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ORIGINAL ARTICLE

Influence of combined hydrolyzed collagen and maltodextrin as carrier agents in spray drying of cocona pulp

Influência da mistura de colágeno hidrolizado e maltodextrina como agentes carreadores na secagem por atomização de polpa de cocona

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Abstract

This work aimed to evaluate the effect of carrier agents containing maltodextrin and protein, represented by hydrolyzed collagen on the spray drying process of cocona (*Solanum sessiliflorum* Dunal), and on the properties of the resulting powders. We used pre-established proportions between the solids of cocona pulp and the carrier agents (P:CA), and among carrier agents themselves, maltodextrin and hydrolyzed collagen, (MD:HC). The process was carried out in a spray dryer at an inlet air temperature of 120 °C. We prepared twelve feed solutions containing 20% of total solids, with P:CA ratios of 1:3, 1:4, 1:5 and 1:6, and MD:HC ratios of 0:100, 50:50, and 100:0. Solids recovery was obtained for the evaluation of the spray drying process. The cocona pulp powders were analyzed for moisture content, water activity, particle size distribution, mean particle diameter, chemical structure (FTIR) and color. For a P:CA of 1:6, for the sample formulated with hydrolyzed collagen only, solids recovery (96.2%) was much higher than that of the sample with maltodextrin only (39.2%). The chemical structure of cocona powders can be considered a sign of a good encapsulation process. The color of the cocona pulp powder was similar to that of the carrier agents. The formulation with highest content of hydrolyzed collagen improved the recovery of solids, guaranteed the cocona pulp encapsulation, and obtained fruit powders with bioactive properties.

Keywords: *Solanum sessiliflorum*; Powder; Microencapsulation; Protein; Particles; Recovery solids; FTIR.

Resumo

O objetivo deste trabalho foi avaliar diferentes tipos de formulações com maltodextrina e colágeno hidrolisado na secagem por atomização de cocona (*Solanum sessiliflorum Dunal*), e nas propriedades dos pós resultantes. Foram avaliadas proporções pré-estabelecidas de sólidos de polpa:sólidos de agente carreador (P:AC) e relação entre

agentes carreadores maltodextrina 10DE:colágeno hidrolisado (MD:HC). O processo foi realizado na secagem por atomização a 120 °C como temperatura de ar de entrada. Foram preparadas 12 soluções de alimentação com 20% de sólidos, variando-se a proporção de P:AC (1:3, 1:4, 1:5 e 1:6) e a relação MD:CH (0:100, 50:50 e 100:0). A recuperação de sólidos foi determinada para avaliar o processo de secagem. Os pós de cocona foram analisados quanto a umidade, atividade de água, distribuição de tamanho e diâmetro médio de partícula, estrutura química (FTIR) e cor. Na proporção P:CA de 1:6, a recuperação de sólidos foi de 96,2% para a amostra com colágeno hidrolisado, valor bem maior quando comparado com o valor da amostra com maltodextrina, que foi de 39,2%. A estrutura química dos pós de cocona pode ser considerada um sinal de um bom processo de encapsulamento. A cor dos pós foi similar à dos agentes carreadores. A formulação com maior teor de colágeno hidrolisado foi capaz de melhorar a recuperação de sólidos, garantir que a polpa de cocona fosse encapsulada e obter pós de frutas com propriedades bioativas.

Palavras-chave: *Solanum sessiliflorum*; Pó; Microencapsulação; Proteína; Partículas; Recuperação de sólidos; FTIR.

1 Introduction

Cocona (*Solanum sessiliflorum* Dunal) is a fruit from Amazon, that is spread around several countries, including the Pacific coast of Colombia, Venezuela, Ecuador, Peru, and Brazil. In these regions, it is used as medicine, food or as a cosmetic (Jiménez, 2018; Serna-Cock et al., 2015). The taste and aroma of cocona are very exotic. Due to its phenolic compounds, carotenoid, pectin, citric acid, iron, and niacin (vitamin B5) content and its antioxidant capacity, cocona has a market potential because of the population current concern with a healthy diet (Cardona et al., 2011; Mascato et al., 2015; Rodrigues et al., 2013; Sereno et al., 2018; Silva et al., 2016). This fruit is recommended for people who have energy intake restrictions (Silva Filho et al., 2005) or high levels of cholesterol and triglycerides in their blood (Pardo, 2004; Yuyama et al., 2005).

This fruit has a climacteric respiration pattern and, thus, storage, transport, and handling may affect its cellular turgor, weight and mechanical properties. It is, therefore, suitable for consumption, in average, up to the fifth day of storage after harvest (Andrade Junior et al., 2017). Spray drying of cocona can be an alternative process to extend its shelf-life (Ferrari et al., 2012a; Oliveira et al., 2013). However, this process is hindered due to the low molecular weight (LMW) sugars and organic acids present in the fruits. These compounds have low glass transition temperature (Tg) and high hygroscopicity (Moser et al., 2017; Samborska et al., 2015), which, upon drying, lead to high stickiness in the drying chamber, causing operational issues and low solids recovery (SR) of the final product. Therefore, the use of a carrier agent of high molecular weight (HMW) is required (Can Karaca et al., 2016).

