).

2.9. Water activity of microcapsules

The water activity of microcapsules was determined using a water activity analyzer (AquaLab 4TE series, Decagon Devices, Pullman, WA, USA) (AOAC, 2016).

2.10. Contents of ascorbic acid, total carotenoids and total phenolic compounds

The bioactive compounds contained in microcapsules, namely AA, TC and PC, were quantified according to the methodologies previously described in Sections 2.4, 2.5 and 2.6 for their quantification in the *in natura* pulp. While AA and TC were determined directly using the microcapsules, to determine PC and antioxidant activity (AO) an extract was prepared dispersing 10 mg of the microencapsulated product in 10 mL of a 50:8:42 (v/v/v) ethanol:acetic acid:distilled water solution. The mixture was vortexed for 1 min and filtered through qualitative filter paper with basis weight of 80 g/m^2 (Saénz, Tapia, Chávez, & Robert, 2009).

2.11. Determination of the antioxidant activity of microcapsules

2.11.1. 2,2-Diphenyl-1-picrylhydrazyl (DPPH•) free radical scavenging method

The capacity of microcapsules to scavenge the 2,2-diphenyl-1-picrylhydrazyl (DPPH•) free radical was determined according to the method described by Brand-Williams, Cuvelier, & Berset (1995), modified by Sánchez-Moreno, Larrauri, & Saura-Calixto (1998). An aliquot (0.1 mL) of the extract prepared as per the previous section was added to a solution of DPPH• in methanol (0.1 M), and the absorbance measured at 515 nm with the aforementioned spectrophotometer until reaching the plateau. The ability to scavenge the DPPH• radical, expressed as a percentage, was calculated in relation to the control (without antioxidant), according to the equation:

$$\%\ \text{scavenging} = \frac{Abs_c - Abs_a}{Abs_c} \times 100$$

where Abs_c and Abs_a are the control and sample absorbances, respectively.

2.11.2. Method of the ferric reducing antioxidant power (FRAP)

The antioxidant activity of microcapsules was also evaluated by the ferric reducing antioxidant power (FRAP) assay according to the methodology reported by Rufino et al. (2006). For this purpose, a standard curve of absorbance at 593 nm versus the ferrous sulfate concentration (500–2000 μM) was constructed to determine the absorbance of 1000 μM ferrous sulfate standard. Three dilutions of the extract (1:30; 1:40; 1:50 v/v) were performed in triplicate. Aliquots (90 μL) of each extract dilution were transferred to test tubes containing 270 μL of distilled water and 2.7 mL of FRAP reagent, previously prepared by mixing 25 mL of 0.3 M sodium acetate/acetic acid buffer (pH 3.6), 2.5 mL of a 10 mM 2,4,6-tri(2-pyridyl)-s-triazine (TPTZ) solution and 2.5 mL of a 20 mM ferric chloride aqueous solution. The mixtures were homogenized by vortexing and kept in a water bath at 37°C for 30 min. Then, reading was performed using the FRAP reagent as a blank to calibrate the spectrophotometer. From the absorbance values obtained from the different dilutions of the extracts, an absorbance curve was constructed as a function of concentration (mg/L), to then obtain the x term corresponding to the sample concentration equivalent to 1000 μM ferrous sulfate.

2.12. Stability of microcapsules

The microcapsules considered the most promising in terms of contents of AA, TC and PC as well as of AO were submitted to a 30-day stability study, in triplicate, at temperatures of 5 and 30°C. Microcapsules were stored in glass jars (40 mL) protected from light, and samples were withdrawn at the beginning and after 7, 15 and 30 days to quantify AA, TC, PC and AO.

2.13. Microcapsule morphology

The morphology of microcapsules was examined by scanning electron microscopy (SEM). Microparticles were adhered to metal stubs with a conventional conductive double-sided adhesive tape. Samples were then metallized with a gold alloy in an EM SCD500 metallizer (Leica, Wetzlar, Germany) and examined with a Quanta 200F scanning electron microscope (FEI, Hillsboro, OR, USA).

2.14. Statistical analysis

The determinations and analyses on the *in natura* pulps and the microcapsules produced with the different mixtures of wall materials were carried out in triplicate. Data were statistically treated by the analysis of variance (ANOVA) and submitted to Tukey's test, using the statistica 7 software (StatSoft, Tulsa, OK, USA). Differences were considered significant at a 5% probability of the null being correct ($p<0.05$).

