biology and life sciences forum

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Proceeding Paper

Coupling Flash Vacuum Expansion and Spray Drying to Produce Stable Polyphenolic Extract from Coffee Exocarp †

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† Presented at the 4th International Electronic Conference on Foods, 15–30 Oct 2023; Available online: https://foods2023.sciforum.net/.

Abstract: In the present work, the effect of the Flash Vacuum-Expansion (FVE) process on the recovery of total phenolic content (TPC) from entire coffee peel was evaluated. The resulting extracts were microencapsulated by spray drying using maltodextrin (MD10) and arabic gum (GA). The FVE increased TPC by 20.97% in the ethanolic extracts. The use of MD10 allowed to obtain smaller particles, less hygroscopic, and with better color and flow properties, as well as a higher concentration of TPC. Both powders were considered microbiologically stable, based on humidity and water activity.

Keywords: emerging technology; agro-industrial waste; microencapsulation

Citation: Castro, U.R.M.; Sánchez, C.A.O.; Ortiz, M.A.V.; Cervantes, M.A.S.; Díaz, M.P.R.; Servent, A.; Arredondo, V.M.R. Coupling Flash Vacuum Expansion and Spray Drying to Produce Stable Polyphenolic Extract from Coffee Exocarp. *Biol. Life Sci. Forum* **2023**, *26*, x. https://doi.org/10.3390/xxxxx

Academic Editor(s): Name

Published: date

1. Introduction

Coffee is a priority crop in Mexico. This country is the tenth worldwide coffee producer. However, despite its economic importance, the use of this crop is restricted to the use of the grain. During coffee processing large amounts of waste are generated, and coffee peel is one of the most abundant one. Recently, the search for alternatives that allow to valorize this by-product has intensified. Most of them focus on the recovery of compounds from the peel to generate extract enriched in natural bioactive compounds, or with a sensorial interest [1]. An emerging technology with high potential for the recovery of phenolic compounds is the Flash Vacuum-Expansion (FVE) [2]. FVE is a mechanical process in which plant material is first steam-heated, generally between 60 and 95 °C, before the expansion stage, that is to say the introduction into a chamber vacuum (2 to 5 kPa) [3]. This pressure drop establishes the physical condition necessary to allow the instantaneous evaporation of constitutive water. It creates micro-channels inside the tissues and break a part of them, depending on processing condition intensities [3]. Entire fruits cab be process by FVE, in order to obtain puree enriched in phenolic compounds thanks to liberation of compounds from peels and seeds [4]. Its versatility is interesting to be an

alternative for the recovery of compounds of interest from entire coffee peel, mainly anthocyanins [5]. The extracts can be then use as food ingredient in powder form, through the coupling of FVE and microencapsulation process. The microencapsulation into a coating material can be achieved by several methods. However, spray drying is the most common technology used in food industry due to its low processing cost and its generalization in food industry for multiple applications [6].

The objective of the present work was to study the feasibility of the fabrication of a powder which can serve as a food ingredient. Whole coffee peels were extracts by FVE in solvent as a mild green processing and dried by spray-drying. The recovery of phenolic compounds, as well as the physicochemical characteristics of the microencapsulates generated using two different coating materials was in-depth.

2. Methods

2.1. Raw Material

The coffee peel of Arabica variety was pulled from cherry coffee in a state of maturity and disinfected with a 100-ppm solution of sodium hypochlorite. The coffee cherries were obtained from the town of Tenampa, Veracruz, Mexico, located at 1060 mbsl. Samples were collected on 10 randomly selected trees from a plantation of 350 coffee trees.

2.2. Flash Vacuum Expansion Processing

The FVE process was carried out in laboratory-scale equipment. For the experiment, around 1 kg of the entire coffee peel were introduced into the heating chamber at atmospheric pressure (101.3 kPa) and subjected to a 10 min thermal exposure time to the vapor flow [3]. Then, sample is instantly transferred into the vacuum chamber (3 kPa). The expansion also causes a rapid cooling of the sample which was collected and stored in hermetic amber bottles. The coffee peel treated by FVE it was used for the polyphenol extraction, the composition of the extraction solvent was selected based on previous work on anthocyanins extraction of coffee husks [7].