Carrier agents such as starches, gums, proteins, and cyclodextrins can be used in the spray drying of fruit juices and pulps to reduce the stickiness phenomenon, thereby improving the SR and, in addition, ensuring the stability of the bioactive compounds (Akhavan Mahdavi et al., 2016). A single carrier agent does not present all these features and, therefore, many studies currently use different proportions of combined starches and proteins to improve the powder's properties, the bioactive compounds protection, and SR (Du et al., 2014; Moser et al., 2017; Robert et al., 2015; Shi et al., 2013). The juice and fruit pulp industry requires high amounts of carrier agents to enhance process yield. Maltodextrin (MD) (40-60%, g/100 g feed solution) is generally used because it is inexpensive and film-forming material. However, it has an amorphous nature under high relative humidity, hence becoming sticky after absorbing water (Wang et al., 2013). On the other hand, proteins form smooth and non-sticky films, resulting in higher SR values, even when added in small amounts (Fang & Bhandari, 2012; Fang et al., 2013). Currently, protein is combined to starches as a carrier agent in order to increase the emulsifying properties, solubility, antioxidant effect, SR and powder fluidity (Moser et al., 2017; Muzaffar & Kumar, 2016).

Hydrolyzed collagen (HC) is a natural protein derived from collagen found in animal skins and bones (bovine, pig, poultry, and fish). It is colorless and has emulsifying, stabilizing, film-forming properties, among others; while also increasing the solubility of the encapsulated product. Its biological properties are notorious for protection of articular cartilages under stress conditions; relief of osteoarthritis and osteoporosis symptoms (García-Coronado et al., 2019; Puigdellivol et al., 2018); stimulation of bone-forming cells; improvement of calcium absorption (Daneault et al., 2017); protection and recovery of connective tissue in response to intense strength and cardiovascular training (Lopez et al., 2015); and, lastly, reduction of visible signs of skin natural aging (Borumand & Sibilla, 2014, 2015; Proksch et al., 2014). Due to its physicochemical and biological properties, HC has been used in the elaboration of functional fruit pulp beverages, hence improving their nutraceutical characteristics (Bilek & Bayram, 2015; Butzge et al., 2014; Rigoto et al., 2018).

This study aimed to evaluate the effects of different proportions of solids of cocona pulp and carrier agents (P:CA), and of different ratios of the carrier agents maltodextrin 10DE and hydrolyzed collagen (MD:HC) on SR, and on the powder characteristics of moisture content, water activity, size distribution, mean particle diameter, chemical structure (FTIR) and color of particles obtained by spray drying.

2 Materials and methods

2.1 Material

Cocona (*Solanum sessiliflorum* Dunal) fruits were acquired from General Warehouses Company of São Paulo (CEAGESP), São Paulo, Brazil, with maturation degrees between 4 and 5, in which peels presented a green to yellowish coloration. The fruits were processed with knife pulping, discarding their peels and seeds. Cocona pulp was filtered through a Tyler sieve of 115 mesh, with an opening of 0.125 mm, in order to withdraw large solids and facilitate their passage through the atomizer nozzle of the spray dryer. The pulp was stored in a freezer at -18 ºC and thawed according to the amount required for each test. Maltodextrin MOR-REX®1910 (10DE) from Ingredion (MD; Mogi-Guaçu, SP, Brazil) and hydrolyzed collagen powder of bovine origin NovaProm® hidro from NovaProm Food Ingredients (HC; gel strength (bloom) = 0); Lins, SP, Brazil) containing 5.5% and 8.0% moisture content, respectively, were used as carrier agents.

2.2 Physicochemical composition of cocona (Solanum sessiliflorum *Dunal*) pulp filtered

The cocona pulp filtered was analyzed for moisture (Method 934.01, vacuum oven), protein content multiplying the conversion factor of 6.25 (Method 984.13), lipid content (Method 920.39), total fiber (Method 978.10), and ash content (Method 942.05), according to methods recommended by the Association of Official Analytical Chemists (2006). Carbohydrate content was calculated using the following formula: Available carbohydrate (%) = 100 – [protein (%) + Moisture (%) + Ash (%) + Fiber (%) + Crude Fat (%)]. Total sugar was quantified by DNS method (Miller, 1959). The titratable acidity was expressed as percentage of citric acid and total soluble solids were determined following the methods 947.05 and 990.20, respectively (Association of Official Analytical Chemists, 2006). pH was measured using a pH meter (Mettler Toledo MP225). The values are presented on a wet basis (w.b.).

2.3 Sample preparation

We prepared twelve feed solutions with 20% total solids consisting of both filtered pulp and carrier agents. The pre-established ratios of solids of pulp to solids of carrier agent (P:CA) were 1:3, 1:4, 1:5, and 1:6, while those for maltodextrin to hydrolyzed collagen (MD:HC) were 0:100, 50:50 and 100:0 (Table 1). The carrier agents were added directly to the pulp under magnetic stirring until complete dissolution, that is, for 30 min.