3. Results and discussion

3.1. Characterization of acerola and ciriguela pulps and of mixed pulp

The results of the physicochemical analyses of acerola and ciriguela pulp as well as the mixed pulp are listed in Table 1.

The values of titratable acidity, soluble solids and pH of acerola were close to those found by Ribeiro et al. (2019), being in both cases in the ranges of 12.0-15.0%, 5.0-5.5 °Brix and pH < 4.0, respectively. In addition, Gualberto et al. (2021) reported for the same pulp contents of moisture (92.10%), proteins (1.68%), lipids (0.39%), ash (0.85%) and carbohydrates (4.98%) close to those obtained in the present study (Table 1).

Maldonado-Astudillo et al. (2014), who studied the physiology of 15 species of *Spondias* sp., found for ciriguela values of pH between 2.65 and 6.0, soluble solids between 8.2 and 17.7 °Brix, titratable acidity from 2.0 to 7.0% and total carbohydrates in the range 5.0–20.0%, while Lira Júnior et al. (2010) reported for the same fruit 77.60% moisture, 18.90% total carbohydrates, 0.36% lipids and 1.20% proteins. All these values are not so far from those obtained in the present study (Table 1).

Although there are few references to exhaustively compare these results with the literature as the mixed acerola and ciriguela pulp is a recent product, its protein, lipid, ash and carbohydrate contents are close to those reported by Ribeiro et al. (2019). With regard to soluble solids, there was a statistically significant difference between the mixed pulp and the individual pulps because the high content of these solids in ciriguela (18.2 ± 0.1 °Brix) led to their enrichment in the mixed

Table 1
Physicochemical characterization and contents of ascorbic acid (AA), total carotenoids (TC) and phenolic compounds (PC) of acerola pulp, ciriguela pulp, and 60:40 (g/g) acerola and ciriguela mixed pulp.

Parameter	Acerola	Ciriguela	Mixed pulp
Moisture (g/100 g)	92.7 ± 0.5[a]	78.8 ± 0.3[b]	87.6 ± 0.2[a]
Sugars (g/100 g)	4.30 ± 0.18[c]	18.46 ± 0.31[a]	9.92 ± 0.08[b]
Lipids (%)	0.32 ± 0.02[a]	0.43 ± 0.02[b]	0.33 ± 0.08[a]
Proteins (%)	1.85 ±0.20[a]	1.57 ±0.12[a]	1.23 ±0.07[a]
Ash (%)	0.82 ±0.02[b]	0.77 ±0.01[a]	0.80 ±0.01[b]
Soluble solids (°Brix)	5.2 ± 0.1[c]	18.2 ± 0.1[a]	10.0 ± 0.1[b]
Titratable acidity (%)	13.2 ± 0.3[a]	5.6 ± 0.2[b]	4.7 ± 0.2[b]
pH	3.13±0.12[a]	3.18±0.25[a]	3.23±0.18[a]
Color (L*)	45.2 ±0.6[a]	60.3 ± 0.1[b]	40.7 ± 0.7[a]
Color (a*)	28.0 ±0.1[a]	14.0 ±0.2[b]	26.8 ±0.5[a]
Color (b*)	13.0 ±0.3[a]	25.8 ±0.2[b]	13.5 ±0.7[a]
AA (mg/100 g)	1578 ± 32[a]	20.5 ± 0.8[c]	914 ± 26[b]
TC (μg/g)	10.1 ± 0.1[c]	18.1 ± 0.1[a]	13.5 ± 0.1[b]
PC (mgGAE/100 g)	4302 ± 11[a]	269 ± 13[c]	2817 ± 27[b]

Values are expressed as mean ± standard deviation. Means followed by the same letter do not differ significantly by the Tukey's test at 5% probability ($p<0.05$). L* = luminosity; a* = intensity of the green-red component; b* = intensity of the blue-yellow component. GAE - Gallic acid equivalent.

pulp when compared to acerola. Surprisingly, the titratable acidity of the mixed pulp was lower than those of both ciriguela and acerola, although not statistically significant in the second case. This reduction in titratable acidity can be attributed to the presence of amines such as tyramine, spermidine and putrescine in the ciriguela pulp, which, during mixing, may have neutralized a portion of the acids present in acerola (Dutra et al., 2017). The colorimetric parameters, namely the luminosity (L*) and the green-red (a*) and blue-yellow (b*) components, indicate similarity between the colors of acerola and mixed pulp, considering that 60% of the composition of mixed pulp comes from acerola.