2.3. Spray Drying Processing

For the spray-drying process, two different encapsulation materials were used. The extract was mixed with a concentrated aqueous solutions of maltodextrin 10 DE and arabic gum as wall material to reach a final concentration of 30 ± 1% of soluble solids, with a ratio of core/wall of 1:1 (*v*/*v*). The mixture was then spray-dried in a Büchi B-290 mini spray dryer (Flawil, Switzerland). Dry conditions were the following: air inlet temperature of 160 ± 2 °C, air outlet temperature of 80 ± 3 °C, the feeding rate of 0.39 $L \cdot h^{-1}$ and a volumetric flow rate of 667 $L \cdot h^{-1}$. Samples were named MD10 and GA if they were encapsulated by maltodextrin or arabic gum respectively.

2.4. Physicochemical Characterization of Powders

MD10 and GA powders were characterized in terms of: moisture content according to the gravimetric method described by the AOAC [8].; water activity measured using a water activity meter at 25 °C; hygroscopicity determined according to Cai and Corke [9], briefly, samples (about 2 g) from the Petri dishes were placed at 25 °C in an hermetic desiccator filled with Na_2SO_4 saturated solution (81% RH). After 1 week, the powders were weighed and expressed as g of moisture per 100 g dry solids (g/100 g); particle size determined by calculating the arithmetic mean of 300 particles, measured using a Motic BA310E optical microscope (NJ, USA); apparent and compact density calculated from the volume occupied by a known mass of dust in a graduated cylinder according to Santhalakshmy [10]; fluidity and cohesiveness of the powders calculated from the apparent and compacted density (expressed in Carr's index and Hausner's ratio respectively) [11,12]; total content of polyphenols expressed in mg gallic acid equivalent and determined using the Folin-Ciocalteu method as described by Singleton et al. [13], the

absorbance was measured at 765 nm using a spectrophotometer (Evolution 260, Thermo Scientific, Waltham, MA, USA), color properties were estimated using a CR400 colorimeter (Minolta, Osaka, Japan). The chroma and Hue parameters were evaluated according to the equations reported by Maskan [14].

2.5. Statistic Analysis

All determinations were carried out in triplicate, and the results are presented as means with standard deviation. Analysis of variance (ANOVA) and Tukey's test of means were used to identify significant differences, all the statistical analysis were performed using Minitab software v. 17 (PA, USA).

3. Results

3.1. Effect of the FVE on the Total Phenols Content

TPC in ethanolic extracts of coffee peel treated or not by FVE were 485.19 ± 1.53 and 401.08 ± 0.76 mg GAE·L^{-1} respectively. Similar results were reported on unroasted Arabica coffee with 473.51 mg GAE·L^{-1} [15]. The application of the FVE allowed an increase of 21% of the total phenolics content. This increase is usual during FVE application on peels and was extensively reported for red grape processing [5,16]. It was attributed to a greater cell disruption, thank to expansion force caused by sudden water evaporation during the expansion, the formation of microchannels which allows a more effective release of the compounds and the solubilization of pectin during the process. The same behavior has been also reported in the literature for different plant matrices treated entire, with peel and seeds, such as avocados and grapes (6 times and 49% more polyphenols after FVE, respectively) [17,18]. After entire mangoes FVE processing, an increase of 38.15% of polyphenols content was reported after 20 min of heat exposition at 100 °C [3]. This polyphenol release was related to a diffusion of the polyphenols from the non-comestible parts to the puree thank to the rupture of cell vacuoles. Vacuoles are the most sensitive organelles to the FVE process, because they store high amount of water and phenolic compounds. This phenomenon could explain the observation on coffee peels diffusion.