Table 1. Formulations of feed solutions and experimental values of solids recovery, moisture content, water activity and mean particle diameter $D_{[4,3]}$ of powder obtained by spray dryer.

	Feed solutions		Solids recovery (%)	Moisture content (%)	Water activity	Mean diameter $D_{[4,3]}$ (µm)
	P:CA	MD:HC				
1	1:3	100:0	41.2 ± 0.5^{Ca}	1.6 ± 0.1^{Cbc}	0.156 ± 0.005^{Bb}	10.343 ± 0.757^{Bb}
2		50:50	56.1 ± 2.0^{Bb}	2.1 ± 0.1^{Bb}	0.213 ± 0.015^{Ab}	9.353 ± 0.110^{Ab}
3		0:100	90.2 ± 0.5^{Ab}	2.7 ± 0.1^{Aab}	0.142 ± 0.005^{Bc}	10.526 ± 0.039^{Bc}
4	1:4	100:0	34.4 ± 0.1^{Cb}	1.1 ± 0.1^{Bd}	0.161 ± 0.004^{Cb}	12.846 ± 0.651^{Cb}
5		50:50	50.9 ± 2.2^{Bc}	2.6 ± 0.1^{Aa}	0.219 ± 0.006^{Ab}	11.124 ± 0.070^{Ab}
6		0:100	92.5 ± 1.1^{Aab}	2.7 ± 0.1^{Aab}	0.194 ± 0.012^{Bb}	10.788 ± 0.068^{Bb}
7	1:5	100:0	39.1 ± 1.9^{Ca}	1.9 ± 0.1^{Bbc}	0.152 ± 0.004^{Cb}	10.889 ± 0.074^{Cb}
8		50:50	59.1 ± 0.6^{Bab}	2.3 ± 0.3^{Bab}	0.248 ± 0.005^{Aa}	9.083 ± 0.046^{Aa}
9		0:100	94.7 ± 1.5^{Aab}	3.0 ± 0.2^{Aa}	0.200 ± 0.004^{Bb}	9.234 ± 0.007^{Bb}
10	1:6	100:0	39.2 ± 0.6^{Ca}	2.8 ± 0.1^{Aa}	0.247 ± 0.006^{Aa}	15.683 ± 0.652^{Aa}
11		50:50	61.3 ± 0.2^{Ba}	2.4 ± 0.1^{Cab}	0.210 ± 0.003^{Cb}	8.795 ± 0.017^{Cb}
12		0:100	96.2 ± 3.0^{Aa}	2.6 ± 0.1^{Bb}	0.224 ± 0.001^{Ba}	10.688 ± 0.022^{Ba}

P:CA = Proportion of solids of cocona pulp and solids of carrier agent. MD:HC = carrier agents maltodextrin and hydrolyzed collagen ratio. The values represent the average between three determinations ± standard deviation. Lowercase and uppercase letters represent the response variation with P:CA and MD:HC, respectively. Averages with the same letters, either lowercase or uppercase, represent no significant difference ($p \leq 0.05$) by Tukey test.

2.4 Spray drying of feed solutions

The feed solutions (Table 1) were added to a cylindrical glass chamber of a laboratory scale spray dryer (SD-06, LabPlant, Reino Unido), with a diameter of 215 mm and a length of 500 mm. The spray nozzle diameter was set at 1.0 mm, the feed flow rate at 4.7 mL/min, the air velocity at 3.9 m/s, the compressed air pressure at 2 x 10^5 Pa and the inlet, and outlet air temperatures at 120 ± 2 ºC and 80 ± 2 ºC, respectively. The temperature of the feed solutions was of 20 ºC.

2.4.1 Solids recovery

Solids recovery was calculated as the ratio of the masses of total solids in the resulting powder ($m_{sol,powder}$) to the feed solution ($m_{sol,feed}$) (Equation 1).

$$SR(\%) = \frac{m_{sol,powder}}{m_{sol,feed}} \times 100 = \frac{m_{powder} \times X_{sol,powder}}{m_{feed} \times X_{sol,feed}} \times 100 \quad (1)$$

Where: SR is solids recovery (%), m is mass (g), X_{sol} is solids content (g/g of powder or feed solution).

2.5 Analytical methods

2.5.1 Moisture content and water activity

Powder moisture content (%) was determined using a vacuum oven at 70 ºC under reduced pressure (13.3 kPa) for 48 h (Association of Official Analytical Chemists, 2006). Water activity was measured by a digital water activity meter by Aqualab (3TE, Decagon, Pullman USA) at 25 ºC.

2.5.2 Size distribution and mean particle diameter

Particle size distribution was measured using a laser light diffraction instrument (Model Mastersizer 2000, Malvern Instruments Ltd, Malvern, UK). Powder samples were dispersed in ethanol 99.5% and each submitted to 5 readings. The mean particle size was expressed as $D_{[4,3]}$.