3.2. Contents of ascorbic acid, total carotenoids and total phenolic compounds in acerola and ciriguela pulps and in mixed pulp

The ascorbic acid (AA) content in acerola pulp (1578 mg/100 g) (Table 1) was within the range (100 - 1700 mg/100 g) reported by Cruz et al. (2019), while that in the pulp of ciriguela was much smaller (20.5 mg/100 g) and within the range (7.36-88.1 mg/100 g) reported by Maldonado-Astudillo et al. (2014).

Another important class of bioactive compounds is that of total carotenoids (TC), whose content in both acerola (10.1 μg/g) and ciriguela (18.1 μg/g) was within the ranges reported in the literature, i.e., 3.71–18.8 μg/g (Belwal et al., 2018) and 13.4–22.6 μg/g (Da Silva, Figueiredo, & De Lima, 2016), respectively. On the other hand, acerola pulp exhibited a content of phenolic compounds (PC) (4302 mg GAE/100 g) well above that found by other authors (1000–2159 mg GAE/100 g) (Alvarez-Suarez et al., 2017; Rezende et al., 2018). The opposite occurred with the ciriguela pulp, whose PC content (269 mg GAE/100 g) was lower than the range (351–862 mg GAE/100 g) reported for 11 different genotypes of ciriguela (Silva, Moreira, Melo, & Lima, 2012). This variability suggests a strong dependence of the content of both bioactive compounds on the strain type. As expected, the mixed acerola and ciriguela pulp had contents of all these components between those of the individual pulps and closer to those of the acerola pulp, due to its higher percentage.

3.3. Characterization of powders obtained by mixed pulp spray drying

Mixed pulp was spry dried using different combinations of maltodextrin (MD), xanthan gum (XG) and gum arabic (GA), with average encapsulations efficiencies for ascorbic acid, total carotenoids and total phenolic compounds of 60, 16 and 53%, respectively.

3.3.1. Moisture content and water activity

Water activity is a thermodynamic measure of water energy in a product that is directly related to the susceptibility of food to microbial action (Damodaran, 2017). Table 2 shows the values of water activity and moisture content of microcapsules obtained by spray drying the 60:40 (g/g) acerola and ciriguela mixed juice. Despite the statistically significant differences among microcapsules prepared with different combinations of the three selected wall materials, namely MD, XG and GA, they all showed low moisture content (2.42-4.05 g/100 g) and water activity (0.167-0.257), being considered microbiologically stable those with water activity lower than 0.6 (Negrão-Murakami et al., 2017).

3.3.2. Ascorbic acid content

The five combinations of wall materials used to microencapsulate the mixed acerola and ciriguela juice ensured values of the AA content that varied between a minimum of 3812 mg/100 g for the binary 99.7:0.3 (g/g) gum arabic and xanthan gum mixture (AX3) and a maximum of 4850 mg/100 g for the ternary 49.9:49.9:0.2 (g/g/g) maltodextrin, gum arabic and xanthan gum mixture (MAX) (Table 2). It can be seen that the use of a higher concentration of XG (0.3%) in the AX3 and 99.7:0.3 (g/g) maltodextrin and xanthan gum (MX3) binary mixtures compared to that (0.1%) in the 99.9:0.1 (g/g) gum arabic and xanthan gum (AX1) and 99.9:0.1 (g/g) maltodextrin and xanthan gum (MX1) mixtures reduced the AA content. As the main characteristic of XG is to form highly viscous solutions even at low concentrations, its addition at the highest ratio may have led to an excessive viscosity increase that may have impaired the formation of droplets in the atomizer, exposing them for a longer time to the high temperatures of the drying chamber, thus affecting the thermosensitive compounds (Xu et al., 2020). However, the highest AA content in microcapsules prepared with the ternary mixture, in which the XG percentage was intermediate (0.2%), suggests a positive aggregating effect of increasing the viscosity up to a maximum value.