After FVE, the enriched extracts were microencapsulated by spray drying.

3.2. Physicochemical Characterization of the Powders

The physicochemical characterization results are shown in Table 1.

Table 1. Physicochemical characterization of MD10 and GA.

Analysis	MD10	GA
Moisture (%)	2.42 ± 0.01 [a]	2.50 ± 0.01 [a]
Water activity	0.23 ± 0.02 [a]	0.17 ± 0.01 [b]
Bulk density (g·mL^{-1})	0.249 ± 0.006 [a]	0.274 ± 0.015 [b]
Tapped density (g·mL^{-1})	0.353 ± 0.010 [a]	0.420 ± 0.024 [b]
Flowability (CI)	29.55	34.65
Cohesiveness (HR)	1.41	1.53
Particle size (μm)	8.61 ± 1.58	12.97 ± 2.40
Hygroscopicity (g of H_2O/100 d.b.)	13.95 ± 0.12 [a]	14.69 ± 0.14 [b]
Color parameters		
L*	89.17 ± 0.71 [a]	57.57 ± 0.80 [b]
a*	7.57 ± 0.30 [a]	2.53 ± 0.02 [b]
b*	11.44 ± 0.34 [a]	8.97 ± 0.08 [b]
Hue angle	56.49 ± 0.30 [a]	74.23 ± 0.09 [b]
Chroma	13.72 ± 0.45 [a]	9.32 ± 0.08 [b]

Different letters in the same row represent a significant difference (ANOVA, $p < 0.05$).

3.2.1. Moisture Content and Water Activity

Moisture of powders is important for the determination of flowability, stickiness, and stability during storage, due to water content effect on glass transition and crystallization [19]. The values obtained in this work are consistent with the reported literature for spray-dried powders using maltodextrin and arabic gum as wall materials with a mean content of 2.45% [20]. This value ensured the microbiological stability of the powder [19]. Indeed, the moisture is an important index, because it indicates the availability of free water in a food system, the "water activity", that is responsible for biochemical reaction. Water activity of MD10 and GA were found within the interval also indicated by Da silva et al. [21], as necessary to ensure the microbiological stability of powders (typically a water activity <0.35).

3.2.2. Hygroscopicity

Hygroscopicity is the ability of a material to adsorb moisture from the surrounding environment. It is a property to consider because it brings information about the powder behavior during storage [22], it helps to understand both the physical stability and the applicability of the product. GA was significantly ($p < 0.05$) more hygroscopic than MD10 (14.69 and 13.95 g of H_2O/100 g of d.b., respectively). The results obtained for both powders were lower than those reported for other microencapsulated products with maltodextrin and gum Arabic, hygroscopicity >19 g of H_2O/100 g of d.b. [20]. There is different operating condition during spray-drying that affect the hygroscopicity of powders, such as the increase in the inlet temperature. The total moisture content also influences this physical parameter. However, in this study the inlet temperature was the same for both wall materials, and the moisture content did not show significant differences. The higher capacity of water adsorption observed for GA powders may be associated with the difference of hydrophilic groups in the structure of each wall material. Arabic gum, which have a greater number of hydrophilic groups, presented greater adsorption of ambient humidity, while maltodextrin 10 DE have less hydrophilic groups [9]. The same behavior was observed by Da Silva et al. [21].

3.2.3. Particle Size and, Bulk and Tapped Densities

Bulk density refers to the density of a material when it is packaged. As reported in Table 1, GA value was significantly higher for this parameter, in agreement with Tonon et al. [23]. The bulk density is related to the molecular weight of the wall materials, the heavier a wall material is, the easier it is to accommodate in the spaces between the particles. Therefore, the powder occupies less space, resulting in higher bulk density values. GA has a higher molecular weight than MD10, this can contribute to explain why its bulk density is higher. An increase in this value indicates a lower amount of air in the powders, which can help prevent oxidation and promote stability during storage [10]. The tapped density is a property that give information on the packaging, transport and commercialization. It is frequently used to determine the properties of compressibility and cohesiveness of the powders. The augmentation of the compacted density could be related to the fact that GA presented a particle size larger than MD10, with means values of 12.97 ± 2.4 and 8.61 ± 1.58 μm respectively. A similar behavior was reported by Ozdikicierler et al. [24], for spray-dried powder of *Gypsophila* plant extracts using maltodextrin as wall material.