2.5.3 FTIR spectroscopy

FTIR spectrum of MD, HC, freeze dried cocona pulp and powder of ratios 1:6 was acquired on a FTIR Spectrophotometer (Nicolet 6700, Thermo Scientific, USA). A small portion of the sample was transferred to the agate mortar. KBr was added in the proportion of 0.5:100 to prepare the pressed tablet (~ 7 ton for 4 min). It was then taken to the equipment for analysis. The measurement was performed in transmittance mode using the snap-in baseplate accessory (KBr method). Spectrum was recorded (32-64 scans) in the transparent mode from 4000 to 600 cm^{-1}, at 4 cm^{-1} resolution.

2.5.4 Color analysis

Sample color was measured using a colorimeter (UltraScanVis, HunterLab, Reston, VA, USA), with a D65 illuminant and a 10º observer angle, expressed by the CIELab scale (L*, a*, and b*), chroma (C*) and hue angle (Hº). Color difference (ΔE) was calculated by Equation 2.

$$E = \sqrt{\left[\left(L^* - L_0^*\right)^2 + \left(a^* - a_0^*\right)^2 + \left(b^* - b_0^*\right)^2\right]} \quad (2)$$

Where: L* is the luminosity; a* and b* are the intensities of the green-red color and the blue-yellow color, respectively. The variables without subscript correspond to the spray dried samples. The subscript '0' denotes a reference sample. In this study, four reference samples were used: pure freeze-dried cocona pulp, each carrier agent, isolated, and its 50:50 mixture.

2.6 Statistical analysis

The tests were carried out in triplicate and the results shown as their mean ± standard error. Results were compared by analysis of variance (ANOVA), with the Tukey test at a 5% level of significance ($p \leq 0.05$), using the Software Microsoft® Excel 2018 (Microsoft Corporation, CA).

3 Results and discussion

The results for the physicochemical composition of cocona pulp filtered (Table 2) were similar to those previously reported in literature (Jiménez, 2018; Serna-Cock et al., 2015): a content high in sugar (48.16%) and acidity (1.94%), justifying the addition of carrier agents to reduce the stickiness during spray drying and powder storage.

Table 2. Physicochemical composition of cocona (*Solanum sessiliflorum* Dunal) pulp filtered.

Analysis		Mean value	Method
Moisture	%, w.b.	94.01 ± 0.01	(Association of Official Analytical Chemists, 2006)
Protein	%, w.b.	0.64 ± 0.03	(Association of Official Analytical Chemists, 2006)
Lipid	%, w.b.	0.65 ± 0.04	(Association of Official Analytical Chemists, 2006)

Table 2. Continued…

Analysis		Mean value	Method
Total fiber	%, w.b.	0.09 ± 0.04	(Association of Official Analytical Chemists, 2006)
Ash	%, w.b.	0.39 ± 0.02	(Association of Official Analytical Chemists, 2006)
Carbohydrate	%, w.b.	4.22 ± 0.16	By difference
Total sugar	%, w.b.	2.88 ± 0.03	(Miller, 1959)
Titratable acidity (citric acid)	%, w.b.	1.94 ± 0.004	(Association of Official Analytical Chemists, 2006)
Total soluble solids	ºBrix, w.b.	4.80 ± 0.35	(Association of Official Analytical Chemists, 2006)
pH		3.05 ± 0.01	pH meter

w.b. = wet basis. Values represent the average of three determinations ± standard deviation.

3.1 Defining the spray drying conditions

Operational conditions of the spray dryer were chosen by preliminary tests (Supplementary Material). The feed flow rate at 4.7 mL/min was chosen, because a great amount of product adhered to the drying chamber at higher feed flow rates.

Dripping was observed inside the drying chamber when we used a maximum feed flow rate (18.17 mL/min), indicating that the compressed air was insufficient to properly atomize the feed solution into the chamber, a fundamental prerequisite for drying and subsequent formation of particles. Therefore, the minimum feed rate (4.67 mL/min) was tested to ensure the occurrence of spray drying of the feed solution and, thus, an increase in the process yield. At low feed rates, the atomized droplets in the drying chamber were smaller and, consequently, the total surface area in contact with hot air was increased. Therefore, higher heat and mass transfers occurred in the dryer. Similarly, upon evaluating the spray drying of pomegranate juice, Thirugnanasambandham & Sivakumar (2015) found that by increasing the feed rate of the process, the heat transfer between the atomized droplets and the drying air was less efficient, resulting in a moister and stickier powder.

A low temperature of the inlet air (120 ºC) was chosen since high temperatures favored the thermal degradation of bioactive compounds present in fruits (Tolun et al., 2016). Mishra et al. (2014) showed that the content of phenolic compounds in amla juice powder (*Emblica Officinalis*) decreased significantly when the inlet air temperature increased from 125 ºC to 175 ºC, and that high temperatures in spray drying of lemon juice negatively affected the structure of phenolic compounds as well, hence diminishing its antioxidant capacity (Mishra et al., 2015).