The values obtained using any of the five different wall material mixtures were satisfactory, taking into account that the recommended AA daily intake (RDI), according to FAO/WHO (2002), is 45 mg/d for an adult. With this in mind, the consumption of 12.4 g of microcapsules of acerola-ciriguela mixed juice prepared with any of the five wall material mixtures would provide 100% of the RDI for an adult; therefore, they could be considered nutritious. Because the MAX blend, containing both XG and GA, would be about 9-fold more expensive than MX1 based on average prices of maltodextrin and gum arabic, the latter would be preferable at an industrial level, as it retained enough AA for the daily needs of an adult.

Some other researchers determined the AA content in microcapsules prepared by spray drying acerola pulp. Rezende et al. (2018) reported AA contents (220.76 - 551.50 mg/100 g) in acerola pulp microcapsules one order of magnitude lower than those obtained in the present study (Tabela 2). The fact that these authors used GA and MD as wall materials instead of XG seems to confirm the positive effect of XG at very low concentrations. However, the values detected in the present study are much lower than those reported by Leyva-López et al. (2019), who obtained 16500 mg/100 g of AA in microcapsules prepared using GA as the wall material.

3.3.3. Content of total carotenoids

Table 2 shows that the content of total carotenoids (TC) ranged between 0.13 μg/g and 3.14 μg/g, i.e., values below the desired level to enable its production at an industrial level. It is known that the efficiency of TC microencapsulation by spray drying and their stability depend on many factors, one of which is the type of wall material used for encapsulation. The most efficient wall materials for encapsulating carotenoids are MD and GA (Labuschagne, 2018), which were used in MX and AX mixtures, respectively. However, microcapsules of mixed acerola-ciriguela juice prepared using the mixtures with the highest XG percentage (0.3%), i.e., MX3 and AX3, exhibited the highest contents of

Table 2
Water activity, moisture content, and ascorbic acid (AA), total carotenoids (TC) and phenolic compounds (PC) contents of microcapsules obtained by spray drying the 60:40 (g/g) acerola and ciriguela mixed juice using different combinations of wall materials.

Wall material	Water activity	Moisture (g/100 g)	AA (mg/100 g)	TC (μg/g)	PC (mg GAE/100 g)
MX3	0.188 ± 0.013^{c}	2.42 ± 0.25^{c}	4011 ± 41^{bc}	3.14 ± 0.26^{a}	1931 ± 64^{b}
MX1	0.167 ± 0.021^{c}	2.47 ± 0.22^{c}	4211 ± 35^{b}	1.43 ± 0.04^{b}	2078 ± 20^{a}
AX3	0.257 ± 0.016^{a}	4.05 ± 0.18^{a}	3812 ± 37^{c}	2.74 ± 0.30^{a}	1120 ± 7^{d}
AX1	0.209 ± 0.005^{b}	3.24 ± 0.12^{b}	4311 ± 26^{b}	0.86 ± 0.18^{c}	2120 ± 27^{a}
MAX	0.211 ± 0.100^{b}	3.07 ± 0.16^{b}	4850 ± 55^{a}	0.13 ± 0.20^{d}	1896 ± 5^{c}

MX3: 99.7:0.3 (g/g) maltodextrin and xanthan gum; MX1: 99.9:0.1 (g/g) maltodextrin and xanthan gum; AX3: 99.7:0.3 (g/g) gum arabic and xanthan gum; AX1: 99.9:0.1 (g/g) gum arabic and xanthan gum; MAX: 49.9:49.9:0.2 (g/g/g) maltodextrin, gum arabic and xanthan gum. Values are expressed as mean ± standard deviation. Means followed by the same letter do not differ significantly by Tukey's test at 5% probability of the null being correct.

these bioactive compounds (Table 2), not only confirming the effectiveness of XG as a wall material, but also suggesting less sensitivity of TC than AA to the viscosity increase caused by it. As carotenoid molecules are hydrophobic, XG may have acted as a stabilizer and its addition to the system allowed for the homogeneity of the mixture, thereby preventing the separation of the different constituents used (Taheri & Jafari, 2019). Therefore, its combination with MD and/or GA resulted in a better preservation of TC in microcapsules of mixed acerola-ciriguela juice.