3.2.4. Flowability and Cohesiveness

The Carr index (IC) evaluates the free flow characteristics and the Hausner ratio (RH) evaluates the cohesion of the powders. The results of IC and RH are shown in Table 1. The values obtained were consistent with Bhusari et al. [25], who reported results from 19.34 to 34.16 and, 1.22 to 1.51 for IC and RH, respectively. Both powders, MD10 and GA were characterized by a medium fluidity and a high cohesiveness according to Carr and

Hausner classification [11,12]. A higher IC value indicates a poor fluidity, therefore, the use of MD10 as wall material improves the fluidity of the powders. This is related to the cohesion of these powders, the lower the cohesion, the better the fluidity of the powders (Ee et al., 2014). Thus, a free-flowing material has a tendency to have a greater consolidation, which is useful to avoid production stops on an industrial scale [26].

3.2.5. Color Properties and total Phenols Content

The use of MD10 as wall material allowed a significant increase in the parameter L*, which indicates the luminosity or opacity of a sample (100 for very bright and 0 for opaque samples). This behavior was associated with the optical characteristics of the maltodextrin, since this compound has a bright white color. On the other side, arabic gum has a yellowness color, less bright. This difference in color of the wall material can influence the perception of the powder color, since those compounds represent a high proportion of the powder dry matter. GA showed a significant decrease in the intensity of the chroma value and hue angle. This sample presented a greater yellow color (74°), while MD10 exhibited a color shade with a greater tendency towards red colors (56°). It can also be observed in the evolution of the parameter a*, which represents the red shades. MD10 a* was similar to reported by Parra-Campos and Ordóñez-Santos [7], for ethanolic extracts of coffee exocarp who reported values in a range of 5.42–6.51. Although this decrease in red could be related to the loss of anthocyanins, a more detailed study is suggested to understand its conformation. Phenols content was significantly higher for MD10. And in agreement with the literature for coffee based products [15]; therefore, MD10 and GA powders could be considered as an antioxidant food ingredient, with a potential beneficial effect on human health.

4. Conclusions

The FVE process proved to be an interesting alternative for the extraction of phenolic compounds from entire coffee peel waste using ethanolic extracts. The use of maltodextrin and arabic gum during spray drying allowed to obtain particles with low moisture and water activity which might to guarantee their microbiological stability. Powders elaborated with maltodextrin as wall material produce less hygroscopic particles, with significant better flow and cohesiveness properties, as well as color properties. Lastly, the contents of total phenols in both powders were in agree with the literature.

Therefore, those products, which can be considered as potential food ingredients with functional properties, added value to coffee production since they are obtained from a waste by a green emerging technology.

Author Contributions: Conceptualization, U.R.M.C. and V.M.R.A.; methodology, U.R.M.C. and A.S.; validation, U.R.M.C., M.A.V.O. and A.S.; formal analysis, U.R.M.C. and M.P.R.D.; investigation, M.A.S.C. and C.A.O.S.; resources, V.M.R.A.; data curation, U.R.M.C., C.A.O.S.; writing—original draft preparation, U.R.M.C., A.S. and M.P.R.D.; writing—review and editing, U.R.M.C. and A.S.; visualization, N.G.C.; supervision, V.M.R.A. and M.P.R.D.; project administration, U.R.M.C. All authors have read and agreed to the published version of the manuscript.

Funding: This project was financed by CONACYT, through the scholarship "Estancias posdoctorales por México 2022" Application No. 2513321, CVU. 592639.