3.2 Solids Recovery (SR)

An increase in the ratio of pulp solids to hydrolyzed collagen improved the SR from 90.2% to 96.2% (Table 1), indicating that stickiness in the drying chamber was minimized. For the feed solution formulated with MD only (100:0), the increase from 1:3 to 1:6 in the P:CA proportion did not favor SR, with no significant difference found upon comparison. The low SR values for formulations with MD only indicated that most of the solids adhered to the wall of the drying chamber. For samples with both MD and HC (50:50), SR was higher than 50%. Fang & Bhandari (2012) observed a significant increase in SR when whey protein isolate (WPI) was used as a carrier agent for spray drying of bayberry juice. By adding 1% of that protein to the formulation, the authors obtained an SR superior to 50%. However, in order to achieve a similar value when MD replaced WPI as a carrier agent, the authors were required to add more than 30% of MD to the

formulation. Shi et al. (2013) observed a positive effect of the addition of WPI on the SR in the spray drying of honey with different ratios of WPI to MD. Therefore, these proteins may totally or partially replace hydrolyzed starch in carrier agent formulations due to their HMW, chain flexibility, and emulsifying ability, which all contribute to increase SR (Akhavan Mahdavi et al., 2016; Robert et al., 2015).

It is noteworthy that the molecular weight (MW) of MD 10DE is 1700 g/mol (Avaltroni et al., 2004), smaller than that of HC (2000-6000 g/mol) (Daneault et al., 2017). Thus, the addition of HC can improve solids recovery on spray drying, counterbalancing the LMW of the sugars and organic acids of cocona pulp.

When subjected to hot air inside the drying chamber, protein migration to the air-droplet interface results in the formation of a high-content protein film on the surface of dried particles (Shi et al., 2013). This new layer has a high Tg, hindering the adhesion of the particles to the drying chamber and, thus, improving product yield (Muzaffar & Kumar, 2016; Samborska et al., 2015). Hence, this film may be the reason for the high recovery of the cocona pulp powder found in this study.

Moser et al. (2017) obtained similar SR values on the spray drying of grape juice when MD was used as a carrier agent combined to whey protein concentrate (WPC) and soy protein isolate (SPI). SR raised proportionally to increases in the concentration of protein, regardless of the type used. On the other hand, the effect of protein to total carrier agent ratio did not significantly change SR.

Currently, the fruit pulp powder industry has a greater interest in processes with higher yields and less waste generation (Akhavan Mahdavi et al., 2016; Muzaffar & Kumar, 2016; Shi et al., 2013; Zareifard et al., 2012). Thus, the formulation of cocona pulp containing only with HC (MD:HC 0:100) with a P:CA 1:6 ratio resulted in higher solids recovery in this study (96.2%). This result is a key factor when considering economic benefits.

3.3 Moisture content and water activity

Moisture content is a powder property to assess drying efficiency. It is related to powder flowability, stickiness and storage stability. The results showed that the moisture content presented a significant difference among cocona pulp powders, ranging from 1.1% to 3.0% (Table 1). The values are consistent to those observed in industrial spray drying (Moser et al., 2017). Higher moisture contents were observed in powders containing greater amounts of HC due to the strong ability of proteins to bind in water. Shi et al. (2013) observed a similar behavior by adding MD and SPI to spray dried honey.

All samples had water activity values below 0.3, which increased the storage stability of powders since there is little water available for microorganism growth and for biochemical reactions (Santana et al., 2017).

3.4 Distribution and mean particle diameter

Figure 1 shows the particle size distribution of the sample with the highest SR, with a P:CA of 1:6 and an MD:HC of 0:100. Particles had diameters ranging from 0.3 to 282 μm and a monomodal size distribution with a multimodal tendency, that is, a predominant peak with two small peaks. The other samples showed a similar behavior. The origin of the larger particles can be attributed to the beginning of the agglomeration process, in which the formation of irreversible link bridges between particle-particle leads to the production of large particles (Moser et al., 2017). The onset of particle agglomeration produced by spray drying of fruit pulp is a physical phenomenon that is affected by the glass transition temperature of the wall material and the hygroscopicity of the particles. Little agglomeration, demonstrated by the third peak of the particle size distribution, maybe due to the absorption of ambient humidity by the particles during packing, and the low temperature of the spray dryer air used (120 °C) as described by Wang et al. (2013) and Du et al. (2014), respectively.

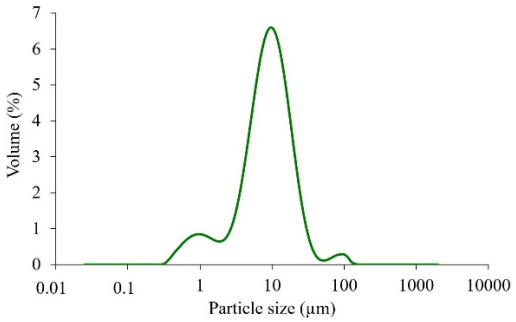

Figure 1. Distribution of the particles size of the cocona pulp powder with hydrolyzed collagen at a P:CA of 1:6 and an MD:HC of 0:100.