3.3.4. Content of total phenolic compounds

Differently from both the AA and TC contents, the microcapsules of mixed acerola-ciriguela juice prepared using as a wall material the mixtures with the lowest XG percentage (AX1 and MX1) exhibited the highest contents of phenolic compounds (PC) (Table 2). It is likely that the low viscosity of these mixtures enabled the formation of amorphous solids and thus gave the microcapsules greater structural support (Labuschagne, 2018). Furthermore, it was reported that during the microencapsulation by spray drying the increased viscosity, caused in this case by an increase in XG concentration, induces the formation of larger droplets in the atomizer, which can impair drying and lead to formation of microcapsules with higher moisture content (Xu et al., 2020). Since the microcapsules obtained by spray drying have semi-crystalline structure in the polymer matrix, extra moisture allows greater mobility of surface water molecules to internal channels, an effect that may lead to structural changes which may expose bioactive compounds to light and oxygen and result in significant losses of such compounds (Hoyos-Leyva, Chavez-Salazar, Castellanos-Galeano, Bello-Perez, & Alvarez-Ramirez, 2018).

These results suggest that the AX1 and MX1 blends would be the best options for spray drying the mixed acerola-ciriguela juice in terms of PC losses; however, it would be preferable to use the MX1 mixture due to the lower cost of MD compared to GA. Rocha et al. (2019) found that GA was the most efficient wall material to preserve PC during microencapsulation of jabuticaba, jussara and blueberry extracts. Finally, unlike this work, Tolun, Altintas, & Artik (2016) obtained better results in terms of preservation of grape polyphenols during spray drying by combining MD and GA.

3.3.5. Antioxidant activity of microcapsules

The 2,2-diphenyl-1-picrylhydrazyl (DPPH•) free radical scavenging method was used to quantify the antioxidant activity (AO) of microcapsules of 60:40 (g/g) acerola and ciriguela mixed juice produced by spray drying with the different wall materials. The microcapsules that showed the highest AO (60.54%) were those prepared using the MX1 mixture (Table 3), although most of the others also showed moderate AO (50-54%) (Nascimento et al., 2018).

On the other hand, the microcapsules prepared with the AX3 mixture showed lower DPPH• radical scavenging activity (38.9%), as expected from its lower PC content (Table 2), hence confirming previous observations (Silva et al., 2016). However, not only the PC content must be taken into account, but also the type and chemical structure of these compounds, since they directly influence the availability of the hydrogen atom to react with the free radical (Xu et al., 2020). A confirmation of this is that the microcapsules obtained with the AX1 and MX1 mixtures showed different AO values by this method despite statistically coincident PC contents (Table 2).

Although the DPPH• radical scavenging test is a useful tool for AO assessment, it has little biological validity, that is, the results obtained by this method do not reflect well the antioxidant capacity of samples in biological systems; therefore, the ferric reducing antioxidant power (FRAP) method, which assesses the ability of a sample to reduce the Fe^{3+} ion to Fe^{2+} ion, is preferred when the objective is to study the effects of antioxidants on the body (Abdel-Aal, Hucl, & Rabalski, 2018).

Unlike what happened with the DPPH• radical scavenging assay, the microcapsules obtained with the MAX ternary mixture had the highest AO by the FRAP method (Table 3), due to the high AA content in this sample (Sethi et al., 2020), followed by those prepared with MX1. However, considering the economic and operational feasibility, it would be preferable to use the latter mixture to spray dry the 60:40 (g/g) acerola and ciriguela mixed juice.

3.4. Stability of bioactive compounds in microcapsules

As the microcapsules prepared with the MX1 mixture showed good AA and PC retention in the mixed acerola-ciriguela juice and good AO according to both DPPH• and FRAP assays, they were submitted to stability tests in light-protected glass jars at storage temperatures of 5 and 30°C, the results of which are illustrated in Fig. 1.

After the first 7 days of storage at both temperatures, there was no significant reduction in the AA content, which, on the contrary, was only 3.3 and 5.0% after 15 and 30 days of storage at 5°C, and 12.3% after 15 days and 34.8% after 30 days at 30°C (Fig. 1a). As for the TC content, there was no significant difference between samples stored either at 5°C or at 30°C after 7 days of storage and at the beginning (Fig. 1b), while after 15 days it was fully preserved at 5°C, but suffered a 40.9% reduction at 30°C. Such a degradation reached, after 30 days, 27.0% at 5°C and no less than 65.1% at 30°C. Similar to what was observed for the AA and TC contents, there was no significant reduction in the PC content of samples stored for 7 days at either temperature (Fig. 1c), while there was a reduction of 9.3% at 5°C and 27.0% at 30°C after 15 days, and 13.5% at 5°C and no less than 86.5% at 30°C after 30 days.