Institutional Review Board Statement:

Informed Consent Statement:

Data Availability Statement:

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Esquivel, P.; Jimenez, V.M. Functional properties of coffee and coffee by-products. *Food Res. Int.* **2012**, *46*, 488–495. https://doi.org/10.1016/j.foodres.2011.05.028.
2. Arias, C.; Rodríguez, P.; Soto, I.; Vaillant, R.; Cortés, M.; Vaillant, F. Flash-vacuum expansion, a low-cost and energy-efficient alternative process to produce high-quality fruit puree: Application to *Physalis peruviana*. *Heliyon* **2023**, *9*, e16969**.**
3. Marin-Castro, U.R.; Garcia-Alvarado, M.A.; Vargas-Ortiz, M.; Pallet, D.; Salgado-Cervantes, M.; Servent, A. Sensory and nutritional qualities of 'Manila' mango ready-to-eat puree enhanced using mild flash vacuum expansion processing. *Fruits* **2021**, *76*, 248–257. https://doi.org/10.17660/th2021/76.5.5.
4. Ayala-Zavala, J.; Castillo-Romero, T.D.J.; Salgado-Cervantes, M.; Marin-Castro, U.R.; Vargas-Ortiz, M.; Pallet, D.; Servent, A. Review flash vacuum expansion: Effect on physicochemical, biochemical and sensory parameters in fruit processing. *Food Rev. Int.* **2023**, 35. https://doi.org/10.1080/87559129.2023.2197997.
5. Morel-Salmi, C.; Souquet, J.M.; Bes, M.; Cheynier, V. Effect of flash release treatment on phenolic extraction and wine composition. *J. Agric. Food Chem.* **2006**, *54*, 4270–4276. https://doi.org/10.1021/jf053153k.
6. Gharsallaoui, A.; Roudaut, G.; Chambin, O.; Voilley, A.; Saurel, R. Applications of spray-drying in microencapsulation of food ingredients: An overview. *Food Res. Int.* **2007**, *40*, 1107–1121.
7. Parra-Campos, A.; Ordóñez-Santos, L.E. Natural pigment extraction optimization from coffee exocarp and its use as a natural dye in French meringue. *Food Chem.* **2019**, *285*, 59–66. https://doi.org/10.1016/j.foodchem.2019.01.158.
8. AOAC. *Official Methods of Analysis*; Association of Official Analytical Chemists: Arlington, VA, USA, 1995.
9. Cai, Y.Z.; Corke, H. Production and properties of spray-dried Amaranthus betacyanin pigments. *J. Food Sci.* **2000**, *65*, 1248–1252. https://doi.org/10.1111/j.1365-2621.2000.tb10273.x.
10. Santhalakshmy, S.; Bosco, S.J.D.; Francis, S.; Sabeena, M. Effect of inlet temperature on physicochemical properties of spray-dried jamun fruit juice powder. *Powder Technol.* **2015**, *274*, 37–43. https://doi.org/10.1016/j.powtec.2015.01.016.
11. Carr, R.L. Evaluating flow properties of solids. *Chem. Eng.* **1965**, *72*, 163–168.
12. Hausner, H.H. *Friction Conditions in a Mass of Metal Powder*; Polytechnic Inst. of Brooklyn: Los Angeles, CA, USA, 1967.
13. Singleton, V.L.; Orthofer, R.; Lamuela-Raventós, R.M. Analysis of total phenols and other oxidation substrates and antioxidants by means of folin-ciocalteu reagent. In *Methods in Enzymology*; Academic Press: Cambridge, MA, USA, 1999; Volume 299, pp. 152–178.
14. Maskan, M. Kinetics of colour change of kiwifruits during hot air and microwave drying. *J. Food Eng.* **2001**, *48*, 169–175. https://doi.org/10.1016/S0260-8774(00)00154-0.