The average particle diameter for all samples was less than 16 µm (Table 1), which can generate good retention of bioactive compounds of the fruit, as explained by Moser et al. (2017) in its anthocyanin retention study (94%) in powdered grape juice with a particle size of 11.57 µm. Generally, small particles are characteristics of powders obtained by spray drying (Gong et al., 2018; Kuck & Noreña, 2016). However, small particle size results in a large total surface area of the system exposed to the environment, favoring the degradation of susceptible compounds present in the fruit pulp, as described by the author. Also, powders composed by small particles lack reconstitution, such as low wettability, which can be improved with stirring and high temperatures during rehydration (Fitzpatrick et al., 2016). Other alternative is to subject spray dried powders to agglomeration process, using a fluidized bed granulator and dryer, in order to improve their reconstitution properties (Ferrari et al., 2012b).

3.5 FTIR spectroscopy

Figure 2 shows the FTIR-spectra of MD, HC, and freeze dried cocona powders, and spray dried sample (P:AC 1:6). The peak of the 2350 cm^{-1} region was disregarded as the CO_2 (g) variation of the environment at the time of measurement. Baseline correction of the spectrum of the cocona pulp was performed. The broadband in the 3700 - 2800 cm^{-1} region can be attributed to the stretching vibration of free and inter-associated hydroxyl groups (O– H) of carbohydrates, carboxylic acids, and residual water of the materials used (Akbarbaglu et al., 2019).

In the structure of HC (Figure 2, curve A), the peaks associated with 3363 cm^{-1} (O-H stretch) and 2960 cm^{-1} (N-H stretch) were shown. The spectrum showed peaks at 1652, 1550, 1455, 1404, and 1249 cm^{-1} similar to the collagen results found in the study of Ding at al. (2015). The region between 800 and 1800 cm^{-1} is considered as a quite useful area for analyzing protein compound, because the peaks in the 1655 and 1537 cm^{-1} are typical of amide I, due to carbonyl stretching (C=O), and are mainly associated with random coil and amide II vibrations which are the result of deformation of the N-H bonds and the C-N bonds stretch (1550 cm^{-1}). This depends of the secondary structure of protein and peptides. The peaks at 1455 and 1404 cm^{-1} are associated to the C-N stretch and C-0 stretch vibrations in the amide II area at short peptide chains. Besides, the observed peak in the 1249 cm^{-1} spectrum is associated with the vibrations in the plane of amide III (Hameed et al., 2015).

The maltodextrin spectrum showed the bands at 3395 cm^{-1} (O-H stretch), 2932 cm^{-1} (C-H stretch), 1638 cm^{-1} (H_2O absorbed in amorphous region), 1417 cm^{-1} (CH_2 bending), 1154 and 1079 cm^{-1} (C-O-H bending, C-O stretch, typical of carbohydrates), 1025 cm^{-1} (angular deformation of =CH and =CH_2 bonds, carbohydrates peak), 931 and 857 cm^{-1} (deformation of CH2 and C1-H), 762 and 711 cm^{-1} (structural condition of the Pyranose ring) (Akbarbaglu et al., 2019).

No spectrum of cocona pulp has been reported in the literature. In the current work, we show, as seen in Figure 2 (curve C), that peaks at 3413 and 1400 cm^{-1} were assigned by the O-H stretch and bending of

alcohols and phenols. The peaks at 2927 and 2848 cm^{-1} were formed by the asymmetric and symmetric stretches of C-H (alkanes) and =CH, characteristic of aldehyde, respectively. The peak at 1725 cm^{-1} was associated with C=O stretching (aliphatic ketone). The peak at 1636 cm^{-1} was typical of amide I (N-H bending). The peaks at 1230 and 1078 cm^{-1} were caused by C-N stretching (aromatic amines). The peak of 1050 cm^{-1} was C-O stretching coupled with C-O bending of the C-OH of carbohydrates. The peak at 784 cm^{-1} was produced by =CH-H or C-H bending (alkenes and aromatics) (Namani et al., 2016; Nnorom & Onuegbu, 2019; Rizwana et al., 2019; Talari et al., 2017).