Fig. 2 shows the results of AO measured by the DPPH• assay in stability tests carried out for 30 days at 5 and 30°C on microcapsules prepared with the MX1 mixture as a wall material. As expected due to the fact that AO depends especially on the PC content, it showed a similar behavior to that of PC in Fig. 1 (b), that is, it remained practically unchanged during the first 7 days of storage at both temperatures, and suffered a limited reduction after 15 days (by 2.0% at 5°C and 55.4% at 30°C), but a very pronounced one (by 21.5% at 5°C and 100% at 30°C) after 30 days.

Table 3
Antioxidant activity of microcapsules of 60:40 (g/g) acerola and ciriguela mixed juice obtained by spray drying using different combinations of wall materials.

Wall material	Percentage of DPPH• radical scavenging (%)	FRAP (mmol of ferrous sulfate/g of sample)
MX3	50.2 ± 0.9^{c}	586 ± 17^{d}
MX1	60.5 ± 0.5^{a}	819 ± 10^{b}
AX3	38.9 ± 0.5^{d}	653 ± 17^{c}
AX1	54.0 ± 0.3^{b}	675 ± 23^{c}
MAX	52.9 ± 2.2^{b}	889 ± 32^{a}

MX3: 99.7:0.3 (g/g) maltodextrin and xanthan gum; MX1: 99.9:0.1 (g/g) maltodextrin and xanthan gum; AX3: 99.7:0.3 (g/g) gum arabic and xanthan gum; AX1: 99.9:0.1 (g/g) gum arabic and xanthan gum; MAX: 49.9:49.9:0.2 (g/g/g) maltodextrin, gum arabic and xanthan gum. DPPH• = 2,2-diphenyl-1-picrylhydrazyl free radical; FRAP = ferric reducing antioxidant power. Values are expressed as mean ± standard deviation. Means followed by the same letter do not differ significantly by Tukey's test at 5% probability of the null being correct.