15. Kaisangsri, N.; Selamassakul, O.; Sonklin, C.; Laohakunjit, N.; Kerdchoechuen, O.; Rungruang, R. Phenolic compounds and biological activities of coffee extract for cosmetic product. *SEATUC J. Sci. Eng.* **2020**, *1*, 71–76.
16. Ntuli, R.G.; Saltman, Y.; Ponangi, R.; Jeffery, D.W.; Bindon, K.; Wilkinson, K.L. Impact of skin contact time, oak and tannin addition on the chemical composition, color stability and sensory profile of Merlot wines made from flash détente treatment. *Food Chem.* **2023**, *405*, 134849. https://doi.org/10.1016/j.foodchem.2022.134849.
17. Vargas-Ortiz, M.; Rodríguez-Jimenes, G.; Salgado-Cervantes, M.; Pallet, D. Minimally Processed Avocado Through Flash Vacuum-Expansion: Its Effect in Major Physicochemical Aspects of the Puree and Stability on Storage. *J. Food Process. Preserv.* **2017**, *41*, e12988. https://doi.org/10.1111/jfpp.12988.
18. Paranjpe, S.S.; Ferruzzi, M.; Morgan, M.T. Effect of a flash vacuum expansion process on grape juice yield and quality. *LWT-Food Sci. Technol.* **2012**, *48*, 147–155. https://doi.org/10.1016/j.lwt.2012.02.021.
19. Bazaria, B.; Kumar, P. Effect of whey protein concentrate as drying aid and drying parameters on physicochemical and functional properties of spray dried beetroot juice concentrate. *Food Biosci.* **2016**, *14*, 21–27.
20. Mahdavi, S.A.; Jafari, S.M.; Assadpoor, E.; Dehnad, D. Microencapsulation optimization of natural anthocyanins with maltodextrin, gum Arabic and gelatin. *Int. J. Biol. Macromol.* **2016**, *85*, 379–385.
21. da Silva Carvalho, A.G.; da Costa Machado, M.T.; da Silva, V.M.; Sartoratto, A.; Rodrigues, R.A.F.; Hubinger, M.D. Physical properties and morphology of spray dried microparticles containing anthocyanins of jussara (*Euterpe edulis* Martius) extract. *Powder Technol.* **2016**, *294*, 421–428. https://doi.org/10.1016/j.powtec.2016.03.007.
22. Daza, L.D.; Fujita, A.; Fávaro-Trindade, C.S.; Rodrigues-Ract, J.N.; Granato, D.; Genovese, M.I. Effect of spray drying conditions on the physical properties of Cagaita (*Eugenia dysenterica* DC.) fruit extracts. *Food Bioprod. Process.* **2016**, *97*, 20–29. https://doi.org/10.1016/j.fbp.2015.10.001.
23. Tonon, R.V.; Brabet, C.; Hubinger, M.D. Anthocyanin stability and antioxidant activity of spray-dried açai (*Euterpe oleracea* Mart.) juice produced with different carrier agents. *Food Res. Int.* **2010**, *43*, 907–914.
24. Ozdikicierler, O.; Dirim, S.N.; Pazir, F. The effects of spray drying process parameters on the characteristic process indices and rheological powder properties of microencapsulated plant (*Gypsophila*) extract powder. *Powder Technol.* **2014**, *253*, 474–480. https://doi.org/10.1016/j.powtec.2013.12.004.
25. Bhusari, S.N.; Muzaffar, K.; Kumar, P. Effect of carrier agents on physical and microstructural properties of spray dried tamarind pulp powder. *Powder Technol.* **2014**, *266*, 354–364. https://doi.org/10.1016/j.powtec.2014.06.038.
26. Lourenço, S.C.; Moldão-Martins, M.; Alves, V.D. Microencapsulation of pineapple peel extract by spray drying using maltodextrin, inulin, and arabic gum as wall matrices. *Foods* **2020**, *9*, 718. https://doi.org/10.3390/foods9060718.