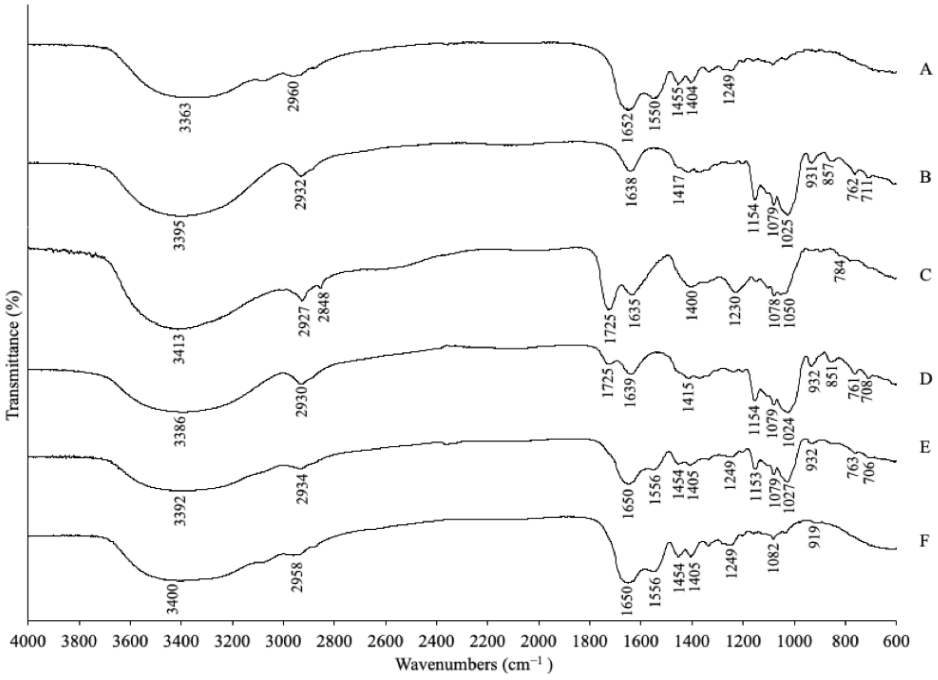

Figure 2. FTIR-spectra of the hydrolysate collagen (A), maltodextrin (B), freeze dried cocona pulp (C) and powder of P:AC 1:6 and MD:HC 100:0 (D), 50:50 (E) and 0:100 (F).

The FTIR results for cocona spray dried powders maintained the characteristic peaks of pulp at 1725 cm^{-1} in MD:HC 100:0 powder (Figure 2, curve D), and 1082 cm^{-1} in MD:HC powder 0:100 (Figure 2, curve F). The other characteristics peaks of cocona pulp (3413, 2927, 2848, 1636, 1230, 1050, and 784 cm^{-1}) disappeared in the microparticles spectrums, which can be considered as a sign of a good encapsulation process (Medina-Torres et al., 2016). In the case of the powder with pure MD and HC, they maintained the characteristic peaks of carbohydrate (1154 to 1024 cm^{-1}) (Figure 2, curve D) and protein (1650, 1556, 1055, 1405, and 1249 cm^{-1}) (Figure 2, curve F), respectively. The powder with the 50:50 ratio showed the characteristic peaks of the two carrier agents used (Figure 2, curve E). The FTIR results demonstrated no change in the chemical structure of the wall materials, since no new peaks were evidenced in the powdered cocona spectrum. They can interact with each other to form the particle wall, where the cocona pulp can also associate through hydrogen bonds (Jafari et al., 2008). Similar results was observed in other studies for spray drying using maltodextrin and protein as carrier agents (Akbarbaglu et al., 2019; Delia et al., 2019; Medina-Torres et al., 2016; Shao et al., 2019; Yingngam et al., 2018).

3.6 Color analysis

Color results for cocona pulp powders are shown in Table 3. We observed no significant differences for L* and C* values of the spray dried powders. However, these powders presented higher brightness values than the freeze-dried pulp, due to greater luminosity of the carrier agents. The C* values indicated a lower

intensity in the spray dried powders than in the freeze-dried pulp. The tonality of the spray dried samples was light yellow, as verified by the Hº values, of approximately 90º. Caparino et al. (2012) reported similar results for L* of spray dried mango powder due to the addition of MD to its formulation.

Table 3. Experimental values of color: luminosity (L*), chroma (C*) and hue angle (Hº) of spray dried cocona pulp. Color difference (ΔE) between spray dried powders, freeze-dried pulp (reference) and the carrier agents maltodextrin (MD) and hydrolyzed collagen (HC).