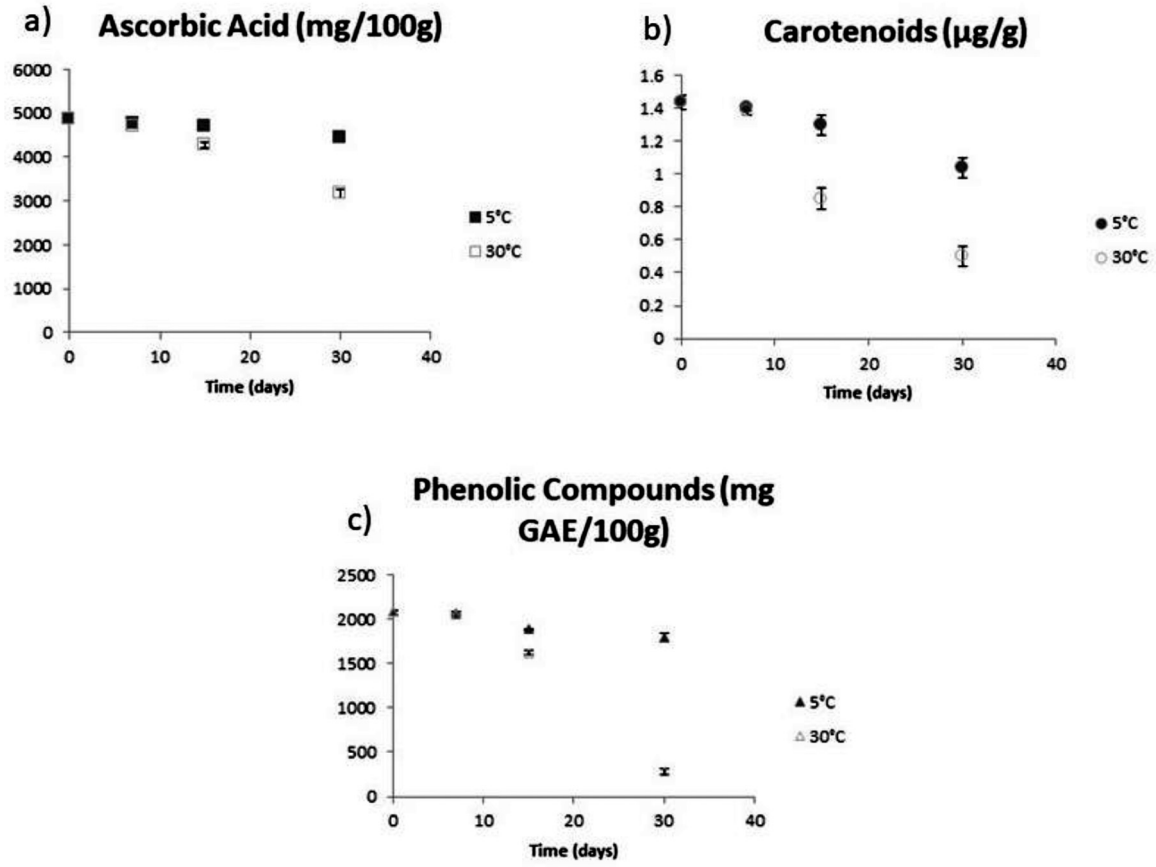

Fig. 1. Results of stability tests on microcapsules containing mixed juice of acerola and ciriguela obtained by spray drying using the MX1 mixture (99.9:0.1 g/g maltodextrin and xanthan gum) as wall material for a 30-day storage period at temperatures of 5°C (full symbols) and 30°C (empty symbols). The values of the contents of (a) ascorbic acid, (b) total carotenoids and (c) phenolic compounds are expressed as mean ± standard deviation. The statistical differences between mean values were determined by the Tukey's test at a 5% probability of the null being correct.

In general, an increased reduction in bioactive compounds when microcapsules were stored at the highest temperature was to be expected, as it is known that their stability decreases and their degradation accelerates with an increase in this parameter (Damodaran, 2017). In particular, the trend observed for PC and AO was qualitatively similar to that reported for microcapsules of plum juice stored at 4 and 25°C (Mishra, Brahma, & Seth, 2017). Similarly, Khalifa, Li, Mamet, & Li (2019) observed that blackberry juice microcapsules stored at 4°C maintained their OA for a period of 60 days.

3.5. Structure of microcapsules

Similar to the stability study, microcapsules were prepared with the MX1 mixture and subjected to scanning electron microscopy (SEM) analysis, the results of which are illustrated in Fig. 3.

SEM examination showed the formation of irregularly shaped microcapsule agglomerates with an approximate size of 10 µm as well as the absence of surface cracks and irregular structures with shrinkage on the particle surface (Fig. 3b). Furthermore, agglomeration occurred

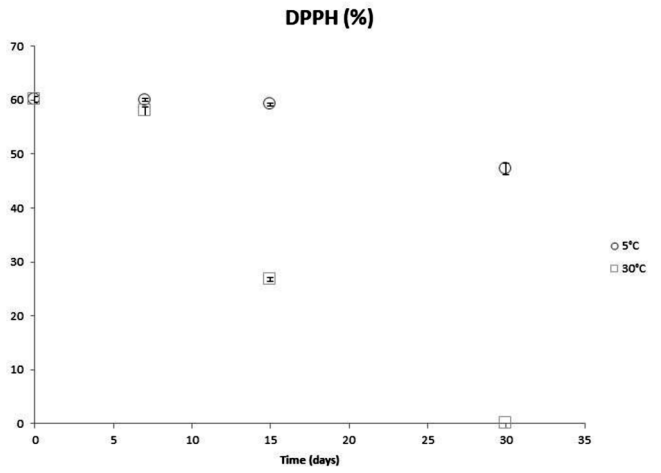

Fig. 2. Results of stability tests on microcapsules containing mixed juice of acerola and ciriguela obtained by spray drying using the MX1 mixture (99.9:0.1 g/g maltodextrin and xanthan gum) as wall material for a 30-day storage period at temperatures of 5°C and 30°C. The values of antioxidant activity percentage reduction determined by the DPPH• method are expressed as mean ± standard deviation. The statistical differences between mean values were determined by the Tukey's test at a 5% probability of the null being correct.

(Fig. 3a) as a result of holes in the surface of some particles, which indicate the existence of hollow structures. In these structures, the core material was evenly dispersed throughout a matrix at the particle perimeter, which can be explained by the fact that the coating and core materials are hydrophilic and combine uniformly to form film and membrane (Delia et al., 2019).