Feed solutions			Color						Color difference (ΔE)			
									Reference sample			
	P:CA	MD:HC	L*		C*		Hº		Freeze-dried pulp		Carrier agents	
1		100:0	90.83	± 0.34^{Ab}	11.62	± 0.61^{Aa}	89.61	± 0.13^{Bc}	27.75	± 1.32^{Aa}	8.78	± 0.96^{Aa}
2	1:3	50:50	91.66	± 0.21^{Ab}	11.44	± 0.23^{Aa}	90.34	± 0.16^{Ab}	28.49	± 1.23^{Aa}	2.30	± 0.12^{Cb}
3		0:100	90.76	± 0:34^{Abc}	11.64	± 0.37^{Aa}	89.87	± 0.32^{ABd}	27.69	± 0.46^{Ab}	4.59	± 0.87^{Ba}
4		100:0	91.71	± 0.45^{Ab}	10.42	± 0.50^{Aab}	90.26	± 0.08^{Bb}	29.20	± 1.58^{Aa}	7.30	± 0.24^{Aab}
5	1:4	50:50	91.67	± 0.44^{Aab}	10.63	± 0.76^{Aab}	90.81	± 0.11^{Ab}	29.12	± 0.46^{Aa}	2.76	± 0.20^{Cb}
6		0:100	90.43	± 0.56^{Bc}	11.86	± 0.51^{Aa}	90.48	± 0.09^{Bc}	27.34	± 0.40^{Ab}	4.42	± 1.04^{Ba}
7		100:0	92.01	± 0.11^{Aa}	9.30	± 0.73^{Ab}	90.51	± 0.16^{Bab}	30.18	± 1.53^{Aa}	6.22	± 0.36^{Ab}
8	1:5	50:50	92.22	± 0.44^{Aab}	9.98	± 0.59^{Aab}	90.82	± 0.20^{Abb}	29.87	± 1.03^{Aa}	3.25	± 0.71^{Bab}
9		0:100	91.72	± 0.29^{Aab}	10.95	± 0.69^{Aab}	91.22	± 0.12^{Ab}	28.89	± 0.42^{Aab}	5.21	± 1.16^{Aba}
10		100:0	91.93	± 0.41^{Aa}	9.29	± 0.48^{Ab}	90.81	± 0.28^{Ba}	30.13	± 0.63^{Aa}	6.27	± 1.15^{Ab}
11	1:6	50:50	92.83	± 0.49^{Aa}	9.13	± 0.67^{Ab}	91.41	± 0.32^{ABa}	30.90	± 0.78^{Aa}	3.93	± 0.16^{Ba}
12		0:100	92.31	± 0.17^{Aa}	9.85	± 0.46^{Ab}	91.90	± 0.09^{Aa}	30.07	± 1:18^{Aa}	6.36	± 0.25^{Aa}
Freeze-dried pulp			70.86	± 0.47	30.55	± 1.14	79.23	± 0.21				
MD		100:0	95.93	± 0.23	4.54	± 0.56	97.84	± 0.76				
HC		0:100	91.18	± 0.37	16.09	± 0.63	93.27	± 0.21				
MD:HC		50:50	92.92	± 0.24	13.00	± 0.49	94.61	± 0.24				

P:CA = Proportion of fruit pulp solids and carrier agents. MD:HC carrier agents maltodextrin and hydrolyzed collagen. The values represent the average of three determinations ± standard deviation. Lowercase and uppercase letters represent the response variation for multiple P:CA and MD:HC, respectively. Averages with the same letters lowercase or uppercase indicate that there is no significant difference ($p \leq 0.05$) by Tukey test.

The color difference (ΔE) between the powders obtained by spray drying and freeze-drying compared with the carrier agent, ranged from 2.30 to 8.78 and 27.34 to 30.90, respectively (Table 3). These results indicated little differences in color between spray dried cocona pulp and carrier agent mixture (2.30 to 3.93). This similarity of colors suggests that the carrier agents coated/encapsulated the solids of cocona (Figure 3). The increase in lightness and decrease color saturation (chroma) were due to the dilution effect (1:6) caused by carrier agent addition to cocona pulp, resulting in loss of color for pulp. Similar results were found to powdered pink guava with MD (Shishir et al., 2014), tamarind pulp with MD - SPI (Muzaffar et al., 2016), and grape juice MD, WPI - SPI (Moser et al., 2017).

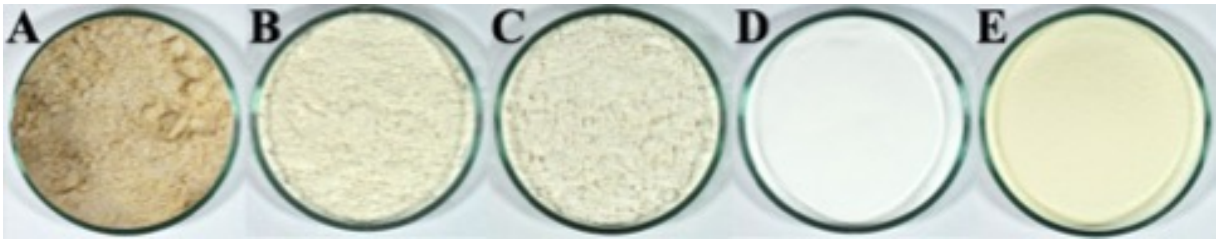

Figure 3. Cocona pulp obtained by freeze-drying (A), cocona pulp powders obtained by spray drying with formulations P:CA 1:6 MD:HC 0:100 (B) and P:CA 1:6 MD:HC 100:0 (C), maltodextrin DE10 pure (D), and pure hydrolyzed collagen (E).

4 Conclusions

We evaluated the combined use of hydrolyzed collagen and maltodextrin as carrier agents on the spray drying of cocona pulp. The addition of HC favored the solids recovery in the spray drying process, reducing losses of the product in the drying chamber. The low values for moisture content and water activity guarantee the stability of the dried product. FTIR spectra showed stretch bands of carbohydrates characteristic of maltodextrin and protein in cocona powders. The absence of most of the cocona pulp peaks shows that the spray drying of cocona pulp can be considered as a good encapsulation process. The color of the powders is very similar to that of the carrier agents. In the present study, we show that HC is an effective additive in the drying of cocona pulp and may replace MD, either partially or fully. Moreover, the addition of HC to cocona pulp powder is relevant because it has potential benefits for human health.

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Supplementary Material

Supplementary material accompanies this paper.

Table 1. Feed solutions with 20% of total solids and experimental values of solids recovery 7 of cocona powder obtained by spray dryer under several feed flow rates.

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