Wrinkled surface formations have been associated with shrinkage of microcapsules during the drying process, due to drastic loss of moisture and sudden cooling (Ortiz-Basurto, Rubio-Ibarra, Ragazzo-Sanchez, Beristain, & Jiménez-Fernández, 2017). Similar results were observed for the microencapsulation of acidified *Opuntia ficus-indica* (Gutiérrez, Utrilla-Coello, & Soto-Castro, 2018). Similar morphologies have been reported for pitaya juice microcapsules protected with maltodextrin (Shaaruddin, Ghazali, Mirhosseini, & Muhammad, 2017). In general, the absence of pores or cracks in microcapsules allows us to infer a low oxygen permeability, which can increase the stability of the color that is notoriously sensitive to the oxidizing atmosphere (Ortiz-Basurto et al., 2017).

4. Conclusions

Microcapsules containing mixed juice of acerola and ciriguela in the proportion of 60:40 (g/g) were produced by spray drying with different mixtures of maltodextrin (MD), arabic gum (GA) and xanthan gum (XG) as wall materials. They showed satisfactory ascorbic acid (AA) retention, which indicates that all of them could be used to successfully preserve this bioactive compound during microencapsulation. On the contrary, none of the mixtures proved to be effective to preserve total carotenoids (TC) during microencapsulation, although those with the highest XG concentration (0.3%) had the best performance. An opposite behavior was observed for the phenolic compounds (PC), which were best preserved using the mixtures with the lowest XG concentration (0.1%). Microcapsules obtained with the 99.9:0.1 (g/g) MD and XG binary mixture (MX1) and the 49.9:49.9:0.2 (g/g/g) MD, GA and XG ternary mixture (MAX) exhibited the highest antioxidant activity measured by both the DPPH• radical scavenging assay and the ferric reducing antioxidant power (FRAP) assay.

The microcapsules obtained with the MX1 blend are the most cost-effective, as they use the cheapest blend of wall materials and have good AO and total AA and PC retention capacity. Therefore, this mixture, which has better chances for industrial application, was used to prepare microcapsules to be submitted to stability tests for 30 days. As expected, microcapsules stored at low temperature (5°C) showed better preservation of bioactive compounds (AA, TC and PC) and AO than those stored at 30°C. Finally, the overall results of stability tests performed on microcapsules containing the 60:40 (g/g) acerola and ciriguela mixed juice prepared using the MX1 mixture suggest that the microencapsulated product, in order to be able to adequately preserve AA, PC, TC and AO for food and commercial purposes, should be kept refrigerated (5°C) for a storage period not exceeding 15 days. In general, the microcapsules did not show pores or cracks, which allows us to infer a low oxygen permeability and, therefore, good oxidative stability.

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.

Acknowledgements

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001 and by the Fundação de Amparo à Ciência e Tecnologia de Pernambuco (FACEPE). Authors are also grateful to the National Institute of Science and Technology of Tropical Fruits and CNPq for the financial support (grant #309443/2019-9).

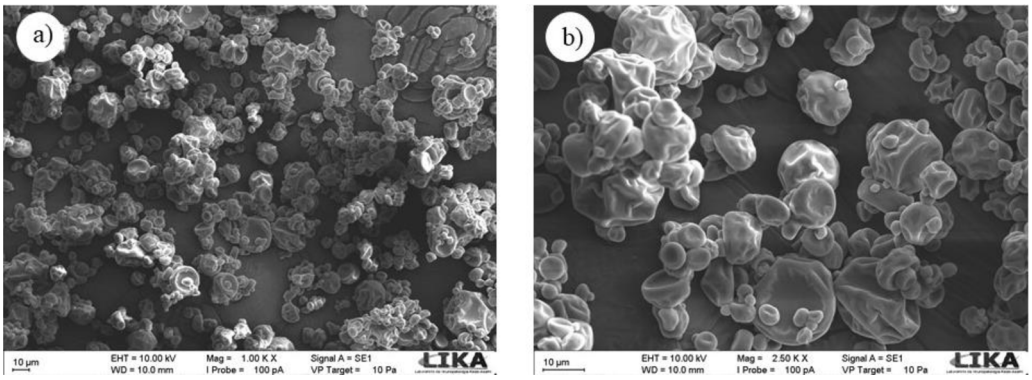

Fig. 3. Micrographs with magnification of (a) 1000x and (b) 2500x of the microcapsules containing mixed juice of acerola and ciriguela obtained by spray drying using the MX1 mixture (99.9: 0.1 g/g maltodextrin and xanthan gum) as wall material.

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